Summer 7-14-2023

Draft Final Revised Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)

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July 14, 2023

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RE: Draft Final Revised Residential Metals Abatement Program (RMAP) QAPP (Residential Parcels)

Agency Representatives:

I am writing to you on behalf of Atlantic Richfield Company and Butte-Silver Bow to submit the Draft Final Revised *Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)*, which addresses residential properties including residential daycares and commercial properties containing living space under the RMAP program. This submittal addresses EPA's informal October 31, 2022, redline comment package. The report and appendices may be downloaded at the following link:

https://pioneertechnicalservices.sharepoint.com/:f:/s/submitted/EsmDAN_xKVpHhlpk0WZTj_UBckIWnOIA_Sqt057d9pw_fQ

If you have any questions or comments, please call me at (907) 355-3914 or Eric Hassler at (406) 497-5042.

Sincerely,

Mike Mc Anulty
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An affiliate of **Atlantic Richfield Company**

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SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

Draft Final Revised

Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)

Butte-Silver Bow County

and

Atlantic Richfield Company

July 14, 2023

SILVER BOW CREEK/BUTTE AREA NPL SITE
BUTTE PRIORITY SOILS OPERABLE UNIT

Draft Final Revised

Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)

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July 14, 2023

APPROVAL PAGE

Quality Assurance Project Plan for
BPSOU Residential Metals Abatement Program
(Residential Parcels)
Butte Area NPL Site

Approved: _____ Date: _____
Nikia Greene, Project Manager, EPA, Region 8
Quality Assurance Approval Official

Approved: _____ Date: _____
Daryl Reed, Project Officer, Montana DEQ

Approved: _____ Date: _____
Eric Hassler, Director
Department of Reclamation and Environmental Services
Butte-Silver Bow County

Approved: _____ Date: _____
Mike Mc Anulty, Liability Manager
Atlantic Richfield Company

Plan is effective on date of approval.

DISTRIBUTION LIST
Quality Assurance Project Plan for
BPSOU Residential Metals Abatement Program
(Residential Parcels)
Butte Area NPL Site

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A complete list of personnel to receive this document is provided on the associated cover letter distribution list. Atlantic Richfield Company will distribute the original Agency-approved document. Subsequent annual revisions will be distributed by the Butte-Silver Bow County Department of Reclamation and Environmental Services Quality Assurance (QA) Manager.

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DOCUMENT MODIFICATION SUMMARY

Modification	Author	Version	Description	Date
Rev 0	Scott Sampson	Draft Final	Issued for Agency Review	6/28/2017
Rev 0	Jesse Schwarzrock	Draft Final Revised	Re-issued for Agency Approval	01/10/2022
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LIST OF ACRONYMS

Acronym	Definition	Acronym	Definition
%D	percent difference	**ID**	Identification
%R	percent recovery	**ICP-AES**	Inducted Coupled Plasma Atomic Emission Spectroscopy
°C	degrees Celsius	**ICP-MS**	Inductively Coupled Plasma Mass Spectrometry
µg/m^3	micrograms per cubic meter	**LBP**	lead-based paint
µm	micron	**LCS**	Laboratory Control Sample
µg/L	microgram per Liter	**LMS**	Laboratory Matrix Spike
Agencies	U.S. Environmental Protection Agency and Montana Department of Environmental Quality	**MDL**	Method Detection Limit
Atlantic Richfield	Atlantic Richfield Company	**mg/cm^2**	
bgs	Below Ground Surface	**mg/kg**	milligram per kilogram
		MS	Matrix Spike
BPSOU	BPSOU Butte Priority Soils Operable Unit	**MSD**	Matrix Spike Duplicate
BSB	Butte-Silver Bow	**NIST**	National Institute of Standards and Technology
CCR	Construction Completion Report	**NPL**	National Priorities List
CFRSSI	Clark Fork River Superfund Site Investigations	**NTU**	Nephelometric Turbidity Unit
CLP	Contract Laboratory Program	**PARCCS**	precision, accuracy, representativeness, comparability, completeness, and sensitivity
COC	Contaminant of Concern	**pdf**	Portable document format
COI	Contaminant of Interest	**QA**	Quality Assurance
CVAA	cold-vapor atomic absorption	**QAPP**	Quality Assurance Project Plan
DEQ	Montana Department of Environmental Quality	**QC**	Quality Control
DQA	Data Quality Assessment	**QMP**	Quality Management Plan
DQO	Data Quality Objectives	**RA**	Remedial Action
DSR	Data Summary Report	**RL**	Reporting Limit
DU	decision unit	**RMAP**	Residential Metals Abatement Program
EBL	Elevated Blood Lead	**ROD**	Record of Decision
EDD	Electronic Data Deliverable	**RPD**	relative percent difference
EPA	U.S. Environmental Protection Agency	**SAP**	Sampling Analysis Plan
ESD	Explanation of Significant Differences	**SDG**	Sample Delivery Group
FSP	Field Sampling Plan	**SOP**	Standard Operating Procedure
HAZWOPER	Hazardous Waste Operations and Emergency Response	**SQL**	Structured Query Language
HEPA	High Efficiency Particulate Air	**SRM**	Standard Reference Material
HUD	U.S. Housing and Urban Development	**SSHASP**	Site-Specific Health and Safety Plan
IC	Institutional controls	**UAO**	Unilateral Administrative Order
ICIAP	Institutional Controls Implementation and Assurance Plan	**XRF**	X-ray fluorescence

1.0 INTRODUCTION

The Butte-Silver Bow (BSB) County *Revised Final Multi-Pathway Residential Metals Abatement Program Plan* (RMAP) (BSB & Atlantic Richfield Company, 2022) (hereafter referred to as the Program) is designed to mitigate exposure to sources of arsenic, lead, and mercury contamination for residents of the Butte Priority Soils Operable Unit (BPSOU), the larger Butte community as a whole, and rural residential development within the Silver Bow Creek/Butte Area Superfund Site. The current Program boundary (depicted as the 2020 RMAP Area) is shown on Figure 1. Medical monitoring is conducted as a sister program to evaluate the effectiveness of the Program. Medical monitoring results are used to prioritize property assessment and remediation activities.

The contamination in the Butte community may originate from mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources (lead-based paint [LBP] and lead solder). Possible sources of arsenic, lead, and/or mercury exposure addressed in the Program include arsenic, lead, and mercury present in yard soil and interior living space dust; lead present in interior and/or exterior LBP and drinking water from pipe solder; arsenic, lead, and mercury present in attic dust when a pathway exists between attic and living space and/or earthen basement soil; and mercury vapor. The Program ensures its effectiveness through the remediation and abatement of contaminated properties and community awareness and education.

The Program requires systematic sampling of residential yard soil, interior living space dust, and attic dust within the BPSOU. Drinking water, LBP, and mercury vapor may also be sampled if evidence indicates this additional sampling is warranted. For areas outside of the BPSOU, but within the 2020 RMAP Area shown on Figure 1, a test-by-request campaign will be implemented in place of a systematic sampling approach. This test-by-request campaign will identify sampling efforts and necessary remedial work. The Program also requires systematic sampling of playground and play areas (e.g., schools and parks) within the 2020 RMAP Area (refer to Figure 1).

Properties with soil or interior living space dust exceeding solid media action levels or indoor air exceeding the mercury vapor action level will be remediated. If attic dust exceeds the action level and a pathway exists between the attic and living space, the attic will be remediated. If both living space and attic dust exceed the action levels, a pathway will be assumed and the attic will be remediated. The Program also includes a process to determine when water samples will be collected and analyzed to determine whether a home has lead pipes and/or lead solder that may be contributing to an unacceptable exposure. The Program contains additional institutional control (IC) measures regarding education, outreach, and tracking programs related to remedial activities at residential properties, as further described in the *BPSOU Institutional Controls Implementation and Assurance Plan* (ICIAP) (BSB & Atlantic Richfield Company, 2019a).

1.1 Purpose

The *BPSOU Quality Management Plan (QMP)* (Atlantic Richfield Company, 2016) provides guidance to ensure environmental data collected for the BPSOU meet the quality requirements mandated by U.S. Environmental Protection Agency (EPA). The purpose of this Quality

Assurance Project Plan (QAPP) is to document the requirements for future RMAP sampling and analysis of residential properties (including residential daycares and commercial properties containing living space) and to describe the quality assurance/quality control (QA/QC) policies and procedures that will be used during these efforts. The current Program boundary (depicted as the 2020 RMAP Area) is shown on Figure 1. This QAPP functions as the Program sampling and analysis plan (SAP) for all future residential sampling activities. The *Final RMAP QAPP (Non-Residential Parcels)* (BSB and Atlantic Richfield Company, 2023) was developed to address non-residential RMAP parcels (e.g., schools, parks, and non-residential daycares).

This QAPP is composed of standard recognized elements referenced in the *EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5* (EPA, 2001), the *Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G4* (EPA, 2006a), and the *EPA Region 8 Quality Assurance Document Review Crosswalk Checklist for Quality Assurance Project Plans* (EPA, 2016) provided in Attachment A. This QAPP includes the following five key elements:

- Program management and organization (Section 2.0).
- Measurement and data acquisition (Section 3.0).
- Reclamation Material (Section 4.0).
- Assessment and oversight (Section 4.0).
- Data review and usability (Section 5.0).

Attachment B through Attachment H provide standard operating procedures (SOPs), sample collection procedures, data validation checklists, manufacturer's instructions, and other detailed information pertaining to this QAPP. Attachment I contains summaries of the annual QAPP revisions.

The sections below provide the project elements and include details for planning, sampling, and analysis within the Program areas. Sections in this QAPP expand on or reference information in other site-wide documents and present project-specific requirements.

2.0 PROGRAM MANAGEMENT AND ORGANIZATION

This section addresses Program and project administrative functions and project background, objectives, and documentation requirements for sampling and analysis activities on each project site within the Program area. Project roles and the responsibilities of these roles are described below.

2.1 Agency Oversight

The EPA and Montana Department of Environmental Quality (DEQ) (the Agencies) are responsible for project oversight and review and approval of all Program generated sampling data and subsequent site-specific remediation plans. Nikia Greene is the EPA Remedial Project Manager, and Daryl Reed is the DEQ Project Officer.

The Agencies also review all sampling results and project completion reports.

2.2 Atlantic Richfield Company

Atlantic Richfield Company (Atlantic Richfield) provides Program funding through an Allocation Agreement between BSB and Atlantic Richfield. The Atlantic Richfield Liability Manager, Mike Mc Anulty, must authorize all reclamation activities under the Program. An Atlantic Richfield project representative, or designated alternate, may complete a site walk-through and assist with approving site-specific work plans for all reclamation projects prior to implementation.

Atlantic Richfield may elect to self-perform portions of the RMAP sampling and analysis work to supplement BSB resources. The Agencies will be consulted prior to Atlantic Richfield conducting any RMAP sampling and analysis work.

2.3 Butte-Silver Bow County Department of Reclamation and Environmental Services

Butte-Silver Bow County is responsible for notifying qualifying property owners of possible exposure within the property, obtaining property owner access to conduct sampling and abatement (as needed), maintaining all Program data, and coordinating abatement activities. Key individuals comprising the BSB Department of Reclamation and Environmental Services are shown on Figure 2. The Program project team responsibilities are described below.

Director – Eric Hassler
The Director will oversee all activities throughout the department and is responsible for maintaining the official approved QAPP and for ensuring that the work is performed according to the QAPP requirements. The Director is also responsible for consulting with the Assistant Director regarding any project deficiencies and resolutions.

Assistant Director – Julia Crain
The Assistant Director will perform various coordinating responsibilities across operable units while assisting with data-related activities.

Manager, Human Health/RMAP Division – Chad Anderson
The Human Health/RMAP Division Manager will coordinate all RMAP activities and oversee division crews and staff. Furthermore, the Manager is responsible for verifying the QAPP requirements and procedures are implemented effectively and scheduling sampling work to be completed. This includes reviewing field and laboratory data and evaluating data quality. The Manager will also complete a site walk-through, prepare a site-specific work plan for approval for all reclamation projects prior to implementation, and provide project oversight.

The Manager will also be responsible for the oversight of field team laborers during abatement activities to complete the duties listed below:

- Scheduling sampling work to be completed.
- Managing requests for property access, tracking the status of access requests, and maintaining copies of completed agreements received from property owners.

- Ensuring completed agreements are photocopied, scanned, and the electronic version stored on a hard drive.
- Ensuring a copy of the individual access agreement is included in the project record files.
- Ensuring that all team members review and properly follow the QAPP during field activities.
- Conducting daily safety meetings, assisting in field activities, and documenting activities in the field logbook or appropriate field collection device.
- Coordinating field activities and managing equipment.
- Solving problems and making decisions in the field.
- Managing technical aspects of the project.
- Maintaining an on-the-ground overview of the project tasks by observing site activities.
- Ensuring compliance with technical project requirements and the Site-Specific Health and Safety Plan (SSHASP).
- Identifying issues during field activities and reporting all issues to the RMAP Coordinator.

Data Management Division/Quality Assurance Manager – Abigail Peltomaa
The Data Management Division Manager assumes the role of Program QA Manager and is responsible for managing data and QA/QC of all field data, reviewing and maintaining laboratory data packages, compiling an annual Data Summary Report (DSR), maintaining quality records (as described in Section 2.9.6), and reporting final remediated property requirements to the Agencies.

2.4 Analytical Laboratory

Pace Analytical Services, LLC will provide analytical laboratory services for the Program. Anticipated laboratory locations will be in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) and Green Bay, Wisconsin (1241 Bellevue Street, Suite 9, Green Bay, WI 54302). Jennifer Anderson is the point of contact for Pace Analytical Services, LLC.

All laboratories contracted to work on Program projects must ensure that the laboratory's QA personnel are familiar with this QAPP and are performing the analytical and QC work as specified in laboratory methods and this QAPP. Laboratory QA personnel are responsible for reviewing final analytical reports produced by the laboratory, coordinating the laboratory analysis schedule, and supervising in-house chain of custody procedures.

2.5 Problem Definition and Background

Contamination of properties described herein may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related sources (LBP and lead solder). The potential sources of arsenic, lead, and/or mercury exposure addressed in the Program include arsenic, lead, and mercury present in yard soil and interior living space dust, lead present in interior and/or exterior LBP and drinking water from pipe solder, and arsenic, lead, and mercury

vapor exposure through attic dust when a pathway exists between the attic and living space and/or earthen basement soil, and mercury vapor.

Sampling is needed to determine remediation or abatement requirements if:

- Residential yard, basement soil, or interior living space dust exceeds solid media action levels.
- Indoor air exceeds the mercury vapor action level.
- Attic dust exceeds the action level and a pathway exists between the attic and living space.
- There is an unacceptable lead exposure at a residence due to painted surfaces, pipes, and/or lead solder.

This QAPP was developed in response to the 2006 BPSOU *Record of Decision* (BPSOU ROD) (EPA, 2006b) and *Explanation of Significant Differences* (ESD) *to the 2006 Butte Priority Soils Operable Unit Record of Decision* (EPA, 2011). The ESD modified the soil sampling depth from 0 to 2 inches to the depth intervals discussed in Section 3.1, changed the soil removal from a minimum depth of 18 inches to the minimum depth of 12 inches or to the soil bedrock interface if less than 12 inches, and extended the project schedule to accommodate the expansion of the Program.

This QAPP was also developed in response to the Agencies 2020 *Unilateral Administrative Order Amendment (UAO Amendment) for "Partial Remedial Design/Remedial Action Implementation and Certain Operation and Maintenance at the Butte Priority Soils Operable Unit/Butte Site"* (EPA Docket No. CERCLA-08-2011-0011) (EPA, 2020a). The UAO Amendment expanded the RMAP boundary (2020 RMAP Area) (refer to Figure 1) and extended the Program to include schools, parks, and daycare facilities.

RMAP program representatives will provide results of sampling data to the Agencies and notify property owners of their sample results and any necessary abatement.

2.6 Project Description and Schedule

The Program is designed to mitigate exposure to sources of arsenic, lead, and mercury contamination, which may originate from both mining-related (waste rock, tailings, aerial emissions) and non-mining-related (LBP and lead solder) sources for residents of the BPSOU and the 2020 RMAP Area.

In 2020, the Program was expanded to perform both attic and yard sampling within the 2020 RMAP Area shown on Figure 1. Specific exclusion areas are also identified on Figure 1. Residential yards and attics outside of the BPSOU but within the 2020 RMAP Area will be sampled on a test-by-request basis.

Components of the Program include environmental sampling and remediation, long-term tracking and data management, and education and outreach. As noted above, the medical monitoring is conducted as a sister program to the Program. Medical monitoring results, such as

elevated blood lead (EBL) measurements, are used to prioritize property assessment and remediation activities. Detailed information on the medical monitoring program is presented in the *BSB Medical Monitoring Program Plan* (BSB & Atlantic Richfield Company, 2021).

The Program specifies sampling residential yard soil, interior living space dust, and attic dust for all contaminants of concern (COCs) within the BPSOU; as appropriate, sampling of interior air for mercury vapor and lead in paint and drinking water may also be performed. Environmental sampling of residential properties outside of the BPSOU, but within the 2020 RMAP Area shown on Figure 1, will occur upon request. Program eligibility is described in Section 1.2 of the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022).

The objectives of this QAPP are as follows:

1. Provide consistent means and methods for sampling and analysis of solid media and mercury vapor associated with the Program sampling activities (soil, interior and attic dust, and lead based paint, drinking water, and interior mercury vapor in air) of residential parcels including residential properties, residential daycares, and commercial properties containing living space and ensure compliance with performance standards.
2. Describe the requirements for sample collection and analysis.
3. Provide data to identify and mitigate potentially harmful exposure to sources of arsenic, lead, or mercury.

2.6.1 Project Schedule

The Program maintains a quantitative implementation schedule, but access to residential properties within the BPSOU and the number of requests from residential properties within the 2020 RMAP Area constrains it. The Program attempts to sample and remediate (if necessary) 100 residential attics per year and 60 residential yards per year. However, since the implementation rate is contingent upon program participation, the achieved rate is likely to vary from that specified.

The 2011 ESD required three attempts within 10 years to perform assessments of all residential properties (including residential quarters within commercial/industrial properties) within the BPSOU with a goal of remediating all contaminated properties within the BPSOU within 20 years, except for those properties for which access cannot be obtained. In November 2020, the 10- and 20-year time frame goals for completing these activities in the BPSOU began.

The directive for outreach requires three attempts (by mail or other documented means) to obtain access from the current owner of record for sampling or remediation of BPSOU residential properties. If these attempts are unsuccessful, assessment and any required remediation will be conducted in the future when and if access to the property is obtained for RMAP purposes.

For residential properties outside of the BPSOU but within the 2020 RMAP Area, the program does not have a fixed schedule or time limit established, but the goal of assessing these properties within 20 years is set to coincide with completing residential remediation within the

BPSOU. Sampling of these parcels will be triggered by individual landowner requests. A passive outreach approach will be implemented and will generally consist of bi-annual public services announcements, public education on the BSB Superfund website, etc. These sampling requests will be prioritized by order of receipt unless site-specific conditions warrant expediting certain requests.

Lead-based paint, drinking water, and mercury vapor may also be sampled if evidence indicates this additional sampling is warranted. Because the Program also stipulates that attic dust will not be cleaned up unless there is an established exposure pathway, it requires that properties be tracked for the duration of the Program to assess and abate attic dust when the possibility of exposure exists.

2.7 Quality Objectives and Criteria

This section discusses the internal QC and review procedures used to ensure that all data collected for this project are of known quality. The Data Quality Objectives (DQOs) were developed according to EPA's *Guidance on Systematic Planning Using the Data Quality Objectives Process* (EPA, 2006a). The DQOs are statements that define the type, quality, quantity, purpose, and use of data to be collected. The EPA developed a seven-step process to establish DQOs to help ensure that data collected during a field-sampling event are adequate to support reliable site-specific decision making (EPA, 2001 & EPA, 2006a). The sections below outline the QAPP DQOs.

2.7.1 Data Quality Objectives

The DQO process specifies project decisions, the data quality required to support those decisions, specific data types needed, data collection requirements, and analytical techniques necessary to generate the specified data quality. The process also ensures justification of the resources required to generate the data. The DQO process consists of seven steps where the output from each step influences the choices that will be made later in the process:

- Step 1: State the Problem.
- Step 2: Identify the Goals of the Study.
- Step 3: Identify the Information Inputs.
- Step 4: Define the Boundaries of the Study.
- Step 5: Develop the Analytic Approach.
- Step 6: Specify Performance or Acceptance Criteria.
- Step 7: Develop the Plan for Obtaining Data.

During the first six steps of the process, the planning team develops decision performance criteria that will be used to develop the data collection design. The final step of the process involves developing the data collection design based on the information from the other steps. The following provides a brief discussion of these steps and their application to this sampling effort.

Step 1: State the Problem - *The purpose of this step is to describe the problem to be studied so that the focus of the investigation will not be ambiguous.*

Describing the problem. The proximity of properties in Butte and within the 2020 RMAP Area (see Figure 1) to mining wastes and operations may have resulted in contamination of residential properties. Residential properties are defined as single- or multi-family residences, residential daycare/preschool facilities, and residential living quarters within a commercial property. Sources not related to mining (e.g., LBP, lead solder in plumbing, mercury in switches and old thermometers, natural mineralization) may also contribute to potential metals exposures. The presence of contaminants and exposure pathways, related and non-related to historical mining activities, may result in a health-based risk to residents.

Establishing the planning team. Project personnel, roles, and responsibilities are detailed in Sections 2.1 through 2.3 of this document.

Describing the conceptual model of the potential hazard. Historical surface and underground mining activities resulted in the presence of contaminants in soil around Butte due to waste dumping and deposition of aerial emissions from smelters/mills. Non-mining sources such as LBP and lead solder in plumbing of older homes, switches and old thermometers that can contain mercury, toys, dishes, cosmetics, natural mineralization, and other sources have also resulted in the presence of lead, arsenic, and mercury in some areas. People may contact contaminated soil and dust at residential properties through pathways such as dermal contact, incidental ingestion, and inhalation of dust or vapors. For example, children playing in a residential yard may have skin contact with exposed soil, some of which could be ingested through hand to mouth transfer. When people contact contaminated media, they may be exposed to contaminants, which could pose a health risk if concentrations are above health-protective concentrations, such as action levels. To investigate this problem, data quantifying contaminant concentrations will need to be collected, compared to the appropriate project action levels, and used for remedial decision making.

Identifying available resources, constraints, and deadlines. Atlantic Richfield (Section 2.2) and BSB (Section 2.3) will provide necessary project resources (financial and staffing) to properly implement the program. Project schedule details are provided in Sections 2.6 and 2.6.1. In addition to the constraints of access to residential properties within the BPSOU described in Section 2.6.1, the number of requests from residential properties within the 2020 RMAP Area can make staff availability another potential scheduling constraint. If necessary, subcontractors may be hired to supplement the field sampling teams to meet the UAO schedule. Additionally, multiple documents governing the sampling program (i.e., the *Revised Final Multi-Pathway RMAP Plan* [BSB & Atlantic Richfield Company, 2022], *RMAP QAPP*, and any associated field sampling plans) must be approved by the Agencies prior to implementation; sample request forms must be in place prior to sampling; field equipment must be procured prior to mobilization; and laboratory subcontracting agreements must be in place prior to analyses.

Step 2: Identify the Goals of the Study – *This step identifies what questions the study will attempt to resolve and what actions may result.*

Key elements/questions. The Program requires that all area residential properties within the BPSOU be sampled and assessed. The goal is to use best efforts to obtain access to all applicable properties within the 2020 RMAP Area (refer to Figure 1) that have not previously been sampled according to current methodology for completing indoor and outdoor assessments (i.e., residential yard soil, earthen basement soil, interior living space dust, attic/crawl space dust). Applicable properties are defined as those within the BPSOU or those outside of the BPSOU but within the 2020 RMAP Area shown on Figure 1 that have requested sampling.

Specifying the primary question. The primary question to be addressed is the following:

Are concentrations of arsenic, lead, and/or mercury in exterior soil that may be contacted during various activities (such as surface soil contact by children playing in a yard or deeper soil contact by a gardener), earthen basement soil, and/or interior living space/attic/crawl space dust at residential properties present at levels that may pose a risk to human health (e.g., above the action levels)?

Additional questions to be addressed under specific conditions, depending on the answer to the primary question, include the following:

For properties where earthen basement soil and/or indoor dust exceeds a mercury action level, are mercury vapor concentrations present at levels that may pose a risk to human health? If EBL levels exist and no other potential source of lead is discovered during the residential investigation, is LBP present? If not, are lead components in pipes a possible source of lead exposure?

Determining alternative actions. Possible alternative actions are as follows:

- Take no action – If all analyte concentrations are below the appropriate project action level, no action is necessary.
- Complete Additional Sampling – If mercury is present in earthen basement soil/indoor dust above the action level, mercury vapor would be sampled. If EBL levels exist and no other potential source of lead is discovered during the residential investigation, additional sampling would be completed to determine if LBP is present, and if lead is present in the tap water and additional abatement would be performed based on the outcome of this assessment.
- Complete Remedial Action (RA) – If an analyte concentration is above the appropriate project action level, remedial action would be completed. Details of remedial actions for residential yard/earthen basement soil, living space/attic/crawl space dust or mercury vapor, LBP, or tap water are provided in the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022).

Specifying the decision statement. The decision statement is as follows:

Determine whether additional sampling (conditional based on soil and dust sampling results) or RA (including soil or dust removal or capping, or other actions such as LBP remediation) is required.

Step 3: Identify the Information Inputs - *The purpose of this step is to identify the informational variables that will be required to resolve the decision statements and determine which variables require environmental measurements.*

Identifying the type of information that is needed to resolve the decision statement. Arsenic, lead, and mercury concentrations should be determined through sampling soil and indoor dust (and, if warranted, indoor air [mercury vapor only], paint [lead only], and/or tap water [lead only]) from residential RMAP properties. The goal of sample collection and analysis is to obtain a reliable estimate of the average concentration of a COC in a potential exposure medium (e.g., soil, dust, air, or water) over a specified area where exposure may occur (or in the case of LBP sampling, to determine if LBP is present). This reliable estimate should be compared with the appropriate action level for that area and medium. The relationship between the average COC concentration and the action level or the determination that LBP is/is not present provides the input needed to resolve the decision statements outlined in Step 2 to determine whether abatement/LBP remediation is required for residential RMAP properties.

Land use information should be used to make decisions about the appropriate sample count/density and depth intervals to be sampled and to identify action levels that are protective of the specified land use. Soil and dust sampling results should also be used to make decisions about the necessity for sampling of other media, such as indoor air (for mercury vapor), and the need to perform sampling of paint (for presence of LBP) and/or tap water (for lead) for properties where an EBL level has been identified. This QAPP addresses residential properties; therefore, subsample count/density, depth intervals to be sampled, and action levels should be consistent with residential assessment guidance (such as the EPA's *Superfund Lead-Contaminated Residential Sites Handbook* [EPA, 2003]) and residential RMAP criteria.

Sample coordinates, house locations, and depth intervals should also be documented so that sample results are linked to specific locations and depths to inform remediation decisions. The surface area of each yard component sampled should also be recorded. If chips from building exterior LBP are identified in a sampled area, this should also be documented as it is likely to influence lead concentrations in soil. Other dissimilar soil material types or surface conditions (i.e., bare ground areas, locations of obvious imported fill materials, etc.) should also be documented.

Identifying the number of variables to be collected. Arsenic, lead, and mercury concentrations should be determined for each yard/earthen basement soil and interior living space/attic/crawl space dust sample collected. Mercury vapor concentrations should also be determined through interior air sampling if interior dust or earthen basement soil concentrations exceed the appropriate mercury action level.

Blood lead data are collected as part of the Medical Monitoring Program, which is a sister program within the RMAP. These biomonitoring data are used to help prioritize properties for assessment. If EBL levels exist and no other potential source of lead is discovered during the residential investigation, painted surfaces should be sampled and analyzed for the presence of lead to investigate whether LBP is a possible source of lead exposure. Drinking water should also be sampled for the presence of lead to investigate whether lead components in pipes are a possible source of lead exposure.

Identifying the appropriate Action Levels. The BPSOU soil/dust and indoor air (mercury vapor only) action levels for lead, arsenic, and mercury were derived based on site-specific data and health protective assumptions in 1993, 1997, and 2003, respectively. The 2006 BPSOU ROD and subsequent analyses in the 5-year ROD reviews conducted in 2010 and 2016 confirmed and maintained these action levels. The lead action level was derived using EPA's Integrated Exposure Uptake Biokinetic model with site-specific soil to dust regression, geometric standard deviation, and bioavailability data and default residential exposure assumptions. The arsenic action level was based on a baseline risk assessment for the BPSOU using site-specific inputs for arsenic in soil and house dust and the bioavailability of arsenic in Anaconda soil based on a study in monkeys (Weis, 1994), which is consistent with results in the BPSOU in swine (EPA 2010). Results of the arsenic human health risk assessments in Anaconda and Butte were similar, and the action levels established for Anaconda were adopted for consistency among the two sites. EPA used exposure assumptions from a risk assessment for Walkerville (URS Operating Services, Inc. 2003) to determine the residential soil and indoor air action levels for mercury and mercury vapor, respectively.

Residential action levels developed for the BPSOU are protective of residential land use and should be used to make cleanup decisions for residential properties. The BPSOU residential action levels for soil and dust (Arsenic – 250 milligrams per kilogram [mg/kg], Lead – 1,200 mg/kg, Mercury – 147 mg/kg) and for indoor air (mercury vapor – 0.43 micrograms per meter cubed [$\mu g/m^3$]) will be used for all work completed under this QAPP (refer to Table 1). LBP does not have an action level. Rather, the presence of LBP is defined as 1.0 milligrams per centimeter squared (mg/cm^2) (see Table 1).

Identifying appropriate sampling and analysis methods. Multiple sampling strategies (discrete, incremental, composite, etc.) should be considered for use on this project. Because of the way that residents tend to access their yards/basements (i.e., moving around the area rather than staying in one specific location) and because interior dust/air can move around living spaces along with the people using the spaces, exclusive discrete sampling may not be the most appropriate option given its common deficiencies including poor spatial coverage, inadequate sample density, and data that cannot be used to statistically represent the entire area of interest with a reasonable level of confidence. While incremental sampling is a type of composite sampling, it would represent a change from current sampling practices within the Silver Bow Creek/Butte Area National Priorities List (NPL) Site. As such, a change could create issues surrounding consistency and comparability with previous RMAP and NPL Site sampling results. In addition to being used historically within the NPL Site and on the RMAP project specifically, composite sampling is the recommended approach for sampling residential parcels provided in EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003). Composite

sampling may also be the most appropriate sampling method for project consistency and comparability with previous RMAP and NPL Site sampling results. X-ray fluorescence (XRF) has been used historically to analyze arsenic and lead concentrations in Butte soil. This method provided a quick output that was used for immediate decision making. However, it is less sensitive than laboratory analytical methods and cannot be used for mercury analysis. Because samples must be packaged and shipped to a laboratory for mercury analysis, it may be more practical to have all three metals analyzed by the laboratory via inorganic analysis. Inorganic analysis data from an analytical laboratory can also be validated. If inorganic analysis methods are used, expedited laboratory analysis turnaround (five to seven business day turn around on data and level 2 data packages and 10 to 12 business day turn around on data and level 4 data packages) and data validation (seven business day turn around after Level 4 data packages are received) options should be investigated to achieve the project assessment and remediation goals.

For the same reasons described above for soil, laboratory analyses may also be preferable for interior dust samples to ensure that accurate and validated dust sample results can be obtained and used for RA decision making. This would also provide comparable soil and dust concentration data.

Prior to laboratory analyses, soil samples should be prepared to ensure the sample matrix is homogenized. In addition, soil samples for lead and arsenic analyses should be air-dried and sieved to 250 micrometers (µm) to represent the fine fraction of soil most likely to adhere to children's hands (see DQO Step 7 for additional discussion of the appropriate size fraction).

RMAP samples should be analyzed to determine metals concentrations via standard EPA analytical methodologies for arsenic, lead, and mercury in soil and dust, mercury in air, and lead in water. These include EPA Method 6010 or 6020B for analysis of lead and arsenic and EPA Method 7471B for cold-vapor atomic absorption (CVAA) analysis of mercury. Paint samples should be analyzed per the manufacturer user manual and Method 6200 as applicable for the XRF unit and the SOP XRF-001 in Attachment B-4 to determine concentrations of lead. Tap water samples should be analyzed per EPA 200.8 for total recoverable metals, as specified in the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022), to quantify the amount of lead that may be ingested by a resident drinking the water. This QAPP provides additional information and requirements that enhance or define QA direction to the laboratories and supplement or replace specific generic laboratory SOP requirements. Analytical methodology and requirements are detailed in Section 3.7.1 of this QAPP.

Mercury vapor samples will be collected if interior dust or earthen basement soil mercury concentrations are above action levels. For this follow-up sampling, it may be more appropriate to collect real-time data rather than shipping samples to a laboratory given the sensitivity around potential mercury vapors present in a living space, although laboratory analyses methods may be more accurate and can be validated. The benefits of each approach should be considered, and the method that best supports the goal of making health-protective RA decisions should be selected.

When it is necessary to sample painted surfaces, XRF has been used historically to analyze lead concentrations. Because the presence and specific locations (if present) of LBP are not known and must be investigated when certain conditions occur (i.e., EBL levels without a known

source) and given the large surface area to be investigated (i.e., exterior and interior painted surfaces), collection of paint samples for laboratory analysis is not practical and may result in missing specific locations where LBP is present. The XRF method that has been used historically enables investigation of a large surface area and immediate identification of locations where RA is needed. Therefore, this method should continue to be used when sampling of painted surfaces is necessary.

Due to the difficultly of gaining access to residences before the first water use of the day and because sampling the first morning's draw of tap water is recommended, drinking water samples, when necessary to collect, should be collected by the resident with consideration of drinking water regulations. Additional instruction on drinking water sampling is contained in Section 3.4.2 and Attachment C.

Step 4: Define the Boundaries of the Study – *The purpose of this step is to define the spatial and temporal boundaries of the problem.*

Specifying the target population. The 2020 Program area addressed under this QAPP will include yard/earthen basement soil and interior living space/attic/crawl space dust of residential properties, including residential daycares and commercial properties containing living space within the 2020 RMAP Area shown on Figure 1. Indoor air (mercury vapor only), drinking water (lead only), and painted surfaces (lead only) at residential properties may also be sampled on an as-needed basis.

Because of differences in potential soil exposures with depth and for consistency and comparability with previous RMAP sampling, yard soil should be sampled separately from discrete depth intervals. For example, EPA recommends sampling soil from the 0- to 2-inch depth interval to assess contact by most activities of children, while some activities may result in contact with deeper soil, and vegetable gardens may involve digging up to 2 feet. Exterior soil sampling should be conducted at multiple depth intervals (0 to 2 inches, 2 to 6 inches, and 6 to 12 inches) to assess potential health risks under different land uses and to obtain data that are comparable to those from previous sampling efforts. Flower/vegetable garden components should be sampled at additional depth intervals of 12 to 18 inches and 18 to 24 inches.

Description of what constitutes a sampling unit. Sampling units should be defined based on land use information and should represent areas where residents may be exposed to arsenic, lead, or mercury. The extents of a sampling unit are defined as the maximum area to be sampled to support decision-making for the residential land use category. EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) and the previous RMAP QAPP were reviewed to inform sampling unit extents for exterior soil. Recommendations consistent with EPA recommendations and other RMAP sampling efforts should inform development of the sampling plans for each property.

Specifically, for residential properties, exterior soil sampling units are defined based on the size of the residential yard and the presence of specific use areas. When the size of the residential property is less than 5,000 square feet, EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) recommends that the front yard, the back yard, and the side yard (if the

size is substantial) are each identified as separate sampling units. If the property is larger than 5,000 square feet, the lead handbook recommends that *"the property be divided into four quadrants of roughly equal surface area. The two quadrants in the front yard should encompass one half of the side yard; likewise for the two quadrants in the back yard."* If the property is larger than one acre, quarter-acre sections are recommended for defining sampling units. EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) also recommends defining distinct play areas and gardens as separate sampling units, when present.

The maximum area represented by a single residential composite sample within a standard residential lot will be 1,200 square feet. This value was calculated based on the size of a typical residential lot (approximately 5,000 square feet) to ensure proper characterization and multiple sampling components per lot. The maximum area represented by a single residential composite sample within a mid-sized residential lot (i.e., between 5,000 square feet and 1 acre in size) will be 6,250 square feet. The maximum area represented by a single residential composite sample within a large residential lot (i.e., greater than 1 acre in size) will be 10,890 square feet.

For interior dust sampling, the sampling unit should be defined based on the area where dust may be contacted (for living spaces) or from which a pathway might occur (for non-living spaces). Because dust and vapor can move around within an indoor space, the samples collected from these media should be representative of the entire space where a particular resident or other receptor (such as a child attending a residential daycare) spends time. For example, the interior living space where residents may contact dust includes entryways, living rooms, kitchens, and bedrooms; all these spaces together should be considered as part of the sampling unit since they are connected and transfer between areas can occur. If part of the home has a different use, such as a residential daycare separated from the family living space, or if the living space is apparently separated by dedicated entrances, the areas within the structure should be considered separate sampling units because different exposures may be applicable for each. Interior non-living spaces, from which a pathway to living spaces may originate, include attic and crawl spaces. As with living spaces, since dust can move around within an attic or crawl space, the sampling unit should include the whole space. Rather than square footage, composite dust sampling requirements are based on achieving a minimum sample mass (0.2 grams).

Similar to other interior spaces, earthen basements (unless separated into distinct areas accessed by different users) should be considered as one exposure area and the sampling unit should be defined as the entire basement or as the area of the basement designated for each use. For LBP and tap water, sampling units are also not size-based. For LBP, the sampling unit will vary depending upon the painted surfaces in question. For example, for an interior room, windowsills may be evaluated separately from interior walls if the paint sources appear to be different. Each potential paint source should be assessed as a separate sampling unit. For tap water, the appropriate sampling unit should be determined based on the faucet most likely to be used as a source for drinking water.

Time frame for collecting data and making the decision. The temporal boundaries of the investigation include the time from when evaluation and sampling actions begin at each property to the time these actions are complete. No temporal variability in residential yard or earthen basement soil concentrations is expected, and if soil is the source of indoor dust concentrations,

then temporal variability should be limited. The sampling effort should therefore be primarily dictated by when it is easiest to conduct sampling, meaning avoiding frozen ground conditions for exterior soil sampling and when residential daycare/preschool facilities are not in use (i.e., after hours). While temporal variability in concentrations should be limited in earthen basement soil and in indoor dust if soil is the source, mercury vapor sampling should be conducted as soon as possible if mercury concentrations in interior dust/earthen basement soil are above action levels since elevated mercury concentrations in indoor dust/earthen basement soil can be a source of mercury vapor, which is volatile. Similarly, painted surface sampling and drinking water sampling should be completed as soon as possible if blood lead levels are elevated and no other source is identified via soil and dust sampling. Temporal variation in LBP is not expected unless the painted area is disturbed or removed. Lead may accumulate as water sits in pipes for longer periods, such as overnight, so tap water samples should be collected as first morning draws to best capture the influence of lead plumbing/solder.

Specifying the scale for decision making. For the residential RMAP properties, the sampling unit extent should be specified as the maximum area for decision-making to ensure that any location where arsenic, lead, or mercury concentrations are above health-protective action levels is remediated. Some properties may have more than one sampling unit for one or more exposure media. By setting the decision unit equal to the sampling unit, decisions to remediate can be made for subareas of a property, rather than on a property-wide basis, and any subarea with analyte concentrations above action levels can be addressed even if property-wide removal is not warranted. For example, the interior living space of a property may require remediation (if dust concentrations exceed action levels), but the non-living spaces may not require remediation if a pathway is not identified. Similarly, if constituents are present above action levels in only a portion of a residential yard, the entire yard (as well as interior living spaces) may not require remediation. LBP remediation would only be necessary for surfaces where LBP is detected and inspections determine that the LBP condition is fair or poor and therefore represents a hazard (see Table 2 and the *Revised Final Multi-Pathway RMAP Plan* [BSB & Atlantic Richfield Company, 2022]). Any required remedial actions will be discussed with the Agencies on a case-by-case basis to determine the appropriate remedial timeline. Setting the decision unit equal to the sampling unit also enables remediation decisions to be made separately for each exposure medium (e.g., yard or basement soil, indoor/attic/crawl space dust, indoor air, paint, or tap water).

Step 5: Develop the Analytic Approach – *The purpose of this step is to define the parameters of interest and integrate any previous DQO inputs into a single statement that describes a logical basis for choosing among alternative actions.*

Identification of the population parameters most relevant for making inferences and conclusions on the target population. Arsenic, lead, and mercury concentrations should be measured for each sampling unit as determined by analysis of each corresponding soil, dust, air (mercury vapor only), drinking water (lead only), or paint (lead only) sample collected. The true arithmetic mean concentration for each sampling unit is the population parameter of interest for decision-making, except for LBP, for which the presence or absence of lead is of interest. The average concentration (or presence/absence of LBP) measured in each sampling unit should provide a reliable estimate of the true arithmetic mean concentration for the sampling unit and is

the population parameter that should be used to make inferences and conclusions for each decision unit (i.e., the decision unit should be set equal to the sampling unit to support health-protective decision-making). The unvalidated Level 2 data will be used for initial decision rules to expedite work.

Specifying the theoretical decision rule. The theoretical decision rule is as follows:

- If the analyte concentration measured in exterior soil in the sampling unit (i.e., the average concentration within each decision unit for either arsenic, lead, or mercury) exceeds the appropriate Residential Action Level detailed in Table 1, then the soil from the corresponding sampling area will be remediated following the process described in the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022).
- If sample results show that the mercury concentration in living space dust or earthen basement soil in the sampling unit (i.e., the average mercury concentration within the decision unit) exceeds the mercury action level (Table 1), interior air will be sampled for mercury vapor.
- If sample results show that the analyte concentration in living space dust in the sampling unit (i.e., the average concentration within the decision unit for either arsenic, lead, or mercury) exceeds either the arsenic, lead, or mercury action levels (Table 1), or that the mercury vapor concentration in indoor air in the sampling unit (i.e., the average mercury within the decision unit) exceeds the mercury vapor action level (Table 1), living space floors will be thoroughly cleaned with a remediation grade/high efficiency particulate air (HEPA) filter vacuum or carpets will be removed and replaced. Non-living spaces will also be cleaned according to the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022) if sample results show that the average concentration within the decision unit for either arsenic, lead, or mercury exceeds an action level and there is either a pathway allowing dust into the living space or the property owner is planning a remodel that will disturb the non-living space dust.
- If soil sample results from earthen basements (i.e., the average concentration within the decision unit for either arsenic, lead, or mercury) exceed action levels, the soil will be capped or enclosed to limit access as appropriate for the space as determined by EPA in consultation with Montana DEQ and BSB.
- If EBL levels exist and no other potential source of lead is discovered during the residential investigation, painted surfaces will be sampled and analyzed for the presence of lead pending the results of the on-site investigation. Drinking water will also be sampled and analyzed for the presence of lead. Additional actions to be taken in the event that LBP or lead in drinking water are detected are specified in the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022).

Step 6: Specify Performance or Acceptance Criteria – *The purpose of this step is to identify baseline conditions, limits, and ranges for decisions and consequences of decision errors.*

The primary decision question identified in Step 2 is *Are concentrations of arsenic, lead and/or mercury in exterior/earthen basement soil and/or interior living space/attic/crawl space dust at*

residential properties present at levels that may pose a risk to human health (e.g., above the action levels)? The same decision question conditionally applies to mercury vapor concentrations in indoor air, the presence of LBP, and lead concentrations in tap water, if applicable, based on the outcome of the primary decision question. In this case, the baseline condition for each decision unit is that the analyte concentration in soil, dust, indoor air (mercury vapor only), or tap water (lead only) is above the action level, or that LBP is present. The alternative condition is that there is not an exceedance or that LBP is not present. Because this is a decision question, the possibility of decision error exists due to variability and uncertainty in the data. Potential decision errors include Type I (false rejection of the baseline condition) and Type II (false acceptance of the baseline condition) errors. In the context of the RMAP residential sampling decision question, a Type I error would mean determining that the arsenic, lead, or mercury concentration in soil, dust, or indoor air (mercury vapor only) is below the action level when in fact it is above the action level. Consequences of this type of error include leaving soil in place that contains a metal at concentrations above the action level, resulting in a potential risk to human health. A Type II error would mean concluding that the arsenic, lead, or mercury concentration is above the action level when it is actually below the action level. Consequences of this type of error include unnecessary RA and increased costs.

Because the goal of the RMAP is to protect human health, the tolerance for making a Type I error is lower than the tolerance for making a Type II error. Therefore, a sampling design and analysis method that minimizes the possibility of Type I decision errors should be selected. Due to the potential for work to occur over more than one season and the need to make decisions on a property-by-property basis, the experiment-wise error rate will likely be difficult to assess, and efforts should be made to reduce the Type I error rate at the decision unit (DU), rather than at the project-wide level.

When discrete sampling methods are used and the resulting population of sample data representing each DU is compared to a standard using hypothesis testing, the chance of making a Type I error can be reduced by setting a lower significance level (α) (i.e., a lower Type I error rate). The chance of making a Type II error is reduced by setting a higher statistical power (β). The significance level and power can be raised or lowered to control the probability of each type of error depending on the tolerance for each. With this type of approach, there is a set tolerance for reaching a conclusion (the action level is or is not exceeded) that is correct for most, but not all, values in a population. Typically, the probability of a Type I error is lower than that of a Type II error; for example, a significance level of 5% (0.05 probability of a Type I error) and a power of 80% (0.2 probability of a Type II error) are often selected. It can be difficult to obtain the sample size needed to achieve a much higher statistical power due to limitations such as the area available for sampling and associated analytical costs.

For the residential RMAP program, the tolerance for Type I decision errors is lower than that for Type II errors. Instead of addressing the decision question through hypothesis testing or estimating an upper confidence limit on the mean concentration using a population of discrete samples collected across a non-residential property (i.e., setting the entire property as the decision unit), the size of the decision unit can be reduced to maximize the possibility of finding an exceedance where present (i.e., to decrease the Type I error rate). If each sample result is compared individually to the action level, this reduces the chance for concluding that the average

contaminant of interest (COI) concentration in the decision unit is below the action level when it is not.

A composite sampling design would best support the goal of reducing Type I error potential by limiting the size of the decision unit to the extent of the sampling unit. The EPA (2003) handbook states that "*the overall goals of the sampling effort are to estimate an average soil concentration for risk assessment purposes and to provide information to determine the scope of required cleanup actions."* The same goal applies to dust/indoor air. The composite sampling method is intended to better approximate potential average exposure to a receptor as they move across an area rather than remaining at a single spatial point, which is less likely to occur. Therefore, collecting a composite sample to estimate the average concentration of each analyte in the applicable exposure medium across the extent of each sampling unit is a preferable approach compared with collecting a discrete sample from one location within each area.

Lead-based paint sample collection should include performing XRF analyses of paint at multiple locations to ensure each painted surface is represented. The specific number and location of XRF measurements should be performed according to the requirements specified in Chapter 7 of the U.S. Department of Housing and Urban Development (HUD) *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing* (HUD, 2012) and RAMP SOP XRF-001 located in Attachment B-4.

To minimize the potential for Type I decision errors for tap water, samples should be intentionally collected during the time frame with the potential for the highest lead concentrations – i.e., as the first draw in the morning, before any pipe flushing has occurred. Although the timing of the water sample collection may require reliance upon the homeowner to collect the sample, provided that they are given detailed instructions on sampling procedures, this should not affect data quality.

In addition to lowering the possibility of Type I errors, study error should be minimized through properly training the field sampling team, sample documentation and handling, the use of appropriate analytical methods that achieve method detection limits below the action levels, analysis of field and analytical QC samples, analysis of precision, accuracy, and other measurement performance criteria (described in detail in Section 2.7.2), and data validation. Decisions should be made using data that meet the performance and acceptance criteria; if these criteria are not met, corrective action steps should be taken.

Step 7: Develop the Plan for Obtaining Data – *The purpose of this step is to develop an optimized plan to complete the task.*

Selecting the sampling design. The data collection scheme is designed to ensure that the information will be of sufficient quality and quantity to determine the component(s) of individual residential properties requiring RA (and the depth to which RA is required for exterior soil). The information and outputs generated in Steps 1 through 6 of the DQO process informed selection of the optimized approach for soil, dust, and, where relevant, indoor air, paint, and tap water sampling and analysis at residential RMAP properties described in this final step of the process.

The RMAP sampling plan generally follows EPA's *Superfund Lead-Contaminated Residential Sites Handbook* (EPA, 2003) composite sampling design (with one composite collected per yard component or interior space representing an exposure area that would be remediated). For this reason and because this approach supports the goals of obtaining average concentrations of arsenic, lead, and mercury across each sampling unit and minimizing the potential for Type I decision errors, the residential program is designed to also rely on composites that reflect portions of exterior exposure areas or entire interior exposure areas. Arsenic, lead, and mercury concentrations in exterior/earthen basement soil, interior/attic/crawl space dust, and indoor air (where applicable) will be determined through composite samples collected from residential RMAP properties (single- and multi-family residences, residential daycare/preschool facilities, and residential living quarters within a commercial property). The goal of composite sample collection and analysis is to obtain a reliable estimate of the average concentration of a COC in the sampled medium over a specified area where exposure may occur for comparison to the appropriate action level for that area.

LBP paint sampling, where applicable, will be completed following the processes described in the HUD guidelines (HUD, 2012). Additionally, where applicable, tap water sampling will be conducted to quantify lead concentrations during first draw, which is the time frame when the potential for lead levels is highest, at the faucet most likely used as a primary drinking water source.

For each property, the extent of the sampling unit will be defined based on the recommendations for residential land use described in Step 4. For consistency with the RMAP and with EPA guidance, the same information used to determine appropriate sampling unit extents for residential properties (EPA's lead handbook and previous RMAP sampling) also informs determination of subsample counts recommended for each sampling unit. Details of the extent and number of subsamples to be collected from each area of a residential property are provided in Table 1 and in Section 3.1. Exterior soil sampling will be conducted at multiple depth intervals (0 to 2 inches, 2 to 6 inches, and 6 to 12 inches) for all residential properties. Flower/vegetable garden components will be sampled at additional depth intervals of 12 to 18 inches and 18 to 24 inches.

Consistent with prior sampling programs, soil samples will be sieved to the less than 250 µm fraction, reflecting the fine fraction of soil most likely to adhere to children's hands. More recent EPA guidance (EPA OLEM Directive 9200.1-128) requires sieving to less than 150 µm based on studies that show lead enrichment in very fine soil fractions (e.g., less than 63 µm). Atlantic Richfield has performed a sieve study to compare the 250 µm to 150 µm results on 129 site-specific soil samples collected in 2021 and provided a final report of the results (Atlantic Richfield Company, 2022a). Since submission and review, a verbal response and direction has been issued by EPA, and the 2023 RMAP site samples will continue to be sieved to 250 µm.

Based on the assessment of the limitations and benefits of sample analysis options completed in Step 3, laboratory analysis was identified as the preferred approach for measurement of arsenic, lead, and mercury concentrations in composite soil and dust samples. Arsenic and lead concentrations will be determined per EPA Method 6010 (inductively coupled plasma atomic emission spectroscopy [ICP-AES]) or EPA Method 6020B (inductively coupled plasma mass

spectrometry [ICP-MS]). Mercury concentrations will be determined per EPA Method 7471B (Manual Cold-Vapor Technique). The detection limits associated with these methods are expected to be well below the applicable action levels (see Table 1).

Real time mercury vapor data will be collected by a Mercury Tracker 3000 unit (or Agency approved equivalent). Paint samples will be analyzed per the manufacturer user manual, Method 6200, as applicable, and the RMAP SOP XRF-001 in Attachment B-3 for the XRF unit to determine concentrations of lead. Drinking water samples will be analyzed per EPA 200.8 to determine concentrations of total recoverable lead.

Decision units will be set equal to the sampling unit. The relationship between the average COC concentration and the action level provides the input needed to resolve the decision statements outlined in Step 2 to determine whether abatement is required for residential RMAP soil and interior dust and whether drinking water and painted surface sampling are needed when EBL levels exist. For each decision unit, the decision question (*Are concentrations of arsenic, lead and/or mercury at residential properties present at levels that may pose a risk to human health (e.g., above the action levels)?*) will be addressed by comparing the composite soil sample result from each sampled depth interval and the interior dust and, where applicable, air (mercury vapor only) and tap water (lead only) sample result from each sampling unit to the corresponding action level. Where sampling for LBP is completed, the decision question will be addressed by determining the presence or absence of LBP and further defined by inspections to determine the condition of the LBP (see Table 2).

Details on how the design should be implemented together with contingency plans for unexpected events. Sampling will be implemented per the media-specific guidelines provided in Sections 3.1 through 3.4. Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out-of-QC performance, which can affect data quality. Corrective action can occur during field activities, laboratory analysis, and data assessment. Corrective action procedures are outlined in Sections 4.1 and 4.2. Any unexpected/unplanned events not specifically addressed by this QAPP will be discussed with Agency personnel and addressed through forthcoming QAPP revisions.

Specifying the Quality Assurance and Quality Control procedures. Sufficient data quality will be achieved through the field and laboratory quality control measures (Sections 3.8 and 3.10, respectively) including the use of appropriate sample collection, handling, chain of custody procedures, and laboratory analytical methods, QC sample analysis (field and laboratory), assessment of the performance criteria described in Section 2.7.2, following the corrective action procedures detailed in Sections 4.1 and 4.2, and analytical data validation (Section 5.0).

2.7.2 Measurement Performance Criteria for Data

Measurement performance criteria are established by defining acceptance criteria and quantitative or qualitative goals (e.g., control limits) for precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) of measurement data. The definitions of precision, accuracy, representativeness, comparability, completeness, and sensitivity are provided below. Acceptance limits are detailed in Section 3.7.7 for each measurement

performance criteria. Equations for calculation of precision, accuracy, and completeness are provided in Table 3. Additional QC acceptance criteria are provided in Table 4.

Precision
Precision is the amount of scatter or variance that occurs in repeated measurements of a particular analyte. Precision is assessed using the relative percent difference (RPD) between a primary sample result and its paired field or laboratory duplicate sample result (for field and laboratory precision, respectively). For example, perfect precision would be a 0% RPD between the primary sample result and its paired field or laboratory duplicate sample result (both samples have the same analytical result). For these sampling events, precision will be assessed based on laboratory prepared and field duplicate sample analysis.

Accuracy/Bias
Accuracy is the ability of the analytical procedure to determine the actual or known quantity of a particular substance in a sample. Accuracy is assessed based on the percent recovery (%R) and percent difference (%D) of various laboratory QC samples. Perfect %R is 100% and perfect %D is 0% (the analysis result is exactly the known concentration of the QC sample). The laboratory control sample (LCS) and laboratory matrix spike (LMS) are used to measure accuracy, based on the % R of the LMS and LCS. Additional laboratory QC samples may be used to assess accuracy as appropriate to the analytical method.

Bias is the systematic or persistent distortion of a measurement process that causes error in one direction (e.g., consistently higher or lower than the true concentration). As with accuracy, analytical bias can also be assessed based on %R of laboratory QC samples. In addition, laboratory blanks, ICP serial dilutions, interference check standards, tunes, and internal standards will also be used to assess accuracy as appropriate during data validation. Sampling bias is addressed through the use of proper sampling design and methods.

Representativeness
Representativeness is the degree to which sample data represent a characteristic of a population, parameter, or environmental condition. Representativeness is a qualitative parameter that is most concerned with proper design of the sampling and analytical schemes. Representativeness is achieved by determining the number and locations of samples and the appropriate sampling techniques needed to depict, as accurately and precisely as necessary, the conditions being measured. Representativeness deals with protocols for sample storage, preservation, and transportation; analyzing samples with appropriate methods, techniques, and instrumentation; and using the methods to document these protocols. Representativeness will be achieved through judicious selection of sampling locations and methods. This QAPP requires that samples are representative of the medium being sampled and that there are a sufficient number of samples to meet the project DQOs and satisfy the project RA design elements.

Comparability
Data comparability is defined as the measure of the confidence with which one data set can be compared with another. Comparability is a qualitative parameter but must be considered in the design of the sampling plan and selection of analytical methods, QC protocols, and data reporting requirements. Comparability will be ensured by analyzing samples obtained according

to this QAPP, applicable laboratory SOPs, and the Program SOPs, which are comparable to the sampling methods used during previous investigations within the BPSOU (Attachment B contains various field and laboratory SOPs). All data will be reported in units consistent with standard reporting procedures so that the results of the analysis can be compared with results from previous investigations. Soil data will be reported in units of mg/kg, water samples will be reported in units of micrograms per Liter (µg/L), and indoor air samples will be reported in $\mu g/m^3$.

Completeness

Completeness is a measure of the amount of valid data obtained from the measurement system. Proposed sample collection points may fail to produce usable data for many reasons (e.g., non-traceable sample identification, sample container breakage, elevated storage temperature, exceeded sample holding time, or data loss). When samples are analyzed, but the data are rejected, the numerator of this calculation becomes the number of valid results minus the number of possible results rejected. Valid data are data not rejected (deemed usable during the data validation process). Completeness describes the amount of valid data that meets the DQOs for representativeness, accuracy, and precision versus the amount of data obtained or considered necessary to achieve a specific level of confidence in decision-making. For relatively clean, homogeneous matrices, data would be expected to be 100% complete. However, as matrix complexity and sample heterogeneity increase, completeness may decrease. Based on the complexity of sample matrices anticipated to be collected from the project sites, the analytical data completeness goal following validation is stated to be greater than or equal to 90% and will be generated on a Sample Delivery Group (SDG) basis.

Project completeness regarding the collection of samples and identified data gaps will be addressed by the data generators and users. A goal of 90% is anticipated for each project location.

In order to more accurately depict the percent analytical completeness, individual analyte completeness will be calculated and reported. In addition to the analyte percent completeness, a summary of completeness for each fraction will be provided in the validation reports. If reanalysis is performed by the laboratory, only a single analytical set (may be a mixture of original and reanalysis data based on usability) will be included in the analytical completeness calculation to prevent double-counting data. Valid results used to meet completeness objectives are those results that provide a defensible estimate of the true concentration of an analyte in a sample. These valid results include data that are not qualified and data that are qualified but that can still be used to meet project objectives. Invalid data are those results for which there is an indication that the prescribed sampling or analytical protocol was not followed or results did not meet QC specifications.

Sensitivity

Sensitivity is related to the ability to compare analytical results with project-specific action levels. Analytical quantitation limits for the sample analytes should be below the level of interest to allow an effective comparison.

Achieving proper sensitivity (i.e., reporting limits) will depend on instrument sensitivity and potential matrix effects. Data sensitivity is the ability of the analytical method to differentiate the target analyte from instrument "noise." With regard to instrument sensitivity, it is important to monitor the instrument performance to ensure its performance is consistent at the low end of the calibration range. Instrument sensitivity will be monitored through analysis of method blanks and calibration check samples. Project data will be reported to the method detection limit (MDL) with variations due to sample amount digested, potential dilutions, and percent moisture correction for mercury analysis. The MDLs are below the soil action limits defined in the DQO steps above.

Additional details regarding bias, sensitivity, and QC acceptance criteria are included in Section 3.7.7.

The method sensitivity for laboratory analysis is determined as part of the laboratory's SOPs. A review of these detection limits will be conducted as part of the data validation process.

2.8 Special Training

All RMAP field personnel will review the requirements of this QAPP and receive training on Program-related tasks during a project meeting held before fieldwork begins. A review of sampling procedures and requirements will be completed prior to field activities to ensure sample collection and handling methods are conducted according to QAPP requirements. Field personnel will be trained in proper use of field equipment, sample collection tools, etc., and procedures according to field data collection SOPs (Attachment B-1) and methods described in the Program. Field personnel performing sampling activities or members who can potentially contact contaminated materials should receive Hazardous Waste Operations and Emergency Response (HAZWOPER) training.

The BSB Department of Reclamation and Environmental Services Director is responsible for ensuring field personnel receive appropriate training and will maintain up-to-date training records and/or certifications. The BSB Department of Reclamation and Environmental Services Human Health/RMAP Division Manager will ensure that each member of the sampling team obtains and is familiar with the most recent version of the QAPP, will maintain signatures of each team member who has read the QAPP (including reviews and addenda, as necessary), and make sure each team member has been trained in the appropriate sample collection methods per the Program. The Human Health/RMAP Division Manager will review the SSHASP with all field personnel prior to fieldwork to assess specific site hazards and the control measurements that have been put in place to mitigate these hazards. The SSHASP review will also cover all other safety aspects of the site including site personnel responsibilities and contact information, additional site-specific safety requirements and procedures, and the emergency response plan. One hard copy of the approved version of this QAPP will be maintained for reference in the field vehicle and/or field office. All field team personnel will have access to Portable Document Format (pdf) files of the complete QAPP.

2.9 Documents and Records

This section describes procedures for documentation management and record keeping for this QAPP from initial record generation through final data formatting and storage. All sampling data collected for all media under the Program, including yard soil, attic/crawl space dust, indoor dust, basement soil, vapor, paint, and water are housed within the Program database. The Program database is housed in an Access Structured Query Language (SQL) server database and maintained by BSB. Document backups are contained in the BPSOU Document SharePoint and EPA document repository. Refer to the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, 2022b) for additional details regarding data management, backup, and storage. Atlantic Richfield and BSB will coordinate Agency testing of the database with the program architects and primary users in a manner to minimize provision of written comment and the potential misinterpretation of those comments.

A Field Sampling Plan (FSP) will be developed for each residential property prior to conducting soil sampling. These plans will consist of a plan view figure showing approximate property boundaries, non-sample areas (structures, sidewalks, etc.), and those areas/components slated for sampling work. For attics and basements, the FSP will consist of a table and a brief narrative detailing the proposed sampling work.

2.9.1 Field Documentation

Field documentation provides a description of site conditions during sampling activities and provides a permanent record of all field activities. Field documentation will primarily be achieved through electronic means (i.e., field tablets). Field documentation includes a sample location map of the site that shows property boundaries, house, garage, structures, driveways, contaminant source material, gardens, lawns, and patios. Any hard copy field data may be converted to electronic storage media. Field personnel creating the sample location map will delineate yard features with an accuracy of approximately plus or minus 2.0 feet. Each yard will be divided into polygons (e.g., east yard, west yard, etc.) for sampling, and these areas will be identified on the map.

Documentation for each site will include the information listed below, at a minimum:

- A description of the field task.
- Time and date fieldwork started.
- Location and description of the work area, including sketches if possible, map references, and references to photographs collected.
- Names and titles of field personnel.
- Name, address, and phone number of any field contacts or site visitors (e.g., Agency representatives, auditors, etc.).
- Details of the fieldwork performed with special attention noted to any deviation from the QAPP or applicable field SOPs. Such deviations will be brought to the attention of and discussed with Agency field oversight personnel. If the deviations are deemed to be

minor by the Agency representative, a resolution and path forward will be determined in the field. If the Agency representative determines that the deviation is major in scope, it will be his/her responsibility to elevate the question internally and to receive Agency direction.

- All field measurements made (e.g., minor field modifications to sampling polygons, delineation of additional sampling polygons, etc.).
- Personnel and equipment decontamination procedures.

For any field sampling work, the field documentation will include all applicable items from the Level A/B Assessment Checklist (see Section 5.1.2 and Attachment D). At a minimum this includes documentation of the following:

- Sample team and/or leader.
- Sample location, depth, and traceable sample designation number.
- Sample type collected.
- Date and time of sample collection.
- Samples taken by other parties (note the type of sample, sample location, time/date, sampler's name, sampler's company, and any other pertinent information).
- Sampling method, particularly any deviations from the field SOPs (Attachment B).
- Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample (if any used in the field), specifically if sample bottles/preservatives are not provided by the laboratory and certified as cleaned.
- Collection of field duplicates.
- Decontamination of sampling equipment.
- Sample custody documentation.
- Sample preservation (if used).

Sufficient information should be recorded to allow the sampling event to be reconstructed without needing to rely on the sampler's memory.

Data recorded for all LBP sampling will include the following:

- Date.
- Legal address.
- XRF unit serial number.
- XRF unit calibration data and time.
- Property owner name and mailing address.
- Personnel conducting risk assessment.
- Sample location (room, wall, interior, exterior).
- Component sampled.

- Substrate (wood, metal, concrete).
- Paint condition.
- Paint color.
- Sample result.
- QC calibration check results.
- QC blank results.
- Replicate sample result.

Data recorded for all mercury vapor sampling will include the following:

- Date.
- Legal address.
- Rental sampling unit serial number.
- Information on rental sampling unit provider (who will calibrate the device prior to use).
- Property owner name and mailing address.
- Personnel conducting sampling.
- Sample locations.
- Volume of air evaluated.
- Sampling durations.
- Sample results (accomplished through sampling unit's internal data logger).

A report containing all the above-listed information will be provided to the property owner and the information recorded in the Program database and tracking system and uploaded to cloud-based databases managed by BSB as detailed in the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, 2022b).

2.9.2 Field Photographs

Field personnel will use a digital camera to take photographs at the site. Photographs may be taken of sampling locations, field activities, and to document site conditions, as necessary. Photographs should include a scale in the picture when practical. Documentation of all photographs taken during sampling activities will be recorded in a bound field logbook or appropriate field collection device and will specifically include the following for each photograph taken:

- The date, time, and site identification.
- A brief description of the subject and the fieldwork portrayed in the picture.
- Sequential number of the photograph.

Electronic files will be placed in project files with copies of supporting documentation from the bound field logbooks/data collection device.

2.9.3 Chain of Custody Records

Each sample collected will be assigned a unique sample number, and the sample container will be labeled with sample designation number, date, and time of collection, and requested analysis. Then the information will be recorded in the field documentation. Chain of custody records ensure that samples are traceable from the time of collection until final disposition. After samples have been collected, they will be maintained under strict chain of custody protocols according to SOPs (Attachment B). A chain of custody record will be initiated by the individual physically in charge of the sample collection. The chain of custody form may be completed concurrently with the field sampling or before shipping or hand delivery of samples to the laboratory. The sampler is personally responsible for the care and custody of the samples until they are shipped or hand delivered to the laboratory. When transferring the sample possession, the individual relinquishing and receiving the sample will sign and record the date and time of day on the chain of custody record.

A copy of each as-transmitted chain of custody form will be scanned and stored on a hard drive. Chain of custody records will also be copied to the project record files (refer to Section 3.12).

2.9.4 Analytical Laboratory Records

Results received from the laboratories will be documented both in report form and in an electronic format. Laboratory documentation includes laboratory confirmation reports such as information on how samples have been batched, the analysis requested, data packages containing the laboratory report and the electronic data deliverable (EDD), and any change requests or corrective action requests. Section 5.1.3 lists the laboratory reporting requirements in detail. The deliverable (data package or report) issued by the laboratory must include data necessary to complete validation of laboratory results. Original reports and electronic files received from laboratories will be maintained with the project quality records. Refer to the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, 2022b) for additional requirements.

2.9.5 Project Data Reports

Upon receipt of laboratory results and completion of the data review/validation process, all analytical data will be uploaded into a project database and submitted to the Agencies for review and approval as part of the annual DSR submittal. This DSR will include figures displaying the location of homes sampled, analytical results, and copies of all field data. As described above, all sampling data will reside in the Project records.

Sampling for remedial design/RA under the RMAP will be documented through annual DSRs submitted for review and approval by the Agencies. Sample data, with their laboratory and data usability qualifiers, will be maintained electronically by BSB/Atlantic Richfield and reported in the annual report. The annual report will be a DSR prepared based on the guidelines in *Clark Fork River Superfund Site Investigations* (CFRSSI) *Pilot Data Report Addendum* (AERL, 2000a) following each year of data collection. The annual report will describe the sampling activities for the year, provide a summary of the data obtained, discuss the results of data validation, and

provide a detailed listing of any deviations from the QAPP. The DSR will also include a data usability assessment for laboratory data. The data usability assessment has a data summary table with all the samples and analyte concentrations listed, along with the laboratory- and data validation-assigned qualifiers and data usability codes (i.e., enforcement, screening, unusable). The Level A/B checklists, laboratory data validation checklists, and data validation summary will provide an overall assessment of the quality and usability of the data. Furthermore, the DSR will also contain copies of all analytical reports, EDDs, and data validation reports. Annual DSRs will be submitted to the Agencies for review approximately along with the annual Construction Completion Report (CCR) which is due annually by the end of February. The annual CCR encompasses properties sampled, remediations performed, individual site work plans, project photographs, SOPs, sample request forms, and access agreements for environmental assessments performed that year. Biomonitoring services, blood lead screening, and case narratives are also included in the report.

2.9.6 Quality Records

Quality records are defined as completed, legible documents that furnish objective evidence of the quality of items or services, activities affecting quality, or the completeness of data. These records will be organized and managed by the BSB Department of Reclamation and Environmental Services Data Management Division Manager/QA Manager (or designee) in cooperation with the BSB Department of Reclamation and Environmental Services Director and will include the following at a minimum:

- This QAPP and any approved revisions or addenda.
- Approved versions of the SSHASP and any addenda.
- Copies of field SOPs for field data collection, with any updates, revisions, or addenda to those SOPs.
- Incoming and outgoing project correspondence (letters, telephone conversation records, and faxes).
- Copies of completed access agreements for the individual properties sampled.
- Individual property maps, including any field drawings and field photographs.
- Field documentation forms.
- Copies of all field documentation/records.
- Copies of all chain of custody forms for samples.
- Copies of all laboratory agreements and amendments.
- Laboratory data packages (printed report and electronic version).
- Documentation of field and/or laboratory audit findings and any corrective actions.
- Draft and final delivered versions of all reports and supporting procedures such as statistical analysis, numerical models, etc.

All project data will be maintained indefinitely in the RMAP Project database. The database has not yet been completely developed, and Atlantic Richfield and BSB will be working with the

Agencies to finalize the database. This is a long-term project with access to the database provided to many interested parties. Any addendums or revisions to this QAPP will be electronically distributed to all parties identified on the distribution list.

3.0 MEASUREMENT AND DATA ACQUISITION

This section addresses all aspects of project design and implementation for generating and acquiring data. Adhering to the procedures provided in Attachment B in this QAPP and described in this section ensures that the appropriate methods for sampling, sample handling, laboratory analysis, field and laboratory QC, instrument/equipment testing, inspection, maintenance, instrument/equipment calibration, data management, and data security are followed. Laboratory SOPs contained within Attachment B have been developed for multiple clients and projects. In the case of any discrepancy between the QAPP text and laboratory SOPs, the language within the QAPP text shall take precedence.

3.1 RMAP Soil Sampling (Residential Parcels)

All RMAP soil sampling work on residential parcels will be conducted as described below to determine the presence of the COCs listed in Table 1. Field personnel will follow the procedures in the RMAP-SOP-1 (Attachment B-1) and will record all information in the field logbook/data collection device. The procedures for RMAP soil sampling are summarized below.

3.1.1 Residential Yard Soil Sampling

The number of residential yards ultimately sampled will depend on the number of sample requests secured by BSB.

Residential daycare/preschool facilities and yards of multi-family residences will be subject to the same sampling procedures applied to single-family residential yards. Likewise, residential living quarters within a commercial property will be subject to the same sampling procedures applied to residential yards.

Sample Density, Location, and Compositing

Sample locations within yard components will be determined by sampling personnel based upon site-specific conditions and the size of the property. Either the property boundary or a smaller natural boundary within the yard/lot will be used to establish the extent of the sample area. The *yard area* is defined as a maximum of 125 feet from the exterior of the residence, unless a property boundary or natural barrier (e.g., fence, hedge, tree line, abrupt change in grade, etc.) is encountered at a distance less than 125 feet. It is anticipated that the property boundaries will define the sampling area for in-town residential parcels. The 125-foot definition is anticipated to be used predominantly on regional/out of town residential yards. In general, RMAP soil sampling stops at 125 feet from the exterior of the residence; thus, in more rural settings, if driveways or other property features extend beyond 125 feet, RMAP soil sampling would not extend beyond this distance. The 125-foot distance is considered a guideline and can be adjusted in the field by Agency personnel, as appropriate.

Standard Residential Lots (< 5,000 square feet)

For standard residential lot (less than 5,000 square feet) sampling components, subsamples will be collected from a minimum of three subsample locations or at a rate of 5 subsamples per 625 square feet (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 1,200 square feet (meaning a maximum of 10 subsamples will be collected from any standard residential lot sampling component) (see Table 1).

Mid-Sized Residential Lots (> 5,000 square feet and < 1 acre)

For mid-sized residential lot (greater than 5,000 square feet and less than 1 acre) sampling components, subsamples will be collected from a minimum of three subsample locations or at a rate of 1 subsample per 625 square feet (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 6,250 square feet (meaning a maximum of 10 subsamples will be collected from any mid-sized residential lot sampling component) (see Table 1).

Large Residential Lots (> 1 acre)

For large residential lot (greater than 1 acre) sampling components, subsamples will be collected from a minimum of three subsample locations or at a rate of 1 subsample per 2,200 square feet in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field, and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 10,890 square feet (meaning a maximum of five subsamples will be collected from any large residential lot sampling component) (see Table 1).

In order to limit disturbance in small components (such as vegetable and flower gardens), only one sample location will be used when the component area is approximately 50 square feet or less in area. For garden sampling components greater than 50 square feet in area, subsamples will be collected from a minimum of two subsample locations or at a rate of 1 subsample per 125 square feet in surface area per sampling component, whichever is greater. When applicable, subsamples from these locations will be composited in the field and a single composite sample per depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 1,200 square feet, meaning a maximum of 10 subsamples will be collected from any garden sampling component (refer to Table 1).

Samples will be thoroughly mixed in a clean 1-gallon resealable plastic bag or stainless-steel bowl. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Sample mass will consist of approximately 500 to 800 grams of material. Samples will be submitted to the laboratory by the samplers under chain of custody procedures.

Soil samples for mercury analysis for this project will be collected in four-ounce amber glass containers provided by the laboratory by removing a subsample aliquot from the homogenized sample contained in the resealable bag during the sample collection process. This process helps to ensure homogenization and representativeness; the aliquots for the mercury subsample will be obtained from several areas of the homogenized sample bag using a clean scoop. This process helps to ensure sample representativeness between the sample aliquots. Additional homogenization of the sample through labor intensive splitter or cone and quartering techniques may lead to volatilization of the mercury. According to *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, US EPA Publication SW 846* (EPA, 2015), the sample jars for mercury analysis will be shipped on ice from the field to the primary laboratory.

The project soil samples, collected in resealable plastic bags for arsenic and lead, will be shipped from the field and stored by the laboratory at ambient temperature conditions.

If the Agency representative or property owner chooses to collect split samples, an adequate quantity of soil will be made available by the sampler at the time of sample collection. However, the Agency representative or property owner will be responsible for providing sample containers and coolers, etc.

Sample Depths

Within residential properties, three depth samples will be collected from each identified yard component. There will be one surface sample (0 to 2 inches below ground surface [bgs]) and two subsurface samples (2 to 6 inches bgs and 6 to 12 inches bgs).

Because most residential yard/lot areas are expected to be covered with grass, the surface sample will be collected immediately beneath the vegetative mat (sod), or in the absence of vegetation, 0 to 2 inches bgs. If a vegetative mat is present, it will be separated from the soil surface with a stainless-steel knife or equivalent. The removed vegetative mat will be shaken and scraped over the sample collection container to dislodge any mineral soil particles. All dislodged soil particles will be included in the composite sample.

Exceptions to this procedure will occur when the sample location falls on a graveled driveway or similar surface. If the surface material is coarse-grained and free of intermixed materials, the sample will be collected from the 0- to 2-inch soil layer immediately beneath the coarse material. However, if the graveled driveway or similar surface contains fine soil/dust material on the surface, the sample will be collected from the surface (0- to 2-inch) layer.

Gardens will be subject to additional subsurface sampling. In addition to the three depth samples described above, two additional subsurface samples will be collected from the 12- to 18-inch and

18- to 24-inch depth intervals, for a total of five depth samples within a vegetable or flower garden.

The decision to collect additional "opportunistic" samples will be made in the field by the sampling crew personnel and/or Agency personnel during the time of sampling. Opportunistic samples will be collected of suspect piles, discolored materials, or notable barren areas greater than approximately 25 feet by 25 feet in area. All opportunistic samples collected will be comprised of a minimum of three subsamples.

Previously Sampled Properties

Butte-Silver Bow County will review the Program database to identify properties that were previously sampled only within the 0- to 2-inch depth interval. Property owners of these previously partially sampled properties where remediation was not performed will be contacted to request access to conduct additional sampling to fill the data gaps. Sampling at previously sampled properties will be prioritized over unsampled properties. The goal will be to produce a complete data set that includes data for all required depth intervals and analytes by completing sampling within the 2- to 6-inch and 6- to 12-inch depth intervals. Sampling protocol described previously will be followed for the 2- to 6-inch and 6- to 12-inch depth intervals.

Areas of the residential property that were sampled at the 0- to 2-inch depth interval and remediated will not be resampled because these components have already been remediated to a 12-inch depth.

3.1.2 Earthen Basements

For residential earthen basement sampling components, subsamples will be collected from a minimum of three subsample locations or at a rate of approximately 5 subsamples per 625 square feet (25 feet by 25 feet) in surface area per sampling component, whichever is greater. Subsamples from these locations will be composited in the field and a single composite sample from the 0- to 2-inch depth interval will be analyzed for arsenic, lead, and mercury. Each subsample should have similar mass so that each location is equally represented in the total sample mass. The maximum area represented by a single composite sample will be 1,200 square feet, meaning a maximum of 10 subsamples will be collected from any residential sampling component (refer to Table 1).

Samples from earthen basements will be collected as described in the RMAP-SOP-2 (Attachment B-1). Samplers will follow the general steps below as well as steps listed in previous sections describing how to collect samples from yards (as applicable for earthen basements):

1. Prior to collection, visually inspect the basement for any hazards.
2. Prepare a sample bag. Basement areas are considered a single composite area. Label a sample bag for each composite sample with the appropriate sample identification number.

3. Collect the sample. Samplers will collect the appropriate number of surficial (0- to 2-inch depth) subsamples spread throughout the earthen portion of the basement and combine the subsamples into one composite sample in the respective labeled sample bag.
4. Complete sample chain of custody form.
5. Add all sampling identification information to the Program's database tracking system.
6. Prior to abatement, photograph the basement area to create a record of the areas before and after abatement is performed.

Duplicate field samples will be collected as described in Section 3.8.1.

3.1.3 Soil Sample Collection Process

Field personnel/samplers will record all information in the field logbook/data collection device. The decision to collect additional "opportunistic" samples will be made in the field by the sampling crew personnel and/or Agency personnel during the time of sampling. All RMAP residential soil samples will be shipped to a certified laboratory for analysis. Sampling crew personnel will follow the steps listed below:

1. Ensure that an executed Sample Request form exists prior to beginning any sampling event.
2. Visually inspect the property to determine the number of polygons needed for composite sampling.
3. Take photographs to create a record to document the pre-sampling condition of all portions of the property scheduled to be sampled (e.g., east, west, south, or north yards). At the end of the project, a copy of the record is provided to the owner (and occupant). Copies will also be made available for review by the Agencies.
4. Create a scaled sample location map of each yard or lot that shows property boundaries, house, garage, structures, driveways, contaminant source material, gardens, lawns, and patios. Figure 3, Figure 4, and Figure 5 show example sample location maps. If a map of the area exists (from prior activities), field personnel can use that map as a starting point, verify components, and add or delete components.

   The sample location map will be developed using conventional and representative methods (i.e., computer or tablet devices). Use measuring devices (standard measuring tape or laser measuring devices) to accurately measure yard features within an accuracy of approximately plus or minus 2.0 feet. Divide each yard into polygons (e.g., east yard, west yard) for sampling and identify these areas on the map. The map should include the following at a minimum:

   - Surface area applicable to each individual yard component (e.g., front yard, back yard, earthen driveway, etc.).
   - Number of subsamples required from each yard component (based upon component surface area).

- Surface area applicable to the "occupation" boundary of each residence (approximately corresponding with the property boundary).
- House location.
- Garage location.
- Location of miscellaneous structures (patios, concrete pads, sidewalks, decks, etc.).
- Any noticeably dissimilar soil material types or surface conditions (i.e., bare ground areas, areas where paint chips were observed, locations of obvious imported fill materials, etc.).

All subsample locations will be plotted on the sample location map by sampling crews in the field.

5. For each composite sample, label the bag with the correct sample identification number (see Section 3.6).
6. Collect composite samples as dictated by the Sample Location Map (placing each composite sample in the corresponding bag). The first composite sample will consist of subsamples from the 0- to 2-inch depth interval, the second composite sample will consist of subsamples from the 2- to 6-inch depth interval, and the third composite sample will consist of subsamples from the 6- to 12-inch depth interval. Flower and vegetable garden components will require additional composite samples from the 12- to 18-inch and 18- to 24-inch depth intervals.
7. Follow chain of custody procedures outlined in Attachment B-1.
8. Ensure all sampling identification information is entered into the Program's database tracking system.

Duplicate field samples will be collected as described in Section 3.8.1.

3.1.4 Soil Sample Equipment Decontamination

Reusable equipment will be decontaminated according to the manufacturer's recommendations and RMAP-SOP-DE-02 (Attachment B) before being reused. Procedures for appropriately decontaminating reusable equipment are as follows:

1. Remove excess soil particles from the equipment prior to "gross wash." This may be achieved by using a dedicated stiff brush or other hand tool such as a flat head screwdriver.
2. Remove gross contamination by manually scrubbing the equipment in the 5-gallon bucket of tap water marked *Gross Wash* and a stiff brush (dedicated to the gross wash step).
3. Move the equipment to the 5-gallon bucket marked *Soap Wash*. Wash equipment in solution of tap water and soap (no phosphate, such as Liquinox©) with a stiff brush (dedicated to the soap wash step).
4. Triple rinse the equipment in the 5-gallon bucket with deionized (DI) water marked *DI Rinse* to remove any soap residue.

5. Perform a second triple rinse of the equipment in a bucket with DI water marked *Final Rinse.* Alternatively, a designated pressurized hand spray bottle (i.e., 2-gallon lawn and garden sprayer) with DI water may be used for final rinse stage.
6. Place equipment on plastic sheeting or foil to air dry.
7. Wrap equipment in foil or plastic wrap to transport or store.
8. Clean decontamination equipment:
    a. Triple rinse equipment from the *Gross Wash* and *Soap Wash* (brushes and buckets) with clean tap water, preferably with pressurized water. Soap can be used on particularly dirty equipment.
    b. Triple rinse all decontamination equipment with DI water, including *DI Rinse* and *Final Rinse* buckets.
    c. Store decontamination equipment, labeled and in a clean location so they are used only for decontamination purposes.

Scoops used for sample bagging or subsampling for mercury analysis will be single-use disposable equipment. Decontamination solutions may be disposed on the ground surface in the same general area in which soil sampling occurred. Disposable supplies will be collected by the field team leader and disposed of at the BPSOU Mine Waste Repository or local landfill, as appropriate. Field equipment blanks will be collected on reusable equipment to ensure proper decontamination is being achieved, as described in Section 3.8 below.

3.1.5 Soil Sample Preparation Methods

The temperature upon mercury sample receipt will be measured and recorded by Pace Analytical Services, LLC personnel located in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) on Sample Condition upon Receipt documentation. The samples will be stored chilled (less than or equal to 6 degrees Celsius [°C], but not frozen) in temperature monitored refrigerators prior to laboratory digestion and analysis within 28 days of sample collection. The mercury digestion and analysis will be performed on "wet" sample aliquots and reported on a dry weight basis.

The project soil samples collected in resealable plastic bags for lead and arsenic will be shipped from the field and stored by the Pace Analytical Services, LLC laboratory located in Green Bay, Wisconsin (1241 Bellevue Street, Suite 9, Green Bay, WI 54302) at ambient temperature conditions. The soil samples will undergo sample drying and sieving (within approximately five days of collection) prior to ambient shipment of the dried sample to the Pace Analytical Services, LLC laboratory located in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) for sample digestion and analyses for lead and arsenic.

The soil samples will be analyzed to determine metals concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury. Sample preparations and analyses will be according to the referenced EPA analytical method specifications as well as standard laboratory practices. A portion of the field and laboratory homogenized field sample

(approximately 2 cups of material from the resealable plastic bag) will be used for air-dry and sieve sample preparation prior to digestion.

Laboratory personnel will place the sample onto a tray lined with brown freezer paper. The paper is folded to create a "boat" to contain the sample and prevent loss or potential cross contamination during the drying process. The soil sample is spread across the entire tray surface and pieces greater than ½-inch are broken by hand. New gloves are used between each sample to prevent cross contamination. The trays are placed on racks and into a room temperature closet containing fans and dried overnight. If samples are not completely dry the next day, the samples will be dried for an additional time.

Once dry, the sample trays are removed from the closet and additional disaggregation is performed by hand. Rocks, twigs, and other foreign material are removed and set aside. Disaggregation is defined as a process for loosening the clump soil around rocks. This process is not a grinding process. The soil is further disaggregated by placing a piece of butcher paper (wax side up) on top of the tray and using a 2.2 kg marble rolling pin. The rolling pin is rolled over the dried soil for 1 to 2 minutes in several directions. No downward pressure is applied to the rolling pin. Alternative methods are also suggested such as a rubber mallet as long as no crushing of rocks is performed according to the SOP.

The air-dried soil sample will be sieved at room temperature. The sample will be sieved to 250 µm. The entire portion of 250-µm material will be placed in a resealable plastic bag, sealed, labeled, and transferred to Pace Analytical Services, LLC in Minneapolis. The fine fraction of the sieved soil will be further homogenized in a sealed bag by gently rolling the sample bag on a laboratory bench, so that fine materials less than 250 µm are not segregated. The sample will then be flattened into all sections of the bag thereby creating a slab cake for sample aliquoting for digestion. The bag will be opened and a portion from each of six areas of the bag will be removed and placed in a sample tube to digest approximately 1 gram of material. The sample aliquots will be digested according to modified EPA Method 3050B, and arsenic and lead concentrations will be determined per EPA Method 6010 (ICP-AES) or EPA Method 6020 (ICP-MS).

The remaining coarse fraction will be placed in a new plastic bag labeled with the original sample number, date of sieving, and "Coarse Fraction" and archived along with the remaining fine fraction until the criteria for sample disposal is met (Section 3.9). The weight of the coarse fraction and the fine fraction will be measured and recorded by the laboratory for each soil sample prepared in this manner. Soil sieving details are provided in SOP ENV-SOP-GBAY-0164 in Attachment B-2.

Consistent with prior sampling programs, samples will be sieved to the less than 250 µm fraction, reflecting the fine fraction of soil most likely to adhere to children's hands. Atlantic Richfield has performed a sieve study to compare the 250 µm to 150 µm results on 129 site-specific soil samples collected in 2021 and provided a final report of the results (Atlantic Richfield Company, 2022a). Since submission and review, a verbal response and direction has been issued by EPA, and the 2023 site samples will continue to be sieved to 250 µm.

Mercury concentrations will be determined per EPA Method 7471B (Manual Cold-Vapor Technique) on the wet sample collected in the field as a subsample from the homogenized sample bag.

3.1.6 Soil Sample Collection Equipment

Soil samples are collected primarily using hand tools that are limited to readily available products. If supplies should be exhausted, replacement supplies can be purchased at nearby retailers. Hand tools may include a sampling probe, Sharpshooter® type shovels, and heavy duty 5- to 6-foot steel pry bars. Single-use scoops and protective (latex/nitrile) gloves will be used to collect and mix the samples. Resealable plastic bags will be used as sample containers for those samples requiring arsenic and lead analyses. Those samples requiring mercury analysis will use 4-ounce amber glass sample jars as sample containers.

3.2 RMAP Dust Sampling (Residential Parcels)

All RMAP dust sampling work will be conducted as described below to determine the presence of the COCs listed in Table 1. Field personnel will follow the procedures in the RMAP-SOP-3, RMAP-SOP-4, and RMAP-SOP-XRF-002 (Attachment B-1 and Attachment B-4, respectively) and will record all information in the field logbook/data collection device. For residential properties where there is more than one use (i.e., a residential daycare area separated from the family living space) or the living space is apparently separated by dedicated entrances, the areas within the structure will be considered separate areas and samples will be collected for each. The procedures for RMAP dust sampling are summarized below.

3.2.1 Residential Attics and Crawl Spaces

This section summarizes the procedures for sampling attics and crawl spaces. Dust in these areas will be sampled as part of the Program assessment within the 2020 RMAP Area (refer to Figure 1), regardless of whether an exposure pathway exists. General procedures are listed below, and sample identification information is outlined in Section 3.6. The procedures described in this section for attics also apply to crawl spaces.

Field personnel will follow the procedures in the RMAP-SOP-3 (Attachment B-1) for all attic dust sampling activities, unless otherwise specified in this QAPP. Attic dust composite sampling (based on a minimum of two subsample locations within the attic) will be conducted via grab samples using a scoop and brush and placed in a 4-ounce amber glass sampling jar as appropriate for the location. All attic dust sampling jars will be shipped from the field on ice to the laboratory. The temperature upon sample receipt is measured and recorded by the laboratory on Sample Condition upon Receipt documentation. The samples will be stored chilled (less than or equal to 6 °C, but not frozen) in temperature monitored refrigerators prior to laboratory digestion and analysis within 28 days of sample collection. The sample digestion and analysis (for arsenic, lead, and mercury) will be performed on “wet” sample aliquots and reported on a dry weight basis.

The amount of dust and insulation present in the attic space will determine how samples are collected. Ideally, samples will be collected near the access point into the attic. This sample collection point will minimize disturbance to the owner's attic insulation as well as minimize the risk of health and safety and property damage incidents. Each sample will consist of at least the minimum amount of material required for laboratory analysis (minimum mass of 0.2 grams, ideally closer to 0.4 grams if site conditions allow). In the event that sampling personnel cannot collect enough sample mass near the attic access point, they will attempt to find additional dust locations within the attic space. Samples will be labeled using a unique sample identification number for tracking. Special care will be taken to ensure enough sample mass is collected for both arsenic/lead and mercury analysis. All sampling equipment will be single use. Therefore, decontamination will not be necessary.

At collection, the sample identification number will be recorded in the sample log and the chain of custody record completed. The log, sample label, and chain of custody record will be checked for identical entries. Sampling crew personnel will follow the steps listed below:

1. Label the amber glass jar for the sample with the appropriate sample identification number.
2. Visually inspect the attic/crawl space for any hazards.
3. Move insulation and/or debris to find the dust.
4. Collect enough dust to meet laboratory specifications for a sample.
5. Record the sample number in the sample log.
6. Complete the chain of custody record. Check the log, sample label, and chain of custody record for identical entries.
7. Take photographs of the attic/crawl space.
8. Replace any disturbed insulation and close access.

Complete chain of custody as described in the RMAP-SOP-SA-04 (Attachment B-1). Ensure all sampling identification information is entered into the Program's database tracking system. Prepare the samples for delivery to a certified laboratory to be analyzed for arsenic, lead, and mercury following the procedures detailed in the RMAP-SOP-SA-01 located in Attachment B-1.

3.2.2 Residential Living Space Dust Sampling

Indoor living space (including living space within a commercial structure), excluding attics and crawl spaces described in Section 3.2.1, dust sampling will be completed using a high volume small surface sampler (HVS3) or Agency-approved equivalent device according to manufacturer's recommendations. An operating manual for the HVS3 is included in Attachment E-1. The HVS3 is no longer manufactured or available for purchase. This method is dependent upon the ability to procure the required sampling equipment. Atlantic Richfield and BSB currently possess multiple HVS3 units which should serve the program for the foreseeable future. If the HVS3 supply starts running low, BSB will work with the Agencies to develop an alternative sampling method.

A composite sample will be collected consisting of subsamples using the following procedure.

1. Label the polyethylene 250 milliliter wide-mouth sampling bottle for the sample with the appropriate sample identification number.
2. Collect samples from the following locations at each residence to provide a composite sample of the minimum amount of material required for laboratory analysis (minimum mass of 0.2 grams, ideally closer to 0.4 grams if site conditions allow):
    a. The floor area directly inside the main entries.
    b. The floor areas in the most frequently occupied rooms (normally the living room and/or kitchen).
    c. The floors in the children's bedrooms.
    d. The floor areas adjacent to or under attic pathways.
3. Empty dust sample from the vacuum into the labeled bottle.
4. Clean vacuum parts per the manufacturer's guidelines (Attachment E-1).
5. Following decontamination, collect solid equipment blank samples at a rate of 1 per sampling day (see Section 3.8.3).

Additional procedures or conditions must include the following:

- Samples must be collected in certified clean sample bottles.
- At the time of sampling, the sampler must record the sample identification number in the sample log and complete the chain of custody record.
- The device must be decontaminated before each use.
- The log, sample label, and chain of custody record must be checked for identical entries.
- All personnel will follow chain of custody procedures.
- All sampling identification information must be entered into the Program's database tracking system.

As detailed in RMAP SOP-04, a composite sample needs a minimum mass of 0.2 grams (and ideally closer to 0.4 grams if site conditions allow) for laboratory analysis. If the sampling crew completes sampling of the areas detailed above in bullet #2 (without moving furniture) and is unable to obtain this minimum mass of dust, no further sampling of the area in question shall be required since dust is essentially not present in the living space due to good housekeeping practices.

All residential living space dust sampling bottles will be analyzed for arsenic, lead, and mercury, and will be shipped from the field on ice to the laboratory. The temperature upon mercury sample receipt is measured and recorded by the laboratory on Sample Condition upon Receipt documentation. The samples will be stored chilled (less than or equal to 6 °C, but not frozen) in temperature monitored refrigerators prior to laboratory digestion and analysis within 28 days of sample collection. The mercury digestion and analysis will be performed on "wet" sample aliquots and reported on a dry weight basis.

If living space dust samples exceed either the arsenic, lead, or mercury action levels, living space floors will be thoroughly cleaned with a remediation grade/HEPA filter vacuum or carpets will be removed and replaced. Non-living spaces will also be cleaned if an action level in those areas is exceeded and there is either a pathway allowing dust into the living space or the property owner is planning a remodel that will disturb the non-living space dust.

Field duplicate samples will be collected as side-by-side duplicates in separate containers rather than a split sample (see Section 3.8.4).

3.2.3 Emergency Dust Sampling

Extenuating circumstances (such as a collapsed ceiling allowing attic dust into the living space) may be presented on a case-by-case basis that will require the ability to provide immediate results to confirm arsenic and lead concentrations near or exceeding action levels. In these cases, dust sampling will be conducted using the Thermo Fisher Scientific Niton XL2 analyzer per the User's Guide in Attachment E-2 and RMAP-SOP-XRF-002 in Attachment B-4. Butte-Silver Bow County, in consultation with the Agencies, will define the site-specific sampling plan in this scenario.

Emergency dust sampling is anticipated to be an infrequent event that requires immediate turnaround and remedial decision making. Because of this, laboratory confirmation sampling is not anticipated to be necessary. Remedial decisions will be based on field XRF values compared with residential dust action levels shown in Table 1. All results will be recorded in the system database.

3.3 RMAP Mercury Vapor Sampling (Residential Parcels)

The following section describes when RMAP residential mercury vapor sampling is required and the procedures to be used.

3.3.1 Residential Mercury Vapor Sampling

If the interior residential dust sample exceeds the residential mercury action level listed in Table 1, air sampling will be performed in the three areas most frequented by children. If the earthen basement residential soil sample exceeds the residential mercury action level, air sampling will also be conducted in the basement. In the event of the mercury exceedances described above, BSB personnel will reach out to the property owner as soon as possible to convey the test results and discuss the next steps. Air sampling will be conducted as soon as a rental monitor can be secured and access is granted by the property owner.

Sampling for mercury is completed using a mobile, active sampling device, which provides real-time data results of mercury vapor concentration in the air using a cold vapor-atomic absorption spectrophotometer. Sampling personnel will perform the sampling by systematically walking through the living space making sure to vary the height of the sampling instrument to ensure proper characterization of the mercury concentrations within the residence.

Air sampling will be completed using a portable field device rented for the duration of sampling. The Human Health/RMAP Division Manager (or designee) will contact the preferred supplier of the specialty equipment to request calibration, setup, and delivery of the unit to allow the supplier to pre-set sampling parameters, calibrate the unit, decontaminate the unit (if necessary), and provide any accompanying documentation. The device must be field ready upon delivery. Field personnel will start the sampling device as described in the manufacturer's operating instructions and verify pre-set parameters. One such device used previously is the Mercury Tracker 3000 unit. This unit has a sensitivity of 0.1 $\mu g/m^3$, which is considerably below the action level of 0.43 $\mu g/m^3$. The unit also has an internal data logger that allows for automatic and continuous storage of the measured concentrations. An operating manual for that unit is included in Attachment E-3.

Air samples will be collected from the following areas most frequented by children:

1. The area directly inside the main entry to the residence.
2. The area in the most frequently occupied room (normally living room or kitchen).
3. The area in the child's bedroom or another frequently occupied room if no children are present in the home.

Additional samples will be collected from the basement area of exposed earthen basement soils if the mercury action level is exceeded in soil samples (Table 1).

Any recorded concentration above the action level (Table 1) will be considered a failing result and will trigger RA. Remedial action procedures are outlined in the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022).

3.4 RMAP Lead Sampling (Residential Parcels)

The following sections describe procedures to collect samples from painted residential surfaces and drinking water. Lead sampling of LBP and drinking water is performed during an EBL investigation. Given the extensive waterline infrastructure upgrades performed in recent years, the likelihood of lead pipe issues is most likely very small.

3.4.1 Residential Lead Paint Sampling

This section summarizes the procedure for sampling painted surfaces in a residential setting. The sampler(s) must follow the manufacturer's procedure for operating the Niton XLp 300 Series XRF instrument (refer to Attachment E-2). Paint assessment will begin with a visual inspection of the building following HUD *Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing* (HUD, 2012) to determine if there are potential LBP hazards (see Table 2). Interior and exterior components of the building, including outbuildings and fences, will be sampled with the portable XRF to determine the presence of LBP. The information obtained during this assessment will be recorded in the field collection device and will contain the following information:

- Date.
- Physical address of residence being sampled.
- XRF serial number.
- XRF calibration data and time.
- Property owner name and mailing address.
- Personnel conducting risk assessment.
- Sample location (room, wall, interior, exterior).
- Component sampled.
- Substrate (wood, metal, concrete).
- Paint condition.
- Paint color.
- Sample identification number and result.
- QC calibration check results

The following steps describe the assessment process the field sampler must follow when conducting paint assessments.

1. Calibrate the Niton XLp 300 series XRF instrument according to manufacturer's specifications (see Attachment E-2) and check calibration using known standards before and after each home is assessed. A calibration check using surface lead paint standard (Standard Reference Material [SRM] 2573) and a blank analysis (SRM 2570) will be performed after every 10 analyses to verify continuing calibration. Calibration check and blank analysis criteria are listed in Table 4 and the RMAP-SOP-XRF-001 provided in Attachment B-4.
2. Visually inspect the property.
3. Start in the interior of the house, in the room farthest from the primary entry point (if possible).
4. Test painted surfaces that are in fair or poor condition in each room, including closets and hallways.
5. After every XRF measurement, document the location, condition, result, etc. (see above).
6. Move to the exterior of the house and test as many different surfaces that are in fair or poor condition as possible on each wall.
7. After every test, repeat the documentation process.
8. After the house has been sampled, move to any outbuildings/fences, and test all painted surfaces that are in fair or poor condition if possible.
9. After every test, repeat the documentation process.
10. At the conclusion of all lead paint testing at a residence, complete the analysis sequence with a calibration standard and blank to bracket the sample results.

11. Download the XRF data onto the computer and enter that data and all other sampling information into the Program's database tracking system.
12. Generate an LBP testing report using the information from the LBP testing documentation. This report will be provided to the property owner.

A report containing all the above information will be provided to the property owner. The report will be recorded in the Program database and tracking system.

Remedial decisions will be based on the presence of LBP (see Table 1) and the condition of the LBP (see Table 2). Remedial action procedures are outlined in the *Revised Final Multi-Pathway RMAP Plan* (BSB & Atlantic Richfield Company, 2022).

3.4.2 Residential Drinking Water Sampling

Drinking water will be sampled as a component of an EBL investigation under the RMAP for the presence of lead on a site-specific basis. Once BSB has determined that residential drinking water should be sampled at a particular residence, BSB will contact the homeowner to initiate and streamline the sampling and analysis process as much as possible for homeowners. When tap water sampling has been deemed necessary, sample bottles and instructions for sampling will be provided by BSB. Once the sample has been collected by the homeowner according to the procedures described in Attachment C , BSB will pick up the bottle and return it to the laboratory, and results will be provided electronically to BSB for review and validation. Draft results indicating a lead exceedance will be reported to homeowners prior to data validation completion. No analysis or shipping costs will be incurred by the homeowner. In the event the sample is not collected by the homeowner in a timely manner, BSB will follow up with the homeowner.

3.5 Sample Handling and Chain of Custody

After collection and labeling, the samples will be maintained under strict chain of custody protocols, according to the sample packaging SOP RMAP-SOP-SA-01 (Attachment B-1). The field sampling personnel will complete a chain of custody form for each shipment/delivery (i.e., batch of coolers) of samples to be delivered to the laboratory for analysis.

Coolers containing soil sample jars that will be analyzed for mercury will be shipped from the field on ice to the Pace Analytical Services, LLC laboratory located in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) for analysis. Coolers containing project soil samples collected in resealable plastic bags that will be analyzed for lead and arsenic will be shipped from the field at ambient temperature conditions to the Pace Analytical Services, LLC laboratory located in Green Bay, Wisconsin (1241 Bellevue Street, Suite 9, Green Bay, WI 54302) for drying and sieving. Upon completion of drying/sieving activities, these samples will be shipped to the Pace Analytical Services, LLC laboratory in Minneapolis for analysis.

Coolers containing dust samples (amber glass jars for attic/crawl space dust and HVS3 sample bottles for living space dust) will be shipped from the field on ice to the Pace Analytical

Services, LLC laboratory in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) for analysis. No drying/sieving is anticipated for dust samples.

Coolers containing water samples will be shipped from the field at ambient temperature to the Pace Analytical Services, LLC laboratory in Minneapolis, Minnesota (1700 Elm Street SE, Minneapolis, MN 55414) for analysis.

Jennifer Anderson is the point of contact for Pace Analytical Services, LLC.

The sampler is responsible for initiating and filling out the chain of custody form. The chain of custody for a shipment/delivery will list only those samples in that shipment/delivery. Any documentation, including chain of custody, should be placed inside a resealable plastic bag, within the shipment/delivery container. Coolers that are to be shipped will be custody sealed, securely taped shut, and have a shipping label securely adhered to the cooler.

The sampling personnel whose signature appears on the chain of custody form is responsible for the custody of the samples from the time samples are collected until custody of the samples is transferred to a designated laboratory, a courier, or to another project employee for the purpose of transporting the samples to the designated laboratory. Custody is transferred when both parties to the transfer complete the portion of the chain of custody under "Relinquished by" and "Received by." Signatures, printed names, company names, dates, and times are required. Upon transfer of custody, the sampling personnel who relinquished the samples will retain the third sheet (pink copy), photocopy, or electronic copy of the chain of custody. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the chain of custody. Copies, receipts, and carbons of Bills of Lading will be retained as part of the permanent documentation in the project file. It is not necessary for courier personnel to sign the chain of custody.

Upon receipt by the laboratory, the samples will be inspected for sample integrity. The chain of custody will be immediately signed, dated, and reviewed by laboratory personnel to verify completeness. Any discrepancies between the chain of custody and sample labels and any problems or questions noted upon sample receipt will be communicated immediately to the Field Team Leader. The laboratory will provide the Field Team Leader and/or the QA Manager with a copy of the chain of custody and associated sample receipt information within two working days of receipt of samples. The sample receipt information routinely provided will include sample receipt date, sample identifications (IDs) transcribed from the chain of custody sample matrix type, and list of analyses to be performed for each sample. Broken custody seals, damaged sample containers, sample labeling discrepancies between container labels and the chain of custody form, and analytical request discrepancies will be noted on the chain of custody form. The Field Team Leader and QA Manager will be notified of any such problems, and the discrepancies or non-conformances will be resolved and addressed before the samples are analyzed.

The laboratory will be responsible for following their internal custody procedures from the time of sample receipt until sample disposal. Samples and extracts will be stored in a secure area controlled by the laboratory's designated sample custodian. Samples will be removed from the

shipping container and stored in their original containers unless damaged. Damaged samples will be disposed of in an appropriate manner after notifying the Field Team Leader and QA Manager and receiving and documenting authorization to dispose. In addition, samples will be stored after completing analysis according to contractual requirements.

3.6 Sample Identification

The RMAP sample ID procedures are detailed in this section. An alphanumeric coding system will be used to uniquely identify each sample collected during RMAP sampling events. Sample identifiers will begin with the matrix, followed by the RMAP Database Resident ID. The Resident ID is a unique identifier that is associated with a specific property (address and/or geocode specific). Following the Resident ID will be the parcel component, QA/QC code (when applicable), and sample depth.

Matrix:

S – Exterior Soil
Attic – Attic Dust
IDV – Interior/Living Space Dust
B – Basement Soil
V – Vapor
P – Paint
W – Water

RMAP Database Resident ID: (example of R-00001)

Site Property Codes:
- R – Residential
- C – Commercial
- V – Vacant
- O – Open Space

Resident ID:
- 00001 – associated with a specific address or geocode

Parcel Component:

Component IDs will be derived on a site-specific basis during development of the Sample Location Map and refined by the sampling team (as necessary). Examples of Component IDs are listed below.

Soil Parcel Components:
- Blvd – Boulevard
- DW – Driveway
- EB – East Boulevard
- EY – East Yard
- FG – Flower Garden

MY – Middle Yard
NB – North Boulevard
NY – North Yard
NEY – Northeast Yard
NWY – Northwest Yard
RG – Rock Garden
PA – Play Area
SA – Sand Box
SB – South Boulevard
SY – South Yard
SEY – Southeast Yard
SWY – Southwest Yard
VG – Vegetable Garden
WB – West Boulevard
WY – West Yard

Quality Control/Quality Assurance Codes:

D – Field Duplicate
F – Field Blank
R – Field Equipment Decontamination Blank

Depth Intervals: Depth intervals are only applicable to soil sampling events.

1. 0 to 2 inches bgs
2. 2 to 6 inches bgs
3. 6 to 12 inches bgs
4. 12 to 18 inches bgs (flower/vegetable gardens only)
5. 18 to 24 inches bgs (flower/vegetable gardens only)

An example sample identification would be S-R-00001-NY-2. This indicates that the soil sample was collected at the residential property R-00001 (corresponding to a physical address and/or geocode) in the north yard at the 2- to 6-inch depth interval. The sample identification for a field duplicate collected at this location would be S-R-00001-NY-D-2.

If multiple parcel components of the same type are encountered on a property, an incremental number will be added to the end of the parcel component, i.e., a site with multiple flower gardens:

- The first flower garden would be assigned S-R-00001-FG-2 for the soil sample that was collected at the residential property R-00001 (corresponding to a physical address and/or geocode) in the flower garden at the 2- to 6-inch depth interval.
- The next flower garden on the same property would be assigned S-R-00001-FG2-2 for the soil sample that was collected at the residential property R-00001 (corresponding to a physical address and/or geocode) in the second flower garden at the 2- to 6-inch depth interval.

Sample identifiers will be documented in field logbooks/data collection device and on the chain of custody forms, as required by the RMAP Field SOPs RMAP-SOP-SA-05 and RMAP-SOP-SA-04 located in Attachment B-1.

3.7 Analysis Methods

The subsections below describe analytical methods the respective laboratories must use to analyze RMAP samples.

3.7.1 Soil Sample Analysis Method

All RMAP soil samples will be analyzed to determine metals concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury. Sample preparations and analyses will be conducted according to the referenced EPA analytical method specifications as well as standard laboratory practices. Arsenic and lead samples will be sieved at the laboratory, and the fine fraction of the sieved soil will be digested according to modified EPA Method 3050B. Arsenic and lead concentrations will then be determined per EPA Method 6010 (ICP-AES) or EPA Method 6020 (ICP-MS). Mercury concentrations will be determined per EPA Method 7471B (Manual Cold-Vapor Technique) on un-sieved samples.

The laboratory SOPs for EPA Methods soil sieving, 3050B, 6010, 6020, and 7471B are included in Attachment B-2.

3.7.2 Dust Sample Analysis Method

All RMAP dust samples with the exception of emergency dust sampling (see Sections 3.2.3 and 3.7.3) will be analyzed to determine metals concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury as appropriate. Discernable objects will be manually removed from bulk dust samples. The dust samples collected using conventional collection methods will then be digested according to EPA-modified Method 3050B and analyzed by EPA Method 6010 (ICP-AES) or EPA Method 6020 (ICP-MS) for arsenic and lead. No drying/sieving is anticipated. Mercury concentrations will be determined per EPA Method 7471B (Manual Cold-Vapor Technique). Results of the dust analysis will be reported on a wet-weight basis, due to the concern of not having a representative mass to perform dry-weight analysis.

The laboratory SOPs for EPA Methods 3050B, 6010, 6020, and 7471B are included in Attachment B-3.

3.7.3 Emergency Dust Sample Analysis Method

Those emergency solid media samples analyzed via field XRF will be completed according to the Thermo Fisher Scientific Niton XL2 analyzer User's Guide included in Attachment E-2 and EPA Method 6200 provided in Attachment B-4 as applicable to dust samples. This will provide real time data to compare to the appropriate action level to make real time remedial decisions. Three replicate XRF measurements will be analyzed for lead for every dust sample collected.

Remedial decisions will be based upon the highest of the three measurements. Additional details on analysis and QA requirements are included in Table 4.

3.7.4 Mercury Vapor Sample Analysis Method

All residential mercury vapor data will be real time data collected by a Mercury Tracker 3000 unit (or Agency-approved equal). The real time data will be compared to the appropriate action level to make remedial decisions.

3.7.5 Lead Paint Sample Analysis Method

All residential lead paint samples will be analyzed per the manufacturer's user manual for the XRF unit (see Attachment E-2) and EPA Method 6200 and the SOP RMAP-SOP-XRF-001 in Attachment B-4 to determine concentrations of lead. Three replicate XRF measurements will be analyzed for lead for every paint sample collected. Remedial decisions will be based upon the highest of the three measurements.

3.7.6 Drinking Water Sample Analysis Method

All residential water samples will be analyzed per EPA 200.8 to determine concentrations of lead. Sample preparation and analysis will be conducted according to the SOPs provided in Attachment B-5. For the total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is less than 1 nephelometric turbidity unit (NTU), the sample is made ready for analysis by the appropriate addition of nitric acid added by the laboratory, mixed, and allowed to equilibrate for the required time prior to analysis. In the event turbidity is greater than 1 NTU, the sample will be digested according to EPA Method 200.8.

3.7.7 Laboratory Quality Control Samples

As outlined in Sections 3.7.1 and 3.7.2, RMAP solid media samples (soil and/or dust) with the exception of emergency residential dust sampling will be analyzed to determine metals concentrations (arsenic, lead, and mercury) via standard laboratory analytical methodologies. Laboratory QC procedures are outlined below.

All analyses will be governed by the appropriate calibration procedures and frequencies that are specified in the laboratory's SOPs (see Attachment B).

Laboratory QC samples will be analyzed in addition to the calibration samples with each QC batch. Laboratory QC samples are introduced into the measurement process to evaluate laboratory performance and sample measurement bias. Control samples may be prepared from environmental samples or generated from standard materials in the laboratory.

Emergency dust sampling analysis by XRF will include the analysis of calibration verification samples after instrument calibration, and SRMs will serve as laboratory control samples as continuing checks on instrument stability for every 10 samples analyzed and at the end of the dust sampling event to bracket the sample measurements. A calibration check using surface lead

paint standard (SRM 2573) and a blank analysis (SRM 2570) will be performed after every 10 analyses to verify continuing calibration. Calibration check and blank analysis criteria are listed in Table 4 and the SOP. Field XRF Field SOPs are included in Attachment B-4.

Control standards associated with interior mercury vapor sampling will be determined at the time of sampling, equipment rental based on unit availability, and manufacturer recommendations. This issue will be discussed with Agency personnel at that time.

Laboratory blanks, laboratory control samples, analytical duplicates, serial dilutions, and pairs of matrix spike/matrix spike duplicate (MS/MSD) samples will be analyzed in each laboratory QC batch with a minimum frequency of 1 per 20 field samples. If less than 20 field samples are submitted, then one set of these QA/QC samples will still be run with the set of less than 20 samples. A second MS sample is not necessary for all laboratory QC batches that already have one MS/MSD.

Laboratory Blanks

Method blanks will be used to monitor laboratory processes and performance. A method blank is a volume of deionized water or a specified weight of inert material for solid samples that is carried through the entire sample preparation and analysis procedures. The method blank volume or weight will be approximately equal to the sample volumes or sample weights being processed. Method blanks are used to monitor interference caused by constituents in solvents and reagents and on glassware and other sampling equipment. Method blank results outside of specified control limits will be rerun/redigested and reanalyzed with all associated samples and/or flagged by the laboratory per the QC requirements of the analytical method. Initial and continuing calibration blanks are also analyzed every 10 samples and samples are reanalyzed within compliant blank analysis. All elements of interest must be evaluated to plus or minus the reporting limit (RL) for Method 6020.

Laboratory Control Samples

A LCS, or a blank spike, is an aqueous or solid control sample of known composition that is analyzed using the same sample preparation, reagents, and analytical methods employed for the Program samples. The LCS is obtained from an outside source or is prepared in the laboratory by spiking reagent water or a clean solid matrix from a stock solution that is different from that used for the calibration standards. The LCS is the primary indicator of process control used to demonstrate whether the sample preparation and analytical steps are in control, apart from sample matrix effects. If the LCS recovery falls outside the specified control limits, the LCS is reanalyzed once. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be redigested and reanalyzed.

Analytical Duplicates

Analytical duplicates are samples that are split in the laboratory at some step in the measurement process and then carried through the remaining steps of the process. Duplicate analysis provides information on the precision of the operations involved. Analytical duplicates are a pair of subsamples from a field sample that are taken through the entire preparation and analysis procedure; any difference between the results indicates the precision of the entire method in the given matrix. Analysis of analytical duplicates and MSDs monitor the precision of the analytical

process. The frequency of analysis, precision goals, and corrective action information pertaining to analytical duplicates are provided in the laboratory SOPs (Attachment B). If the analytical duplicate precision falls outside the specified control limits, the samples will be rerun and/or flagged by the laboratory per the QC requirements of the analytical method.

Serial Dilutions
Serial dilutions are performed in conjunction with EPA Method 6010 or 6020 to determine whether significant physical or chemical interferences exist due to the sample matrix. A serial dilution is performed by analyzing a five-fold dilution of a field sample (field blanks may not be used) and calculating the percent difference between the original determination and the serial dilution result. Serial dilutions are only applicable for analyte concentrations that are greater than 50 times the MDL. The frequency of analysis, precision goals, and corrective action information pertaining to serial dilutions are provided in the laboratory SOPs in Attachment B.

Matrix Spikes
Laboratory MS samples are used to evaluate potential sample matrix effects on the accurate quantitation of an analyte using the prescribed analytical method. The MS/MSDs are prepared by adding an analyte to a subsample of a field sample before sample preparation and analysis. A percent recovery is calculated from the concentrations of the analyte in the spiked and unspiked samples. A post digestion spike will be performed on any elements that fail to meet criteria. If the %R for the MS and MSD falls outside the control limits, the results are flagged by the laboratory that they are outside acceptance criteria along with the parent sample.

Additional Quality Control Samples
The laboratory will also analyze ICP/MS interference check, internal standards, and ICP/MS instrument tunes as part of the analytical sequence for Method 6020. These instrument QC samples will be evaluated against the method requirements during data validation.

Table 4 contains acceptance criteria for the QC samples detailed above.

3.8 Field Quality Control Samples

Field QC samples are used to identify any biases from transportation, storage, and field handling processes during sample collection and to determine sampling precision. All field QC samples will be delivered with field samples to the laboratory. This section includes brief descriptions of the QC samples to be collected during sampling activities along with frequency, collection, and analytical instructions.

Sampling protocols will be consistent with the Field SOPs included in Attachment B-1 and will include 1 field duplicate collected for every 20 primary samples or once per sampling event (e.g. once per sampling day), whichever is more frequent (according to Level A/B Field Screening/Data Review Criteria included in Attachment D). Any disposable sampling equipment used (i.e., sampling equipment that is anticipated to be “one time use”) will not be subject to external contamination blank/cross-contamination blank samples unless the equipment is decontaminated and used between samples. Reusable sampling equipment will be subject to field equipment blanks once per crew per sampling event (e.g., once per day for each crew).

Equipment used for attic dust sampling will be single use/disposable. Equipment used for residential living space dust sampling will be decontaminated per manufacturer's instructions (see Attachment E) and solid equipment blanks collected once per sampling day. Any required decontamination of mercury vapor testing equipment will be handled by the rental house providing the equipment per the manufacturer's current specifications. Any deviation from the SOPs or this QAPP will be identified in the logbook/data collection device and discussed in the annual DSR.

3.8.1 Field Duplicate (Soil Samples)

A field duplicate consists of one well-mixed and homogenized sample that is split in the field into two samples and placed in different sample containers for separate analysis.

As with all other samples, samples to be split for duplicate samples will be thoroughly mixed in a clean 1-gallon resealable plastic bag or stainless-steel bowl to ensure the representativeness of the aliquot ultimately submitted for analysis. During this homogenization process, particles greater than 0.5 inches in diameter will be discarded. Once the homogenization process is complete, the natural sample will be split into two samples (prior to subsampling for mercury analysis). Soil samples for mercury analysis for this project will be collected by removing several subsample aliquots from the homogenized samples contained in the resealable plastic bag (e.g., Ziploc®) during the sample collection process and placed in glass containers. This process helps to ensure sample representativeness between the sample aliquots. Additional homogenization of the sample through labor intensive splitter or cone and quartering techniques may lead to volatilization of the mercury. According to *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, U.S. EPA Publication SW 846* (EPA, 2015), the sample jars for mercury analysis will be shipped from the field on ice to the primary laboratory. Each split will have its own sample number. Both split samples will be analyzed for identical chemical parameters. The results of the field duplicate will be compared to determine laboratory and sampling precision. Field duplicate samples will be collected at a frequency of 1 per 20 samples or once per sampling event (e.g., once per sampling day), whichever is more frequent.

The RPD field precision goal for soil field duplicates will be 35% for sample pairs with both sample results being greater than 5 times the RL. For soil field duplicate/primary sample pairs with one or both sample results being less than five times the RL, an absolute difference of less than or equal to 2 times the RL (difference less than or equal to 2 times the RL) will be used as the precision goal.

3.8.2 Field Equipment Blank (Soil Samples)

A field equipment blank consists of a sample collected after the decontamination of sampling equipment is completed and before sampling. Equipment blanks are collected by pouring a fine solid matrix (e.g., silica sand) through or over the cleaned sampling equipment and collecting the solid in the same zipper style bag as used for sample collection. The goal of each field equipment blank is to confirm no measurable contamination is present. However, if contamination is measured, no additional corrective action can be taken at that time for that blank. The data

validation process will evaluate the effects on sample data. Additional field corrective action can be taken on subsequently collected field equipment blanks through evaluation of the current practice and adding additional cleaning steps to reduce the potential of equipment-based cross contamination.

The field equipment blank results will be used to determine blank qualification during data validation. A minimum of one field equipment blank will be collected per crew per sampling event (e.g., once per day per crew). Field equipment blanks are not necessary for one-use or disposable sampling equipment that is not being used for collection of more than one natural sample.

Field Equipment blank samples will be analyzed to determine metals concentrations via standard laboratory analytical methodologies for arsenic, lead, and mercury. Sample preparations and analyses will be according to referenced EPA analytical method specifications and standard laboratory practices. Arsenic and lead concentrations will be determined per EPA Method 6010 (ICP-AES) or EPA Method 6020B (ICP-MS). Mercury concentrations will be determined by EPA Method 7470A: Mercury in Liquid Wastes (manual cold-vapor technique).

3.8.3 Field Equipment Blanks (Residential Living Space Dust Samples)

Equipment blanks are collected to evaluate potential cross-contamination between samples collected with the HVS3 vacuum. For this sampling effort, equipment blanks will be collected at a rate of one per crew per sampling day. Equipment blanks will be collected after the first sample has been collected and the HSV3 has been decontaminated. Approximately 5 grams of fine solid matrix (e.g., silica sand) will be poured through the sample collection chamber into the sample catchment container.

3.8.4 Field Duplicate (Dust Samples)

Field duplicate samples associated with dust sampling will be collected as side-by-side duplicates in separate containers rather than a split sample. Each duplicate sample will have its own sample number. Both the original and duplicate sample will be analyzed for identical chemical parameters. The results of the field duplicate will be compared to determine laboratory and field precision. Field duplicate samples will be collected at a frequency of once per sampling event (e.g., once per day) when sufficient attic dust mass is available. The Field Sampler will use a visual inspection of the area or location to be sampled when side-by-side duplicates are collected. The Field Sampler should visually divide the area or location to be sampled into two equal areas prior to sampling (i.e., dividing a windowsill, attic rafter, or floor area). The two approximately equal areas will be collected using the same method as other dust samples into separate sample collection jars. If multiple areas of the same room or attic are sampled, this technique should be used for each area sampled and the collected dust placed into the total dust sample collected for the original and field replicate samples.

3.8.5 Field XRF Quality Control (Paint Only)

The XRF field instrument(s) will be used to analyze painted surfaces. The QC steps identified in Table 4 will be performed daily.

Daily Calibration

The XRF should be auto calibrated daily prior to use or if the unit is turned off during the day. Instructions for daily calibration are provided in the SOP RMAP-SOP-XRF-001 (Attachment B-4). The instrument auto calibration process is internally performed and will not operate unless the calibration is acceptable based on the instrument programming.

Calibration Verification Check Samples

Calibration verification check samples help check the accuracy of the analytes of interest. Results for the pre-investigation calibration sample will be recorded and identified as a calibration check sample. There will be National Institute of Standards and Technology (NIST) SRM check samples provided by the manufacturer. A calibration check using surface lead paint standard (SRM 2573) and a blank analysis (SRM 2570) will be performed after every 10 analyses to verify continuing calibration. Calibration check and blank analyses criteria are listed in Table 4 and the SOP RMAP-SOP-XRF-001 (Attachment B-4). The measured values of a standard will be compared to the expected results and if a measured value falls outside this range specified in Table 4, then the check sample will be reanalyzed. If the value continues to fall outside the acceptance range, this information will be noted on the XRF log. If any of the check sample results indicate that the XRF is not analyzing accurately, the XRF will be cleaned, turned off, and the energy calibration rerun. This information will be noted in the logbook/data collection device. In the event the XRF unit cannot be calibrated it will be removed from service.

Emergency dust sample collection will include an analysis of a clean sample matrix after sample calibration and verification. These blanks may include a solid matrix for dust samples. The XRF window will be cleaned with a soft cloth or cotton swab to remove any surficial dust between samples to minimize potential contamination from previously analyzed samples.

3.9 Sample Disposal

All samples shipped to the laboratory for analysis will be held until the laboratory analyses are completed, the Agencies have reviewed and approved all subsequent project laboratory data and work plans, and the sample hold times have expired. At this point, the laboratory may dispose of samples or return them to BSB for disposal. Any excess soil mass that was not included in the aliquot submitted to the laboratory will be subject to the same disposal criteria.

3.10 Instrument/Equipment Testing, Inspection, and Maintenance

To ensure continual quality performance of any instruments or equipment, the testing, inspection, and maintenance activities listed in the sections below will be performed and recorded.

3.10.1 Field Equipment

Field equipment will be examined to certify that it is in proper operating order prior to its use. Equipment, instruments, tools, and other items requiring preventative maintenance will be serviced according to the manufacturer's specified recommendations. Field equipment will be cleaned and safely stored between each use. Any routine maintenance recommended by the equipment manufacturer will also be performed and documented in field logbooks/data collection device. Equipment will be inspected, and the calibration checked, if applicable, before it is transported to a field setting for use.

Portable field XRF analyzers will be calibrated prior to day of use, following the manufacturer's instructions and using manufacturer-recommended calibration standards. Calibration logs will be stored electronically, within project files. Calibration failures will result in the field XRF being immediately removed from service. Once repaired, and successfully calibrated, the field XRF will be returned to service.

Portable field mercury vapor analyzers will be rented from a supplier of the specialty equipment when mercury vapor sampling is required. The supplier will be requested to provide pre-sampling calibration, setup, and delivery of the unit. The device must be field ready upon delivery. Field personnel will start the sampling device as described in the manufacturer's operating instructions and verify pre-set parameters. Any issues with calibration or device set up will be reported to the supplier to be rectified before the unit is returned to service.

3.10.2 Laboratory Equipment

Instruments used by the laboratories will be maintained according to each laboratory's QA plan and analytical method requirements. All analytical measurement instruments and equipment used by the laboratory will be controlled by a formal calibration and preventive maintenance program.

The laboratories will keep maintenance records and make them available for review, if requested, during laboratory audits. Laboratory preventive maintenance will include routine equipment inspections and calibrations at the beginning of each day or each analytical batch, per the laboratory's internal SOPs and method requirements.

3.11 Inspection/Acceptance of Supplies and Consumables

All supplies and consumables received for the project (e.g., sampling equipment, supplies, etc.) will be checked for damage and other deficiencies that could affect their performance. The types of equipment that will be needed to complete sampling activities are described in the relevant SOPs. Inspections of field supplies will be performed by field team members.

The personnel at each laboratory will be responsible for performing inspections of laboratory supplies according to their QA plan.

3.12 Data Management Procedures

This section describes the management of data for the project, including field and laboratory data. The Program quality records will be maintained by the Data Management Division Manager, as described in the BPSOU *Final Data Management Plan* (Atlantic Richfield Company, 2022b). These records, either electronic or hard copy in form, may include the following:

- Project work plans with any approved modifications, updates, and addenda.
- Individual property maps (hard copy or scanned field drawings and electronic files).
- Project QAPP, including this QAPP, with any approved modifications, updates, addenda, and corrective or preventative actions.
- Access agreements from property owners.
- Field documentation.
- Chain of custody records.
- Laboratory documentation (results received from the laboratory will be documented both in report form and in an electronic format).
- Annual completion report.

Hard copy field and laboratory records will be maintained in the project's central data file, where original field and laboratory documents are filed chronologically for future reference. These records are also scanned to produce electronic copies. The electronic versions of these records are maintained on a central server system with backup scheduled daily.

Before field and laboratory data are incorporated into the project database, the data and supporting documentation will be subject to appropriate review to ensure the accuracy and completeness of original data records. Field data that have been reviewed in a hard-copy format will be entered into electronic data files for upload to the project database. All manual data entry into an electronic format will be reviewed by a separate party before the information is incorporated into the database. Laboratory EDDs and related data packages will be reviewed as part of the internal data review process. The Data Management Division Manager, or designated alternate, will be responsible for ensuring data integrity prior to database uploads. Following these review steps, field and laboratory electronic data files will be imported to the project database. Upon completion of the data validation process described in Section 5.0, the uploaded laboratory EDD will be qualified and usability qualifiers and reason codes added. Once the EDD has been verified as accurate it will be uploaded to the BSB database as final data.

Standardized data import formats and procedures will be used to upload both field and laboratory data into the electronic database. Standardized parameter names, numerical formats, and units of measure may be applied to the original information to facilitate comparability across all datasets and within the database. Data management activities for the RMAP program will be further defined in the *BPSOU Final Data Management Plan* (Atlantic Richfield Company, 2022b).

3.12.1 Requests for Data

Requests for data can be made to the Data Management Division Manager or to the Agencies who can access data directly through the secure Project database. Refer to the *Butte-Silver Bow Information Management System Plan* (BSB & Atlantic Richfield Company, 2016) for additional details and specific examples of the Program's database and tracking system. The *Institutional Controls Management System Plan* (BSB & Atlantic Richfield Company, 2019b) is in Appendix G of the ICIAP (BSB & Atlantic Richfield Company, 2019a).

4.0 ASSESSMENT AND OVERSIGHT

Assessment and oversight of data collection and reporting activities are designed to verify that sampling and analysis are performed according to the procedures established in this QAPP. The audits of field and laboratory activities include two independent parts: internal and external audits.

An internal field audit will be considered in 2023 to ensure compliance with the QAPP and consistency between individual crews. An internal laboratory audit of the Pace Analytical Services, LLC Green Bay, Wisconsin, and Minneapolis, Minnesota, facilities was recently conducted as part of the RMAP school sampling effort in July 2021. Due to the recency of these internal laboratory audits with essentially the same analytical scope, a second internal laboratory audit is not necessary at this time.

Internal audits may be performed by Atlantic Richfield or their approved representative, BSB, their contractor(s), or a contracted laboratory, as necessary. For Atlantic Richfield-led work, all internal audits will be conducted by Atlantic Richfield's contractor, Environmental Standards, Inc.

External audits may be performed by the Agencies as necessary for either BSB- or Atlantic Richfield-led sampling efforts.

Performance and system audits of field and laboratory data collection and reporting procedures are described in this section.

4.1 Corrective Actions

Corrective action is the process of identifying, recommending, approving, and implementing measures to counter unacceptable procedures or out-of-QC performance, which can affect data quality. Corrective action can occur during field activities, laboratory analysis, and data assessment. A corrective action template is provided in Attachment G.

Non-conforming equipment, items, activities, conditions, and unusual incidents that could affect data quality and attainment of the project's quality objectives will be identified, controlled, and reported in a timely manner. For the purpose of this QAPP, a non-conformance is defined as a malfunction, failure, deficiency, or deviation that renders the quality of an item unacceptable or indeterminate in meeting the project's quality objectives.

Corrective action in the laboratory may occur prior to, during, and after initial analysis. Several conditions such as broken sample containers, preservation or holding-time issues, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Corrective actions to address these conditions will be taken in consultation with the Human Health/RMAP Division Manager or the Data Management Division Manager/QA Manager. In the event that corrective action requests are not in complete accordance with approved project planning documents, the Agencies will be consulted, and concurrence will be obtained before the change is implemented, or new samples may be obtained.

If during analysis of the samples the associated laboratory QC results fall outside of the project's performance criteria, the laboratory should initiate corrective actions immediately. Following consultation with laboratory analysts and section leaders, it may be necessary for the contract laboratory's QA officer to approve implementing a corrective action. These conditions may include dilution of samples, additional sample extract cleanup, or automatic reinjection/reanalysis when certain QC criteria are not met, etc. If the laboratory cannot correct the situation that caused the non-conformance and an out-of-control situation continues to occur or is expected to occur, then the laboratory will immediately contact the Human Health/RMAP Division Manager and/or the BSB QA Manager and request instructions regarding how to proceed with sample analysis.

Completion of any corrective action should be evidenced by data once again falling within the project's performance criteria. If this is not the case, and an error in laboratory procedures or sample collection and handling procedures cannot be found, the results will be reviewed by the BSB QA Manager to assess whether reanalysis or resampling is required.

All corrective actions taken by the laboratory will be documented in writing by the laboratory project manager and reported to the BSB QA Manager. All corrective actions taken by field crews will be documented in writing by the Human Health/RMAP Division Manager and reported to the BSB QA Manager. In the event that corrective action requests are not in complete accordance with approved project planning documents, the Agencies will be consulted, and concurrence will be obtained before the change is implemented. All corrective action records will be included in the QAPP quality records.

4.2 Corrective Action During Data Assessment

The need for corrective action may be identified by any member of the project team during data assessment. Potential types of corrective action may include resampling by the field team, reanalysis of samples by the laboratory, or resubmitting data packages with corrected clerical errors. The appropriate and feasible corrective actions are dependent upon the ability to mobilize the field team and whether the data to be collected is necessary to meet the required QA objectives (e.g., the holding time for samples is not exceeded). In the event that corrective action requests are not in complete accordance with approved project planning documents, the Agencies will be consulted and concurrence will be obtained before the change is implemented. Corrective actions of this type will be documented by the BSB QA Manager on a Corrective Action Report and will be included in any subsequent reports.

4.3 Reports to Management

Upon receipt of laboratory results and completion of the data review/validation process, all validated analytical data will be uploaded into a project database and submitted to the Agencies for review and approval.

RMAP personnel will have the ability to distribute landowner result letters prior to completion of data validation work and formal EPA approval following the template established during the 2021 Butte area school soil sampling project. There are circumstances (extremely high metals concentrations in soils, attic sampling results, EBL scenarios, etc.) where expedited turnaround of sampling results and implementing resultant RA is vital to the health of the RMAP program and the homeowners it supports. The sample results (for all analytes) will be reported to individual landowners along with a letter explaining what the results indicate. The action levels for arsenic, lead, and mercury will be reported along with sample results.

After site investigations and remedial actions are complete, the Data Management Division Manager/QA Manager will prepare an annual DSR (Section 2.9.5) summarizing the sampling activities. The laboratory turnaround time for providing sample results will be adequate to allow data review and completion of the annual DSR. The report will describe specific field sampling activities performed during implementation of the QAPP. Each annual report will include field documentation, documentation of field QC procedures, results of all field and laboratory data, data validation results, and data usability assessments.

A separate report will be prepared by the BSB QA Manager, as needed, to communicate the results of performance evaluations or program audits to identify specific significant QA issues and provided to the Agencies for review. Any corrective action reporting described in Section 4.2 will be summarized and included as appropriate.

5.0 DATA REVIEW AND USABILITY

The following sections address the final project checks that will be conducted after the data collection phase of the project is complete to confirm that the data obtained meet the project objectives and to estimate the effect of any deviations on data usability for the express purposes of achieving the stated DQOs (Section 2.7.1). The data review/validation process under this QAPP is streamlined to support the post-BPSOU ROD (EPA, 2006b) decision-making process. The analytical data collected under this QAPP and produced by analytical laboratories will undergo a combination of Stage 4 and 2B data validation. The field documentation will be subject to Level A/B criteria review, and analytical data will be validated per the *CFRSSI Data Management/Data Validation Plan* (ARCO, 1992a), the *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2020b), and the project DQOs. Data review and validation will be conducted by a qualified technical consultant who is independent from the sampling consultant (i.e., an individual other than the individual who performed sampling).

5.1 Data Review, Verification, and Validation

This section describes the review, verification, and validation process for field data and laboratory data. The section also details laboratory data reporting requirements, which describe how results are conveyed to data users.

5.1.1 Data Review Requirements

Data review is performed by the data producer to ensure that the data have been recorded, transmitted, and processed correctly.

Field Data Review

Raw field data will be entered in field logbooks/data collection device and reviewed for accuracy and completeness by the Human Health/RMAP Division Manager, QA Manager, or Field Team Leader before those records are considered final. The overall quality of the field data from any given sampling round will be further evaluated during the process of data reduction and reporting. The field data will be reviewed quarterly by the Program QA Manager, or designated alternate.

Field data reduction procedures will be minimal in scope compared to those implemented in the laboratory setting. Field data review will include verification that any QC checks and calibrations, if necessary, are recorded properly in the field logbooks/data collection device and that any necessary and appropriate corrective actions were implemented and recorded. Such data will be recorded in the field logbook/data collection device immediately after measurements are taken. If errors are made, results will be legibly crossed out, initialed, and dated by the field member, and corrected in a space adjacent to the original (erroneous) entry. Later, the Field Team Leader will proof the field logbooks/data collection device to determine whether any transcription errors have been made by the field crew. If transcription errors have been made, the Field Team Leader and field crew will address the errors to provide resolution.

As appropriate, field measurement data will be entered into electronic files for import to the project database. Data entries will be made from the reviewed logbooks/data collection device, and all data entries will be reviewed for accuracy and completeness by a separate party before the electronic file is provided to the database manager. Electronic files of field measurement data will be maintained as part of the project's quality records.

Laboratory Data Review

Internal laboratory data reduction procedures will be according to each laboratory's quality management plan. At a minimum, paper records will be maintained by the analysts to document sample identification number and the sample tag number with sample results and other details, such as the analytical method used (e.g., method SOP #), name of analyst, the date of analysis, matrix sampled, reagent concentrations, instrument settings, and the raw data. These records will be signed and dated by the analyst. Secondary review of these records by the Laboratory Supervisor (or designee) will take place prior to final data reporting. The laboratory is

responsible for assigning appropriate flags/qualifiers according to the analytical method and internal laboratory SOPs.

5.1.2 Data Verification Requirements

Data verification is the process for evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual specifications.

Field Data Verification

The Level A/B review (see checklist in Attachment D), as described in the *CFRSSI Data Management/Data Validation Plan* (ARCO, 1992a) and the *CFRSSI DM/DV Plan Addendum* (AERL, 2000b), will be used in the verification process for field documentation related to samples collected for laboratory analysis.

The Level A criteria include:

- Sampling date.
- Sample team and/or leader.
- Physical description of sample location.
- Sample depth (soils).
- Sample collection technique.
- Field preservation technique.
- Sample shipping records.

The Level B criteria include:

- Field instrumentation methods and standardization completed.
- Sample containers preparations.
- Collection of field duplicates.
- Proper and decontaminated sampling equipment.
- Field custody documentation.
- Shipping custody documentation.
- Traceable sample designation number.
- Field notebook(s), custody records in secure repository.
- Complete field forms.

5.1.3 Laboratory Data Verification

The laboratory will prepare Level 2 and Level 4 data packages for transmittal of results and associated QC information to the Human Health/RMAP Division Manager or its designee within a standard turnaround time unless otherwise required.

These data packages will be prepared according to *EPA Contract Laboratory Program Statement of Work for Superfund Analytical Methods (Multi-Media, Multi-Concentration) SFAM01.1* (EPA, 2020c). Deviations from these specifications may be acceptable based on the SW-846 Methods provided the report presents all of the requested types of information in an organized, consistent, and readily reviewable format.

Each data package, as described above, will be accompanied by an EDD prepared by the laboratory. If data qualifiers are required, they will be added to the laboratory EDD and provided for uploading to the database. Additional laboratory QC data can be included in the EDD. The EDDs will be cross checked against corresponding data reports to confirm consistency in results reported in these two separate formats. This cross check will take place as part of the data verification process. All data will be submitted in both Level 2 and Level 4 format.

Resolution of Deficiencies

Any deficiencies found during the verification process will be discussed with the data producer and may be resolved with a revised data package.

5.1.4 Data Validation Requirements

The purpose of analytical data validation is to provide an assessment of data quality. Data validation will be performed by qualified, independent data validation personnel, who are not associated with data collection or sampling responsibilities and have applicable training. Data validation categorizes data as acceptable for use, unacceptable for use, or qualified for select use. The validation effort routinely identifies data use limitations and corrects reporting and quantitation errors. The data packages provided for validation will be evaluated for compliance with respect to the requested analytical methods and/or the QAPP and completeness of requested deliverables. Concurrent with the data validation efforts, analytical data usability will also be assessed. Analytical data usability is the determination of whether a data set is sufficiently complete and of sufficient quality for further evaluation by the data user as detailed in Section 5.3 of the QAPP to support a decision or action.

The data will be validated during the data validation process with guidance from the *CFRSSI QAPP* (ARCO, 1992b), the *CFRSSI Data Management/Data Validation Plan* (ARCO, 1992a), the *CFRSSI Data Management/Data Validation Plan Addendum* (AERL, 2000b), the *National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2020b), laboratory-specific QC criteria, and/or method-specific criteria where applicable. The use of the Functional Guidelines versions listed above is important to maintain consistency between data validation and qualification of data currently being performed and future work to be performed under the RMAP program. It should be noted that the US EPA National Functional Guidelines, which were developed for the validation of data generated according to the Contract Laboratory Program (CLP), are not directly applicable to the type of analysis/protocols associated with the analysis for this project. EPA National Functional Guidelines qualifies data based on strict contractual CLP method requirements and acceptance criteria which may not be consistent with the requirements and acceptance criteria presented in SW-846 methods. Data validators will apply

EPA guidelines as appropriate, assess the data relative to method QC protocols and DQOs in this QAPP, and use professional judgment according to the documents listed above.

5.2 Verification and Validation Methods

The Level A/B Assessment checklists included in Attachment D are based on the *CFRSSI Data Management/Data Validation Plan Addendum* (AERL, 2000b) guidance and will be used for field data verification as detailed in Section 5.1.2.

Data qualifiers will follow those used in the EPA *National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2020b). Data validation for each laboratory data package will be documented on the data validation checklists based on the *CFRSSI Data Management/Data Validation Plan Addendum* (AERL, 2000b) guidance (Attachment H).

The Data Validator will be responsible for reviewing field documentation associated with sample collection, conducting the verification and validation of laboratory-produced data, and completing a data validation report, which will be reviewed by the Human Health/RMAP Division Manager and QA Manager.

Qualifiers that may be applied to the data during the data validation process include the following:

U	The result is qualified as non-detect due to the detection of the analyte in an associated QC blank.
J	The analyte was positively identified; the associated numerical value is an estimate of the concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
J-	The result is an estimated quantity, but the result may be biased low.
UJ	The analyte was not detected above the sample reporting limit. However, the reporting limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
R	The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.
No Flag	Result accepted without qualification.

5.2.1 Differences Between Stage 2B and Stage 4 Validation

The content and scope of the Stage 2B and Stage 4 data validation will be performed with guidance from *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, OSWER No. 9200.1-85, EPA 540-R-08-005, 13* (EPA, 2009). The major difference between Stage 2B and Stage 4 data validation is the detail level of the data evaluation. Stage 4 data validation is an in-depth process that consists of a comparison between raw data and

summary forms to check for inconsistencies between reported data and raw data. Stage 2B data validation does not involve evaluating raw data or checking reported data and raw data and assumes that all results and recoveries are correctly reported.

Stage 2B and Stage 4 data validations and reports are generated by an initial reviewer on a per-SDG or sampling location basis from the complete Level 4 data package to ensure completeness and data usability of data packages. Level 2 data packages are a condensed version of final data prior to completion and receipt of Level 4 data packages. Level 3 data packages contain the same information as the Level 4 data packages with the exception that instrumental QC (i.e., instrument tunes and raw data) to support the sample and the QA/QC results are not provided. Each validation report is reviewed by a senior chemist for accuracy to ensure that the initial reviewer has rigorously evaluated the recoveries/results and applied the applicable qualifiers to the data.

5.2.2 Stage 2B and Stage 4 Validation Procedure

A comprehensive QA review will be performed to independently verify compliance with the required analytical protocols and to determine the qualitative and quantitative reliability of the data. Stage 4 data validation includes a detailed review and interpretation of the data generated by the laboratory. Stage 4 data validation includes the review of the summary forms for all QC procedures and all sample and quality control raw data (including instrument calibration) to support the results reported. The purpose of a Stage 2B validation is to qualify data based on identified data quality limitations.

For each of the inorganic constituents the Stage 4 Verification and Validation checks include an evaluation of the following, as applicable for each analytical method. A Stage 2B validation focuses solely on data usability and does not include a review of raw data.

- Completeness of laboratory data package.
- Requested analytical methods performed.
- Compliance with the QAPP, analytical method, and analyte list.
- Proper sample collection, custody, preservation, and handling procedures.
- Holding times.
- Reported detection limits.
- Dilution factors.
- Tuning.
- Instrument Calibration.
- Initial and Continuing Calibration Verification Standards.
- Initial and Continuing Calibration Blank Standards.
- ICP and ICP/MS interference check samples.
- Method blanks.
- LCSs.

- Reporting Limit Check Standard recoveries.
- Field duplicate results.
- MS/MSDs (pre-digestion and post-digestion for inorganics only).
- ICP/MS internal standard recoveries.
- ICP and ICP/MS serial dilutions.
- Results verification and reported detection limits.
- Sample Preparation and Analytical Run Logs.

5.2.3 Data Validation Ratios

A goal of 10% of the project data will undergo Stage 4 validation. Since the number of samples in each residential sampling may vary depending on the size of the property and extent of sampling, the Stage 4 validation will be performed on the first complete data package and the 10% frequency monitored and additional Stage 4 validation performed to ensure the 10% goal is maintained throughout the sampling year. This process will ensure Stage 4 validation is performed periodically and consistently throughout the entire sampling event. This approach should allow the data validator to identify and have the laboratory correct any non-compliances early in the data collection process. In the event significant problems or issues are identified during the 10% Stage 4 data validation effort, it may be necessary to increase the percent of data subjected to Stage 4 validation to ensure that all errors and non-compliances have been appropriately corrected. The remaining 90% of the data will be validated at a Stage 2B level. In addition, the Consultant Project Manager can also offer guidance or request a greater percentage of Stage 4 data validation as the required level of validation based on project DQOs.

5.3 Reconciliation and User Requirements

A Data Quality Assessment (DQA) process described in the *CFRSSI Data Management/Data Validation Plan Addendum* (AERL, 2000b) and the *Guidance for Data Quality Assessment, Practical Methods for Data Analysis EPA QA/G-9* (EPA, 2000) will be performed to determine whether the project-specific DQOs have been satisfied. The DQA consists of five steps that relate the quality of the results to the intended use of the data:

Step 1: Review DQOs and sampling design.
Step 2: Conduct preliminary data review.
Step 3: Apply statistical test(s) as described in this QAPP to the data set.
Step 4: Verify assumptions.
Step 5: Draw conclusions about the quality of the data (data report will not include interpretation of results but will state conclusions regarding the quality of the results).

If, as a result of the DQA process, it is determined that data do not satisfy all DQOs, then corrective action(s) should be recommended and documented in the data reporting. Corrective actions include, but are not limited to, revision of the DQOs based on the results of the investigation or collection of more information or data. It may be determined that corrective

actions are not required, or the decision process may continue with the existing data, with recognition of the data limitations.

The PARCCS data quality indicators (Section 2.7.2) will be used when conducting the DQA. If the PARCCS assessment satisfies the project DQOs, then usability of the data will follow the enforcement/screening/unusable data categories as described in the *CFRSSI Data Management/Data Validation Plan* (ARCO, 1992a):

1. Enforcement Quality (Unrestricted Use). Data Enforcement quality data may be used for all purposes under the Superfund program including the following: site characterization, health and safety, environmental evaluation/cost analysis, remedial investigation/feasibility study, alternatives evaluation, confirmational purposes, risk assessment, and engineering design.
2. Screening Quality (Restricted Use). Potential uses of screening quality data, depending upon their quality, include site characterization, determining the presence or absence of contaminants, developing or refining sampling and analysis techniques, determining relative concentrations, scoping and planning for future studies, engineering studies and engineering design, and monitoring during implementation of the response action.
3. Unusable Data. These data are not usable for Superfund-related activities.

Data that meet the Level A and Level B criteria and are not qualified as estimated or rejected during the data validation process are assessed as enforcement quality data and can be used for all Superfund purposes and activities. Data that meet only the Level A criteria and are not rejected during the data validation process can be assessed as screening quality data. Screening quality data can be used only for certain activities, which include engineering studies and design. Data that do not meet the Level A and/or B criteria and/or are rejected during the data validation process are designated as unusable. The data are assigned one of the following qualifiers:

E = Enforcement quality. No qualifiers or U qualifier and meets Level A and B criteria.
S = Screening quality. J or UJ qualifier and/or meets only Level A criteria.
R = Unusable. R qualifier and/or does not meet Level A or B requirements.

Enforcement/Screening Designation

	Meets Level A and B	Meets Level A	Does not meet Level A or B
No qualifier, A, or U, or laboratory results reported between the MDL and RL with a J qualifier	E	S	R
J, J+, J-, or UJ	S	S	R
R	R	R	R

Note: It is appropriate to note that sample results qualified as estimated "J" by the laboratory because the reported result is between the MDL and RL values are considered enforcement data if no other qualifiers were required during validation.

Results of the QA review and/or validation will be included in any subsequent report, which will provide a basis for meaningful interpretation of the data quality and evaluate the need for corrective actions. Furthermore, all data validation information including usability designations and qualifiers will be captured in the project database.

Evaluation of Results

The analytical results that have been validated in accordance with Sections 5.1 and 5.2 of this QAPP will be compared to the BPSOU residential action levels (Arsenic – 250 mg/kg, Lead – 1,200 mg/kg, Mercury – 147 mg/kg) for all work completed under this QAPP (see Table 1). Analytical results will be compared to the action levels and the three outcome statements below will be used for identifying data groupings for decision-making purposes. These statements assume the primary and field duplicate results are valid and not qualified for other QA/QC deficiencies. If either the primary and/or field duplicate sample are qualified for other reasons, professional judgement will be used with Agency engagement and approval in the decision-making process.

1. **Below Action Level:** Primary and field duplicate sample results are non-detect (MDL is less than the action level) or detected results are less than the action level(s).
2. **Above Action Level:** Primary and field duplicate sample results are greater than the action level(s).
3. **Mixed Outcome:** Primary and field duplicate sample results show one result is greater than the action level(s) and the other result is less than the action level(s). The sample results will be evaluated using the following criteria.
    a. If the RPD between the primary and field duplicate results is less than 35% and the results are unqualified for field duplicate precision, then the highest of the primary and duplicate results will be used for decision making.
    b. If the RPD between the primary and field duplicate results is greater than 35% and the results are qualified for field duplicate precision, the data are considered screening quality "S" in accordance with the QAPP. For exterior soils, repreparation and reanalysis of sample pairs will occur when the RPD is greater than 35%, and all other depth interval sample concentrations in the same yard component are less than the action level(s). In this scenario, if the RPD is greater than 35% and any other of the depth interval sample concentrations in the same yard component are above the action level(s), no further repreparation and reanalysis will be required. The concentration(s) above the action level will drive remedial action for that yard component.
    c. For interior soils, repreparation and reanalysis of the sample pairs will occur when the RPD is greater than 35%. For interior dust where sample mass is limited or where samples were collected using filter cartridges, repreparation and reanalysis of the sample pairs may not be possible; recollection of samples and analysis may be necessary. If resampling is not possible then the highest of the primary and duplicate results will be used for decision making.

If these conditions are met for soil samples, then both the parent and the field duplicate sample will be reprepared from the air-dried, sieved soil and reanalyzed by the laboratory.

Upon reanalysis, the revised results will be reviewed:

d. The parent sample and field duplicate sample results are below the action level(s), and the RPD is less than 35%, it will be determined the results are below the action level. If the RPD based on the reanalysis continues to be greater than 35%, the highest of the primary and duplicate results will be used for decision making.

4. Results near the Action Limit. Positive results within 5% of the action limit are acceptable if the results are not qualified and designated as Enforcement Quality "E", whereas screening level (i.e., J-qualified) data within 5% below the action level samples are redigested and reanalyzed to potentially obtain an unqualified, enforcement-quality result. If the results are qualified after initial validation, the sample will be re-extracted from the dried soil and reanalyzed, If sufficient soil remains to perform a sample specific MS/MSD, these QC samples will also be processed with the soil sample. Upon reanalysis if the result is above the Action Limit, the higher value will be used; If the results are below the action limit, the reanalysis result confirms the quantitated result from the original analysis. In the event any sample within the soil column is above an action limit, reanalysis will not be performed.

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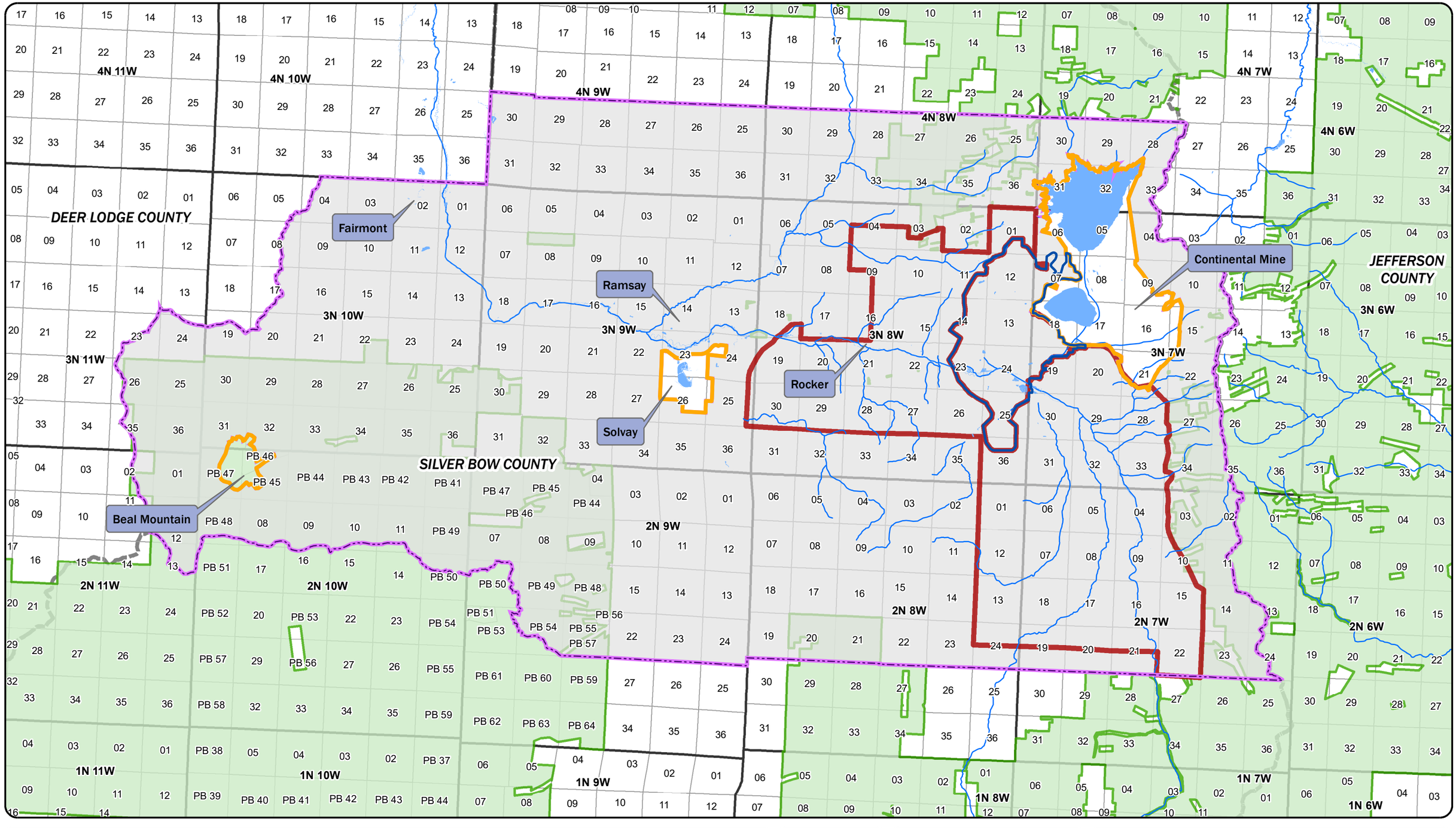
FIGURES



LEGEND

- BPSOU BOUNDARY
- 2020 RMAP BOUNDARY AREA
- AREAS EXCLUDED FROM RMAP (CONTINENTAL MINE, SOLVAY, BEAL MOUNTAIN)
- 2011 RESIDENTIAL METALS EXPANDED AREA
- COUNTY BOUNDARY
- US FOREST SERVICE

N

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UNITS: INTN'L FT
SOURCE: PIONEER/BSB/NRIS

0 1 2 4
Miles

FIGURE 1

2020 RMAP / BPSOU BOUNDARIES

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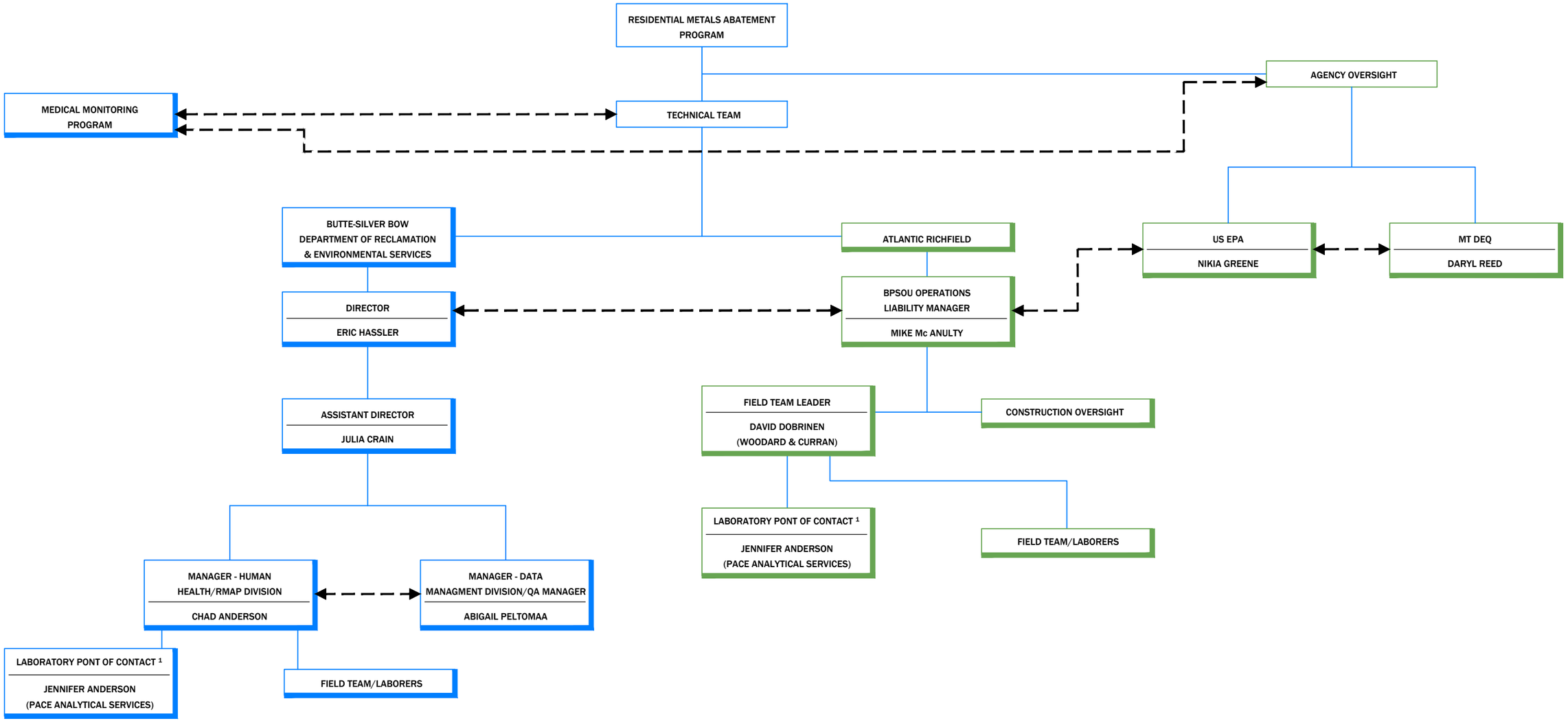

AUTHORITY

COMMUNICATION

1 LABS ARE CONTRACTED DIRECTLY TO AR AND BSB

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UNITS: NA
SOURCE: PIONEER

SCALE IN FEET
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FIGURE 2

RMAP ORGANIZATIONAL & COMMUNICATIONS STRUCTURE

DATE: 1/10/2023

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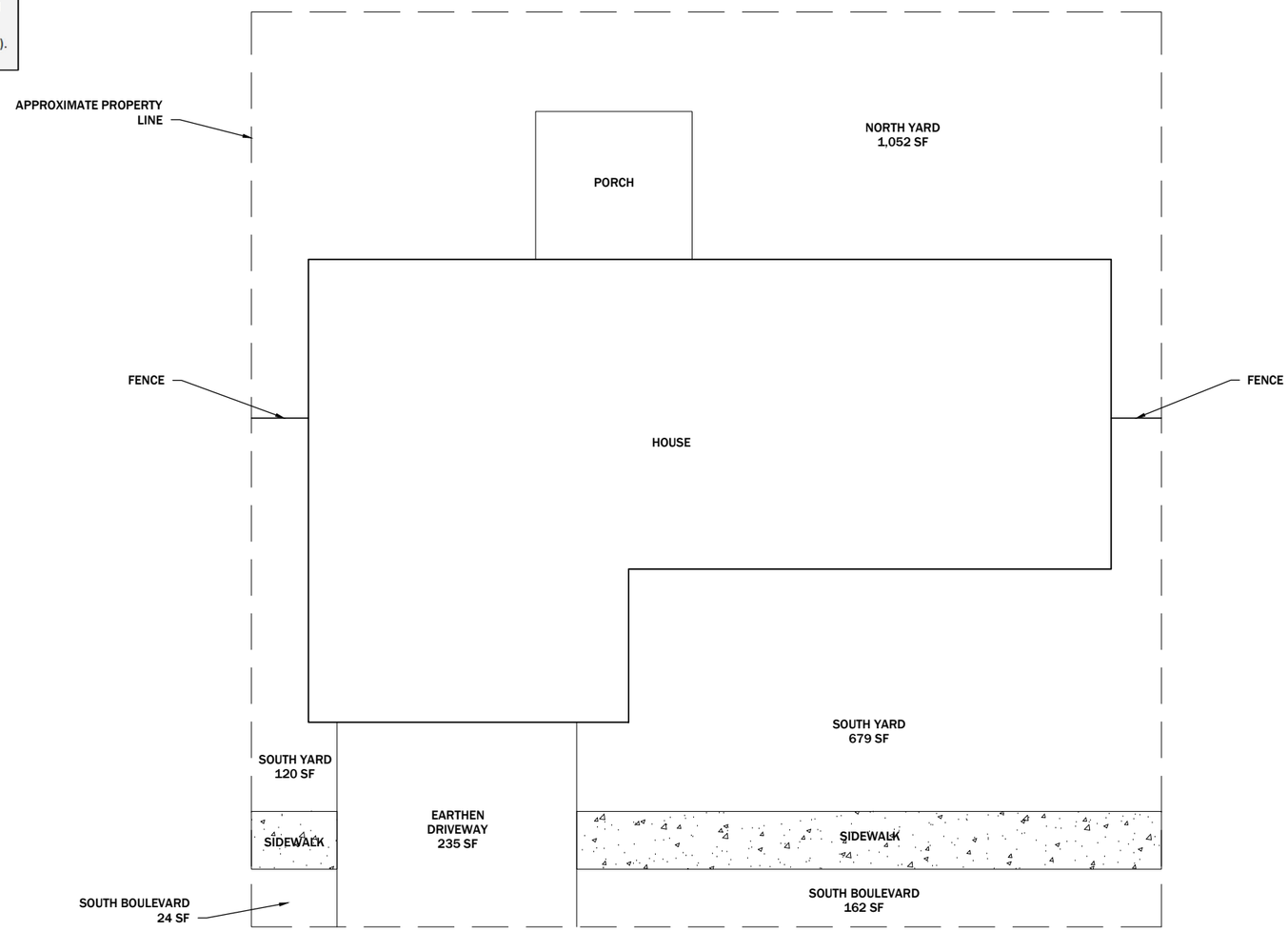
Example Type	Total Area (SF)	Polygon ID	Polygon Areas (SF)	# of Subsample Locations	# of Composite Samples	0-2"	2-6"	6-12"	12-18"	18-24"	Notes
Example Standard Residential Lot (< 5,000 sf)	4,000	North Yard 1	1,052	9	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Yard 1	799	7	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Boulevard	186	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		Earthen Driveway	235	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		Non-Sample Area (House/Porch/Sidewalk)	1,728	-	-	-	-	-	-	-	-
Totals (SF):	**4,000**	**-**	**4,000**	**22**	**12**						
Totals (AC):	**0.09**	**-**	**0.09**								

SAMPLE DESIGN LOGIC		
	Subsample Frequency	**Max Component Size**
Standard Residential Lots (< 5,000 sf)	3 subsamples minimum per component or 5 sub/625 sf (whichever is greater).	Max of 1,200 for all standard residential lot components (max of 10 subs/component).



LEGEND:

- PROPERTY LINE
- FENCE
- CONCRETE
- (SY) SOUTH YARD SUBSAMPLE
- (NY) NORTH YARD SUBSAMPLE
- (G) VEGETABLE GARDEN AREA SUBSAMPLE
- (DW) EARTHEN DRIVEWAY SUBSAMPLE
- (BA) BARE AREA SUBSAMPLE
- (G) FLOWER GARDEN SUBSAMPLE
- (SB) SOUTH BOULEVARD AREA SUBSAMPLE
- (PA) PLAY AREA SUBSAMPLE
- (SA) SOURCE AREA SUBSAMPLE

N

DISPLAYED AS:
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DATUM: NA
UNITS: NTS
SOURCE: PIONEER

SCALE IN FEET
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FIGURE 3

EXAMPLE STANDARD RESIDENTIAL LOT SOIL SAMPLE LOCATION MAP (<5,000 SF)

PIONEER TECHNICAL SERVICES, INC.
BUTTE, MONTANA
1101 SOUTH MONTANA
(406) 782-5177

DATE: 3/30/2022

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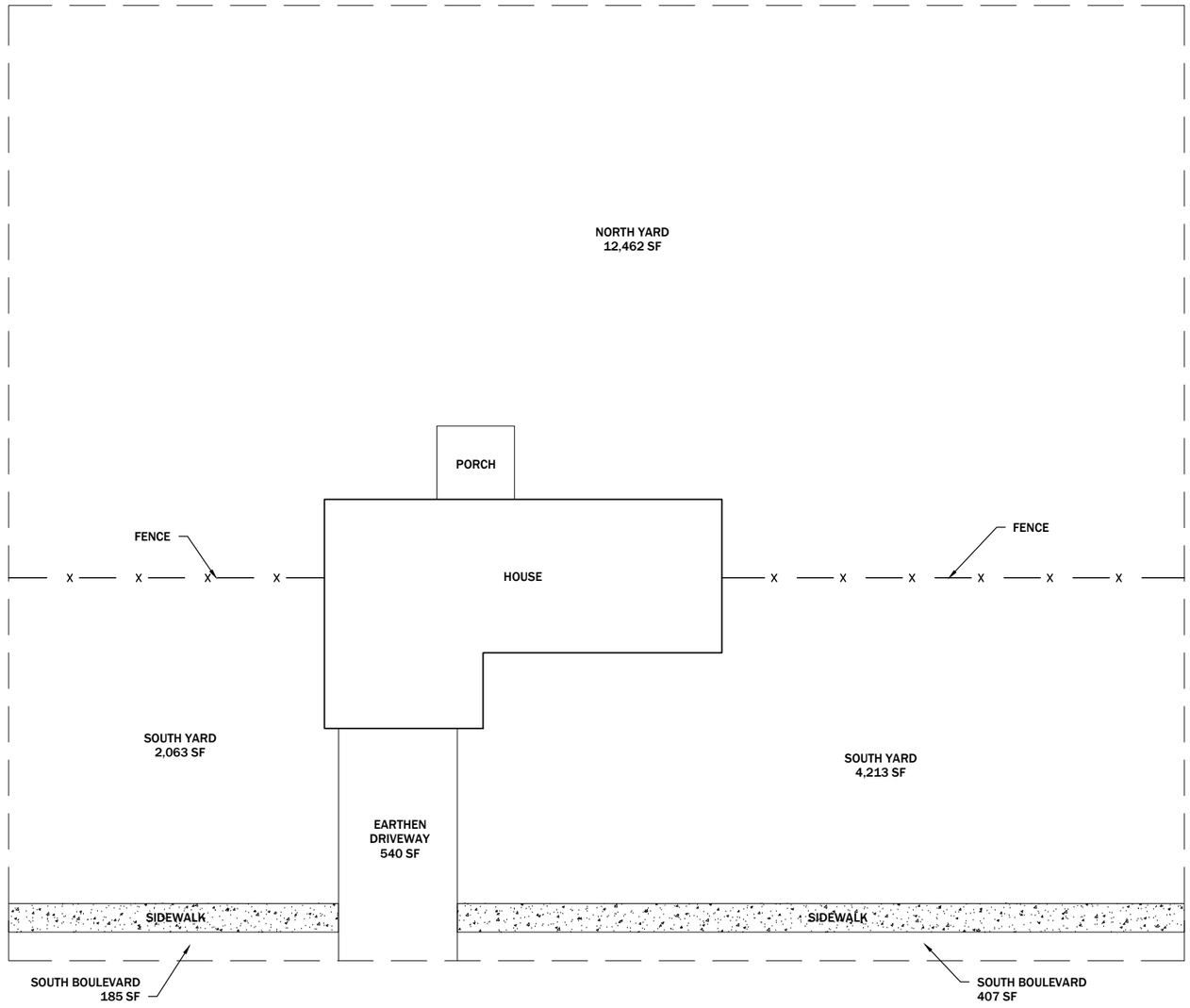
Example Type	Total Area (SF)	Polygon ID	Polygon Areas (SF)	# of Subsample Locations	# of Composite Samples	0-2"	2-6"	6-12"	12-18"	18-24"	Notes
Example Mid-Size Residential Lot (> 5,000 sf and < 1 acre)	22,000	North Yard 1	6,250	10	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		North Yard 2	6,212	10	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Yard 1	6,250	10	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Yard 2	26	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Boulevard	592	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		Earthen Driveway 1	540	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		Non-Sample Area (House/Porch/Sidewalk)	2,130	-	-	-	-	-	-	-	-
Totals (SF):	**22,000**	**-**	**22,000**	**39**	**18**						
Totals (AC):	**0.51**	**-**	**0.51**								

SAMPLE DESIGN LOGIC	Subsample Frequency	Max Component Size
Mid-Size Residential Lots (> 5,000 sf and < 1 acre)	3 subs minimum per component or 1 sub/625 sf (whichever is greater).	Max of 6,250 for all standard residential lot components (max of 10 subs/component).



LEGEND:

- PROPERTY LINE
- CONCRETE
- FENCE
- (SY) SOUTH YARD SUBSAMPLE
- (NY) NORTH YARD SUBSAMPLE
- (G) VEGETABLE GARDEN AREA SUBSAMPLE
- EARTHEN DRIVEWAY SUBSAMPLE
- (BA) BARE AREA SUBSAMPLE
- (G) FLOWER GARDEN SUBSAMPLE
- (SB) SOUTH BOULEVARD AREA SUBSAMPLE
- (PA) PLAY AREA SUBSAMPLE
- (SA) SOURCE AREA SUBSAMPLE

N

DISPLAYED AS:
COORD SYS/ZONE: NA
DATUM: NA
UNITS: NTS
SOURCE: PIONEER

SCALE IN FEET
0 NTS NTS

FIGURE 4

PIONEER TECHNICAL SERVICES, INC.
BUTTE, MONTANA
1101 SOUTH MONTANA
(406) 782-5177

EXAMPLE MID-SIZE RESIDENTIAL LOT SOIL SAMPLE LOCATION MAP (> 5,000 SF & < 1 ACRE)

DATE: 3/30/2022

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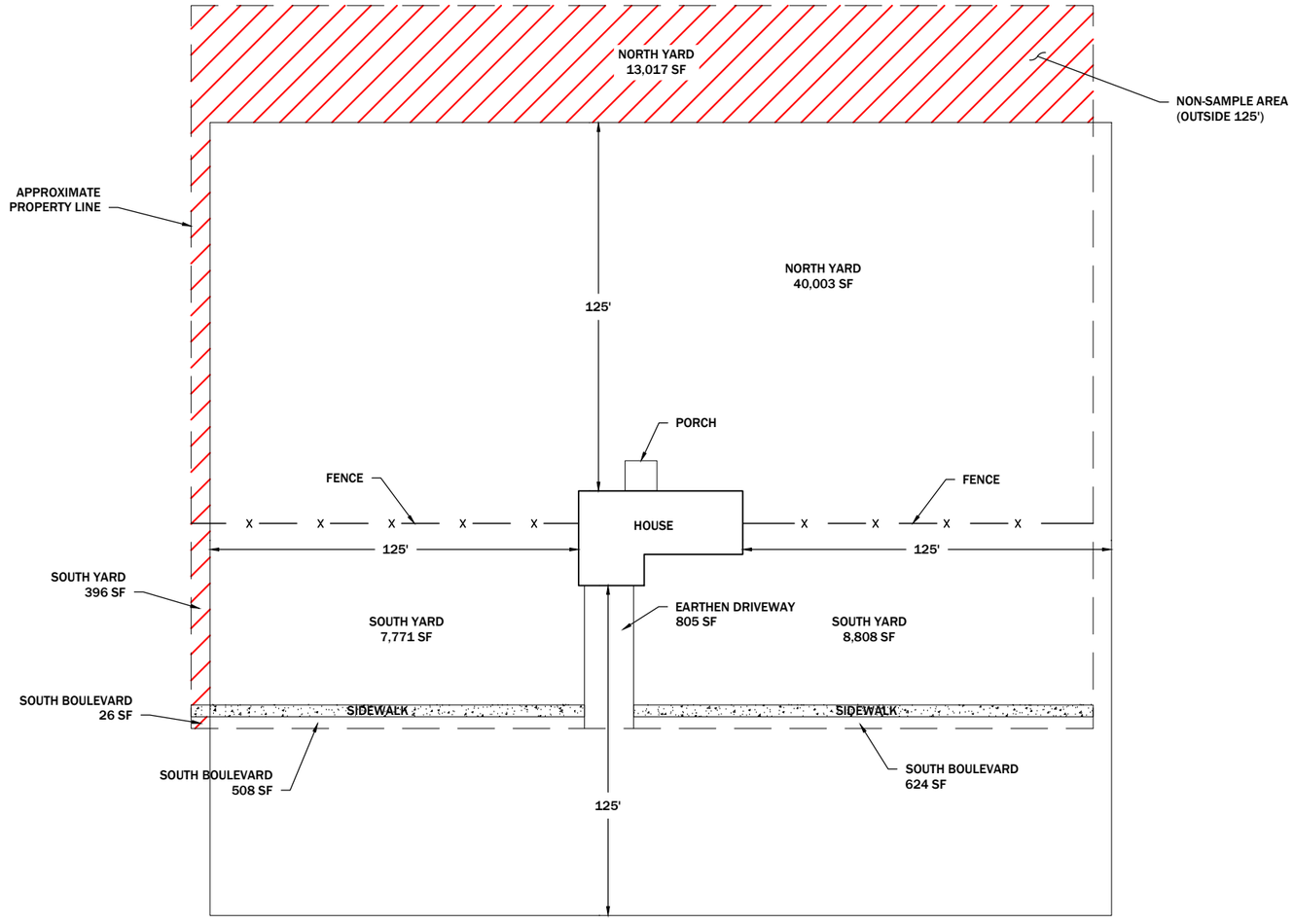
Example Type	Total Area (SF)	Polygon ID	Polygon Areas (SF)	# of Subsample Locations	# of Composite Samples	0-2"	2-6"	6-12"	12-18"	18-24"	Notes
Example Large Residential Lot (> 1 acre)	74,632	North Yard 1	10,890	5	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		North Yard 2	10,890	5	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		North Yard 3	10,890	5	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		North Yard 4	7,333	4	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Yard 1	10,890	5	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Yard 2	5,689	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		South Boulevard	1,132	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		Earthen Driveway 1	805	3	3	As/Pb/Hg	As/Pb/Hg	As/Pb/Hg	-	-	-
		North Yard (Non-Sample Area)	13,017	-	-	-	-	-	-	-	-
		South Yard (Non-Sample Area)	396	-	-	-	-	-	-	-	-
		South Boulevard (Non-Sample Area)	26	-	-	-	-	-	-	-	-
		Non-Sample Area (House/Porch/Sidewalk)	2,674	-	-	-	-	-	-	-	-
Totals (SF):	**74,632**	**-**	**74,632**	**33**	**24**						
Totals (AC):	**1.71**	**-**	**1.71**								

SAMPLE DESIGN LOGIC		
	Subsample Frequency	**Max Component Size**
Large Residential Lots (> 1 acre)	3 subs minimum per component or 1 sub/2,200 sf (whichever is greater).	Max of 10,890 sf (1/4 acre) for large lot components (max of 5 subs/component).



LEGEND:

- PROPERTY LINE
- FENCE
- CONCRETE
- (SY) SOUTH YARD SUBSAMPLE
- (NY) NORTH YARD SUBSAMPLE
- (G) VEGETABLE GARDEN AREA SUBSAMPLE
- (DW) EARTHEN DRIVEWAY SUBSAMPLE
- (BA) BARE AREA SUBSAMPLE
- (G) FLOWER GARDEN SUBSAMPLE
- (SB) SOUTH BOULEVARD AREA SUBSAMPLE
- (PA) PLAY AREA SUBSAMPLE
- (SA) SOURCE AREA SUBSAMPLE

N

DISPLAYED AS:
COORD SYS/ZONE: NA
DATUM: NA
UNITS: NTS
SOURCE: PIONEER

SCALE IN FEET
0 NTS NTS

FIGURE 5

EXAMPLE LARGE RESIDENTIAL LOT SOIL SAMPLE LOCATION MAP (> 1 ACRE)

PIONEER TECHNICAL SERVICES, INC.
BUTTE, MONTANA
1101 SOUTH MONTANA
(406) 782-5177

DATE: 3/30/2022

TABLES

TABLE 1: 2023 RMAP ACTION LEVELS AND SAMPLE PROTOCOL
(for RMAP Residential Parcels)

Contaminant of Concern:		Lead				Arsenic				Mercury						
Matrix	**Exposure Scenario**	**Action Levels Concentration**	**Analytical Methods**	**Reporting Limit (RL)**	**Method Detection Limits (MDLs)[1]**	**Action Levels Concentration**	**Analytical Methods**	**Reporting Limit (RL)**	**Method Detection Limits (MDLs)[1]**	**Action Levels Concentration**	**Analytical Methods**	**Reporting Limit (RL)**	**Method Detection Limits (MDLs)[1]**	**Sample Frequency**	**Sample Depth Intervals**	**Sample Density**
Soil	Standard Residential Lots (< 5,000 sf)	Residential - 1,200 mg/kg	EPA Methodology (EPA 6020B)	0.50 mg/kg	0.0931 mg/kg (6020B)	Residential - 250 mg/kg	EPA Methodology (EPA 6020B)	0.50 mg/kg	0.143 mg/kg (6020B)	Residential - 147 mg/kg	EPA Methodology (EPA 7471B)	0.02 mg/kg	0.008 mg/kg (7471B)	1 composite sample per component per depth interval	0-2 inches; 2 - 6 inches; and 6 - 12 inches[2]	Minimum of 3 subsample locations per component or 5 subsamples location per 625 sf (whichever is greater). Maximum area represented by a single composite sample is 1,200 sf (or 10 subsample locations).
Soil	Mid Sized Residential Lots (> 5,000 sf and < 1 acre)															Minimum of 3 subsample locations per component or one subsample location per 625 sf (whichever is greater). Maximum area represented by a single composite sample is 6,250 sf (or 10 subsample locations).
Soil	Large Residential Lots (> 1 acre)															Minimum of 3 subsample locations per component or one subsample location per 2,200 sf (whichever is greater). Maximum area represented by a single composite sample is 10,890 sf (or 5 subsample locations).
Dust	Residential		EPA Methodology (EPA 6020B) or Thermo Fisher Scientific Niton XL2 analyzer XRF Manufacturer Procedures/EPA Method 6200 in emergency situation)	0.50 mg/kg and 0.035 mg/kg by XRF (6200)	0.0931 mg/kg (6020B)		EPA Methodology (EPA 6020B or Thermo Fisher Scientific Niton XL2 analyzer XRF Manufacturer Procedures/EPA Method 6200 in emergency situation)	0.50 mg/kg and 0.035 mg/kg by XRF (6200)	0.143 mg/kg (6020B)					Site specific		
Paint	Residential	N/A[3]	Niton XLp 300 Series XRF Manufacturer Procedures		1 mg/cm^2	N/A	N/A		N/A	N/A	N/A	N/A	N/A	Site specific		
Water	Residential	15 µg/L[4]	EPA 200.8	1 ug/L	0.23 µg/L	N/A	N/A		N/A	N/A	N/A	N/A	N/A	Site specific		
Air	Residential (Vapor)	N/A	N/A		N/A	N/A	N/A		N/A	0.43 µg/m^3	Mercury Tracker-3000 Manufacturer Procedures (real time data collection)	N/A	0.1 µg/m^3 [5]	Site specific		

[1] - Detection limits will be re-evaluated and may change on a quarterly basis, but will typically be within ±5 mg/kg of the values listed above.

[2] - Flower & Vegetable Gardens require additional samples at 12-18" and 18-24".

[3] - Presence of lead paint is defined by HUD as > than 1.0 mg/cm^2.

[4] - MDEQ action level is 15 µg/L.

[5] - Tracker 3000 User's Manual (Attachment G-2, Section 7.1).

TABLE 2: RMAP LEAD BASED PAINT DEFINITIONS

	Total area of deteriorated paint on each component		
Type of building component	Intact*	Fair*	Poor*
Exterior components with large surface areas	Entire surface is intact	Less than or equal to 10 square feet	More than 10 square feet
Interior components with large surface areas (walls, ceilings, floors, doors)	Entire surface is intact	Less than or equal to 2 square feet	More than 2 square feet
Interior and exterior components with small surface areas (window sills, baseboards, soffits, trim, etc.)	Entire surface is intact	Less than or equal to 10 percent of the total surface area of the component	More than 10 percent of the total surface area of the component

*Intact surfaces require only monitoring and are not considered lead-based paint hazards.
*Surfaces in fair condition may be repaired and/or monitored but are not considered to be lead-based paint hazards.
*Surfaces in poor condition are considered to be lead-based paint hazards and should be addressed through remediation or interim controls.

TABLE 3: PRECISION, ACCURACY AND COMPLETENESS CALCULATION EQUATIONS

Characteristic	Formula	Symbols
Precision (as relative percent difference, RPD)	$RPD = \frac{(x_i - x_j)}{\left(\frac{x_i + x_j}{2}\right)} \times 100$	x_i, x_j: replicate values of x
Precision (as relative standard deviation, RSD, otherwise known as coefficient of variation)	$RSD = \frac{\sigma}{\bar{x}} \times 100$	σ: sample standard deviation $\bar{x}$: sample mean
Accuracy (as percent recovery, R, for samples without a background level of the analyte, such as reference materials, laboratory control samples and performance evaluation samples)	$R = \frac{x}{t} \times 100$	x: sample value t: true or assumed value
Completeness (as a percentage, C)	$C = \frac{n}{N} \times 100$	n: number of valid data points produced N: total number of samples taken

TABLE 4: QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA

Analyte	Method	Residential Action Limit (mg/Kg)	Method Detection Limit (MDL) (mg/Kg)[1]	Reporting Limit (RL) (mg/Kg)[1]	Laboratory Control Sample (LCS) Recovery Limits	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recovery Limits[2]	MS/MSD Relative Percent Different (RPD)[2]	Laboratory Duplicate Precision	Field Duplicate Precision[3]
Arsenic	Method 6020AB	1,200	0.156	0.50	80-120%	75-125%	± 20%	± 20%	± 35%
Lead		250	0.0870	0.20	80-120%	75-125%	± 20%	± 20%	± 35%
Mercury	Method 7471A	147	0.00868	0.02	80-120%	75-125%	± 20%	± 20%	± 35%

Analyte	Method	MDEQ Action Level (mg/L)	Method Detection Limit (MDL) (mg/L)[1]	Reporting Limit (RL) (mg/L)[1]	Laboratory Control Sample (LCS) Recovery Limits	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recovery Limits[2]	MS/MSD Relative Percent Different (RPD)[2]	Laboratory Duplicate Precision	Field Duplicate Precision[3]
Lead In Drinking Water	Method 200.8	0.015 mg/L	0.0000409 mg/L		85-115%	1 per batch of 10 or fewer samples for 200.8 80-120%	n/a[4]	± 20%	± 20%

TABLE 4: QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA

Analyte	Method	HUD LBP Definition (mg/cm^2)	Method Detection Limit (mg/cm^2)	Reporting Limit (RL) (mg/cm^2)[1]	Lead Reference Standard Recovery Limits	Lead Blank Analysis Limits	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recovery and RPD Limits[2]	Laboratory Duplicate Precision	Field Replicate Precision[5]
Lead In Paint by X-ray Fluorescence	Method 6200 and Attachment H of QAPP	1 mg/cm^2	n/a	1 mg/cm^2	± 20% (Within NIST acceptance limits for calibration check std SRM 2573)	< RL	n/a	n/a	± 35%

Analyte	Method	Action Level (ug/m^3)	Method Detection Limit (ug/m^3)	Reporting Limit (RL) (ug/m^3)[1]	Laboratory Control Sample (LCS) Recovery Limits	Matrix Spike/Matrix Spike Duplicate (MS/MSD) Recovery Limits[2]	MS/MSD Relative Percent Different (RPD)[2]	Laboratory Duplicate Precision	Field Replicate Precision[5]
Mercury in Air	Mercury Tracker 3000	0.43 ug/m^3	0.1 ug/m^3		n/a	n/a	n/a	n/a	n/a

TABLE 4: QUALITY CONTROL SAMPLE ACCEPTANCE CRITERIA

Notes:

[1] The MDLs and RLs are considered the laboratory base values. Soil samples for arsenic and lead will be dried prior to sample digestion and will not be dry weight corrected. Sample results for mercury will be reported on a dry weight basis, since soil samples will be digested on an "as received basis. MDLs and RLs may also be affected based on the actual weight of sample digested and potential dilutions required for high concentration samples. Aqueous sample MDLs and RLs may be affected by sample dilutions. The BPSOU residential action levels (Arsenic – 250 mg/kg, Lead – 1,200 mg/kg, Mercury – 147 mg/kg) will be utilized for all work completed under this QAPP.

[2] The percent recovery for each analyte in the MS and MSD and the RPD should be within the limits on the table with the exception when native sample results exceed the concentration of the added spike by 4 or more. Sample results will not be qualified in the event of this condition.

[3] The RPD field precision goal for soil field duplicates will be 35% for sample pairs with both sample results being greater than 5 times the reporting limit (RL). For soil field duplicate/primary sample pairs with 1 or both sample results being less than 5 times the RL, an absolute difference of less than or equal to 2 times the RL (difference ≤ 2xRL) will be used as the precision goal.

[4]Individual matrix spike samples are prepared for method 200.8 on a frequency of 1 per batch of 10 or fewer samples. No MSD is prepared and therefore MS/MSD RPD cannot be determined.

ATTACHMENT A

QAPP CROSSWALK

EPA REGION 8 QA DOCUMENT REVIEW CROSSWALK

QAPP/FSP/SAP for: *(check appropriate box)* ☐ **GRANTEE** ☐ **CONTRACTOR** ☐ **EPA** ☐ **Other**	**Entity** *(grantee, contract, EPA AO, EPA Program, Other)* BSB County and AR	**Regulatory Authority and/or Funding Mechanism**	___ **2 CFR 1500 for Grantee/Cooperative Agreements** ___ **48 CFR 46 for Contracts** ___ **Interagency Agreement** ___ **EPA/Court Order** ___ **EPA Program Funding** ___ **EPA Program Regulation** ___ **EPA CIO 2105**
Document Title *[Note: Title will be repeated in Header]*	BPSOU Final Residential Metals Abatement Program QAPP (Residential Parcels) (7/14/2023)		
QAPP/FSP/SAP Preparer	AR and BSB County		
Period of Performance *(of **QAPP**/FSP/SAP)*	**2023**	**Date Submitted for Review**	01/10/2022 (Draft Final) 09/30/2022 (Final) 07/14/2023 (Draft Final)
EPA Project Officer **EPA Project Manager**	Nikia Greene	**PO Phone #** **PM Phone #**	406-457-5019
QA Program Reviewer or Approving Official	Nikia Greene	**Date of Review**	2/28/2022

Documents Submitted for QAPP Review (QA Reviewer must complete)*:*

1. QA Document(s) submitted for review:

QA Document	Document Date	Document Stand-alone	Document with QAPP
QAPP	**6/29/17**	**Yes** / No	
FSP		Yes / No	Yes / No
SAP		Yes / No	Yes / No
SOP(s)	**(attached)**		**Yes** / No

2. WP/SOW/TO/PP/RP Date _____
WP/SOW/TO/RP Performance Period _____

3. QA document consistent with the:
WP/SOW/PP for grants? Yes / No
SOW/TO for contracts? Yes / No

4. QARF signed by R8 QAM Yes / No / NA
Funding Mechanism IA / contract / grant / NA
Amount _____

Notes for Document Submittals:

1. A QAPP written by a Grantee, EPA, or Federal Partner must include for review: Work Plan(WP) / Statement of Work (SOW) / Program Plan (PP) / Research Proposal (RP) and funding mechanism

2. A QAPP written by Contractor must include for review:
- **a)** Copy of Task Order Work Assignment/SOW
- **b)** Reference to a hard or electronic copy of the contractor's approved QMP
- **c)** Copy of Contract SOW if no QMP has been approved
- **d)** Copy of EPA/Court Order, if applicable
- **e)** The QA Review must determine (with the EPA CO or PO) if a QARF was completed for the environmental data activity described in the QAPP.

3. a. Field Sampling Plan (FSP) and/or Sampling & Analyses Plan (SAP) must include the Project QAPP ***or*** must be a stand-alone QA document that contain all QAPP required elements (Project Management, Data Generation/Acquisition, Assessment and Oversight, and Data Validation and Usability).

c. SOPs must be submitted with a QA document that contains all QAPP required elements.

1. This crosswalk has been updated to reflect this 2022 review and provided along with an official comment letter. The BSB County and AR must address the comments provided in the submitted comment letter and include a "Response (date)" and Resolved (date)".

Element	Acceptable *Yes/No/NA*	Page/ Section	Comments
A. Project Management			
A1. Title and Approval Sheet			
a. Contains project title	Yes	Title page and page i	EPA: No comments.
b. Date and revision number line (for when needed)	Yes	Title page and page i	EPA: No comments
c. Indicates organization's name	Yes	Title page	EPA: No comments.
d. Date and signature line for organization's project manager	Yes	Page i	EPA: No comments.
e. Date and signature line for organization's QA manager	Yes	Page i	EPA: No comments
f. Other date and signatures lines, as needed	Yes	Page i	EPA: No comments.
A2. Table of Contents			
a. Lists QA Project Plan information sections	Yes	Pages iii to vi	EPA: No comments.
b. Document control information indicated	Yes	Page v	EPA: No comments.
A3. Distribution List			
Includes all individuals who are to receive a copy of the QA Project Plan and identifies their organization	Yes	Page ii	EPA: No comments.
A4. Project/Task Organization			
a. Identifies key individuals involved in all major aspects of the project, including contractors	Yes	Sections 2.0 through 2.3	EPA: No comments.
b. Discusses their responsibilities	Yes	Sections 2.0 through 2.3	EPA: No comments.
c. Project QA Manager position indicates independence from unit generating data	Yes	Section 2.3, Figure 2	EPA: No comments.
d. Identifies individual responsible for maintaining the official, approved QA Project Plan	Yes	Section 2.3	EPA: No comments.
e. Organizational chart shows lines of authority and reporting responsibilities	Yes	Figure 2	EPA: See comments in comment letter regarding Figure 2. **Atlantic Richfield Response (9/30/22): Figure 2 has been updated to address Agency comments. See Comment Response letter.**
A5. Problem Definition/Background			
a. States decision(s) to be made, actions to be taken, or outcomes expected from the information to be obtained	No	Sections 1.0 and 2.5	EPA: No comments.

b. Clearly explains the reason (site background or historical context) for initiating this project	Yes	Sections 2.5 & 2.6	EPA: No comments.
c. Identifies regulatory information, applicable criteria, action limits, etc. necessary to the project	Yes	Section 2.1	EPA: No comments.
A6. Project/Task Description			
a. Summarizes work to be performed, for example, measurements to be made, data files to be obtained, etc., that support the projects goals	Yes	Sections 1.0 and 2.6	EPA: No comments.
b. Provides work schedule indicating critical project points, e.g., start and completion dates for activities such as sampling, analysis, data or file reviews, and assessments	No	Section 2.6	EPA: See comment letter. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
c. Details geographical locations to be studied, including maps where possible	Yes	Sections 1.0 and 2.6 , Figure 1	EPA: No comments.
d. Discusses resource and time constraints, if applicable	Yes	Section 2.6.1	EPA: No comments.
A7. Quality Objectives and Criteria			
a. Identifies - performance/measurement criteria for all information to be collected and acceptance criteria for information obtained from previous studies, - including project action limits and laboratory detection limits and - range of anticipated concentrations of each parameter of interest	No	Section 2.7.1	EPA: See comment letter. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
b. Discusses precision	Yes	Sections 2.7.2	EPA: No comments.
c. Addresses bias	Yes	Sections 2.7.2	EPA: No comments.
d. Discusses representativeness	Yes	Sections 2.7.2	EPA: No comments.
e. Identifies the need for completeness	Yes	Sections 2.7.2	EPA: No comments.
f. Describes the need for comparability	Yes	Sections 2.7.2	EPA: No comments.
g. Discusses desired method sensitivity	Yes	Section 2.7.2	EPA: No comments.
A8. Special Training/Certifications			
a. Identifies any project personnel specialized training or certifications	Yes	Section 2.8	EPA: No comments.

b. Discusses how this training will be provided	Yes	Section 2.8	EPA: No comments.
c. Indicates personnel responsible for assuring training/certifications are satisfied	Yes	Section 2.8	EPA: No comments.
d. identifies where this information is documented	Yes	Section 2.8	EPA: No comments.
A9. Documentation and Records			
a. Identifies report format and summarizes all data report package information	Yes	Section 2.9	EPA: No comments.
b. Lists all other project documents, records, and electronic files that will be produced	Yes	Section 2.9	EPA: No comments.
c. Identifies where project information should be kept and for how long	Yes	Section 2.9	EPA: No comments.
d. Discusses back up plans for records stored electronically	Yes	Section 2.9	EPA: No comments.
e. States how individuals identified in A3 will receive the most current copy of the approved QA Project Plan, identifying the individual responsible for this	No	Section 2.8	EPA: No comments.
B. Data Generation/Acquisition			
B1. Sampling Process Design (Experimental Design)			
a. Describes and justifies design strategy, indicating size of the area, volume, or time period to be represented by a sample	No	Section 3.0	EPA: See comment letter. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
b. Details the type and total number of sample types/matrix or test runs/trials expected and needed	No	Sections 3.2, 3.3, 3.4, and 3.5	EPA: See comment letter. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
c. Indicates where samples should be taken, how sites will be identified/located	No	Section 3.2.1	EPA: See comment letter **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
d. Discusses what to do if sampling sites become inaccessible	Yes	Section 3.1	EPA: No comments.
e. Identifies project activity schedules such as each sampling event, times samples should be sent to the laboratory, etc.	Yes	Sections 3.2, 3.3, 3.4, and 3.5	EPA: No comments.

f. Specifies what information is critical and what is for informational purposes only	Yes	Sections 3.1, 3.2, 3.3, and 3.4	EPA: No comments.
g. Identifies sources of variability and how this variability should be reconciled with project information	Yes	Step 6	EPA: No comments.
B2. Sampling Methods			
a. Identifies all sampling SOPs by number, date, and regulatory citation, indicating sampling options or modifications to be taken	No	Sections 3.2 and 3.3	EPA: See comment letter regarding specific SOP comments. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
b. Indicates how each sample/matrix type should be collected	Yes	Sections 3.1, 3.2, 3.3, and 3.4	EPA: No comments.
c. If in situ monitoring, indicates how instruments should be deployed and operated to avoid contamination and ensure maintenance of proper data	NA	NA	EPA: No in-situ instruments will be deployed.
d. If continuous monitoring, indicates averaging time and how instruments should store and maintain raw data, or data averages	NA	NA	EPA: No continuous monitoring instruments will be deployed.
e. Indicates how samples are to be homogenized, composited, split, or filtered, if needed	Yes	Section 3.7.2	EPA: No comments.
f. Indicates what sample containers and sample volumes should be used	No	Sections 3.1, 3.2, 3.3, and 3.4	EPA: See comment letter for comments regarding sample volumes. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
g. Identifies whether samples should be preserved and indicates methods that should be followed	Yes	Section 3.4.2	EPA: No comments.
h. Indicates whether sampling equipment and samplers should be cleaned and/or decontaminated, identifying how this should be done and by-products disposed of	No	Section 3.1.4, SOP DE-02, Manuals	EPA: See comment letter regarding decontamination procedures. **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
i. Identifies any equipment and support facilities needed	Yes	TBD	EPA: No comments.
j. Addresses actions to be taken when problems occur, identifying individual(s) responsible for corrective action and how this should be documented	Yes	Section 4.0	EPA: No comments.
B3. Sample Handling and Custody			

a. States maximum holding times allowed from sample collection to extraction and/or analysis for each sample type and, for in-situ or continuous monitoring, the maximum time before retrieval of information	Yes	Section 3.5	EPA: No comments.
b. Identifies how samples or information should be physically handled, transported, and then received and held in the laboratory or office (including temperature upon receipt)	Yes	Section 3.5	EPA: No comments.
c. Indicates how sample or information handling and custody information should be documented, such as in field notebooks and forms, identifying individual responsible	Yes	Section 2.9.3	EPA: No comments.
d. Discusses system for identifying samples, for example, numbering system, sample tags and labels, and attaches forms to the plan	Yes	Section 3.6	EPA: No comments.
e. Identifies chain-of-custody procedures and includes form to track custody	Yes	Section 2.9.3	EPA: No comments.
B4. Analytical Methods			
a. Identifies all analytical SOPs (field, laboratory and/or office) that should be followed by number, date, and regulatory citation, indicating options or modifications to be taken, such as sub-sampling and extraction procedures	Yes	Section 3.7, Table 1, Attachment B, Section 6	EPA: No comments
b. Identifies equipment or instrumentation needed	Yes	Section 3.10	EPA: No comments.
c. Specifies any specific method performance criteria	Yes	Sections 2.7.2 and Section 3.7.7	EPA: No comments.
d. Identifies procedures to follow when failures occur, identifying individual responsible for corrective action and appropriate documentation	Yes	Section 4.0	EPA: No comments.
e. Identifies sample disposal procedures	Yes	Section 3.9	EPA: No comments.
f. Specifies laboratory turnaround times needed	Yes	Section 4.3	EPA: No comments.
g. Provides method validation information and SOPs for nonstandard methods	Yes	Section 5.0	EPA: No comments.
B5. Quality Control			

a. For each type of sampling, analysis, or measurement technique, identifies QC activities which should be used, for example, blanks, spikes, duplicates, etc., and at what frequency	No	Sections 3.1, 3.2, 3.3, 3.4, and 3.8	EPA: No comments.
b. Details what should be done when control limits are exceeded, and how effectiveness of control actions will be determined and documented	Yes	Section 4.0	EPA: No comments.
c. Identifies procedures and formulas for calculating applicable QC statistics, for example, for precision, bias, outliers and missing data	No	3.8	EPA: No comments.
B6. Instrument/Equipment Testing, Inspection, and Maintenance			
a. Identifies field and laboratory equipment needing periodic maintenance, and the schedule for this	Yes	Section 3.10	EPA: No comments.
b. Identifies testing criteria	Yes	Section 3.10	EPA: No comments.
c. Notes availability and location of spare parts	Yes	Section 3.10	EPA: No comments.
d. Indicates procedures in place for inspecting equipment before usage	Yes	Section 3.10	EPA: No comments.
e. Identifies individual(s) responsible for testing, inspection and maintenance	Yes	Section 3.10	EPA: No comments.
f. Indicates how deficiencies found should be resolved, re-inspections performed, and effectiveness of corrective action determined and documented	Yes	Section 3.10	EPA: No comments.
B7. Instrument/Equipment Calibration and Frequency			
a. Identifies equipment, tools, and instruments that should be calibrated and the frequency for this calibration	Yes	Sections 2.7, 2.7.2, 3.9	EPA: No comments.
b. Describes how calibrations should be performed and documented, indicating test criteria and standards or certified equipment	Yes	Sections 2.7, 2.7.2, 3.9	EPA: No comments.
c. Identifies how deficiencies should be resolved and documented	Yes	Section 4.0	EPA: No comments.
B8. Inspection/Acceptance for Supplies and Consumables			
a. Identifies critical supplies and consumables for field and laboratory, noting supply source, acceptance criteria, and procedures for tracking, storing and retrieving these materials	Yes	Section 3.11	EPA: No comments.
b. Identifies the individual(s) responsible for this	Yes	Section 3.11	EPA: No comments.

B9. Use of Existing Data (Non-direct Measurements)			
a. Identifies data sources, for example, computer databases or literature files, or models that should be accessed and used	Yes	Section 5.0	EPA: No comments.
b. Describes the intended use of this information and the rationale for their selection, i.e., its relevance to project	Yes	Section 5.0	EPA: No comments.
c. Indicates the acceptance criteria for these data sources and/or models	Yes	Section 5.0	EPA: No comments.
d. Identifies key resources/support facilities needed	Yes	Section 5.0	EPA: No comments.
e. Describes how limits to validity and operating conditions should be determined, for example, internal checks of the program and Beta testing	Yes	Section 5.0	EPA: No comments.
B10. Data Management			
a. Describes data management scheme from field to final use and storage	Yes	Section 3.12	EPA: No comments.
b. Discusses standard record-keeping and tracking practices, and the document control system or cites other written documentation such as SOPs	Yes	Section 3.12	EPA: No comments.
c. Identifies data handling equipment/procedures that should be used to process, compile, analyze, and transmit data reliably and accurately	Yes	Section 3.12	EPA: No comments.
d. Identifies individual(s) responsible for this	Yes	Section 3.12	EPA: No comments.
e. Describes the process for data archival and retrieval	Yes	Section 3.12	EPA: No comments.
f. Describes procedures to demonstrate acceptability of hardware and software configurations	Yes	Section 3.12	EPA: No comments.
g. Attaches checklists and forms that should be used	Yes	Section 3.12	EPA: No comments, QAPP references the BPSOU Data Management Plan with a TBD completion data. **Atlantic Richfield Response (9/30/22): Document has been updated to reference the most current version of the BPSOU Data Management Plan.**
C. Assessment and Oversight			
C1. Assessments and Response Actions			

a. Lists the number, frequency, and type of assessment activities that should be conducted, with the approximate dates	Yes	Section 4.0	EPA: No comments.
b. Identifies individual(s) responsible for conducting assessments, indicating their authority to issue stop work orders, and any other possible participants in the assessment process	Yes	Section 4.0	EPA: No comments.
c. Describes how and to whom assessment information should be reported	Yes	Section 4.1 and 4.2	EPA: No comments.
d. Identifies how corrective actions should be addressed and by whom, and how they should be verified and documented	Yes	Section 4.1 and 4.2	EPA: No comments.
C2. Reports to Management			
a. Identifies what project QA status reports are needed and how frequently	Yes	Section 4.3	EPA: No comments.
b. Identifies who should write these reports and who should receive this information	Yes	Section 4.3	EPA: No comments.
D. Data Validation and Usability			
D1. Data Review, Verification, and Validation			
Describes criteria that should be used for accepting, rejecting, or qualifying project data	Yes	Section 5.0	EPA: See comment letter **Atlantic Richfield Response (9/30/22): Document has been updated to address Agency comments. See Comment Response letter.**
D2. Verification and Validation Methods			
a. Describes process for data verification and validation, providing SOPs and indicating what data validation software should be used, if any	Yes	Section 5.0	EPA: No comments.
b. Identifies who is responsible for verifying and validating different components of the project data/information, for example, chain-of-custody forms, receipt logs, calibration information, etc.	Yes	Section 5.0	EPA: No comments.
c. Identifies issue resolution process, and method and individual responsible for conveying these results to data users	Yes	Section 5.0	EPA: No comments.
d. Attaches checklists, forms, and calculations	Yes	Section 5.0	EPA: No comments.
D3. Reconciliation with User Requirements			

a. Describes procedures to evaluate the uncertainty of the validated data	Yes	Section 5.0	EPA: No comments.
b. Describes how limitations on data use should be reported to the data users	Yes	Section 5.0	EPA: No comments.

ATTACHMENT B

STANDARD OPERATING PROCEDURES

ATTACHMENT B-1

FIELD SOPs

Attachment B-1
Field SOPs
Index

SOP Number	SOP Title	# Pages
RMAP-SOP-1	Residential Yard Soil Sampling	4
RMAP-SOP-2	Earthen Basement Soil Sampling	2
RMAP-SOP-3	Attic/Crawl Space Dust Sampling	2
RMAP-SOP-4	Residential Living Space Dust Sampling	2
RMAP-SOP-5	Interior Air Monitoring	3
RMAP-SOP-DE-01	Personnel Decontamination Procedures	2
RMAP-SOP-DE-02	Soil Sampling Equipment Decontamination	2
RMAP-SOP-S-01	Surface Soil Sampling	5
RMAP-SOP-SA-01	General Soil and Water Sampling Packaging and Shipping	2
RMAP-SOP-SA-04	Chain of Custody Forms for Environmental Samples	3
RMAP-SOP-SA-05	Project Documentation	3

RMAP SOP-1

RESIDENTIAL YARD SOIL SAMPLING

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform site sampling tasks at residences under the Residential Metals Abatement Program (RMAP) within Butte Priority Soils Operating Unit (BPSOU) and the expanded area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sampling collection and handling, and chain of custody protocol required to complete RMAP residential yard soil sampling.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Pre-Sampling Tasks	1. Schedule time to conduct sampling with the property owner. 2. Request underground utility locates. 3. Have the property owner fill out the Sample Request Form. **4.** Record documentation per SOP SA-05.
2.Visual inspection and map	1. **A scaled map** of each yard or lot will be prepared ahead of the sampling event that shows property boundaries, house, garage, structures, driveways, contaminant source material, gardens, lawns, and patios. Measure yard features within an accuracy of approximately plus or minus 2.0 feet. The map will divide each yard into polygons (e.g., east yard, west yard) based on aerial images for sampling and identify these areas on the map. The map will be verified (and modified if necessary) in the field based on site-specific conditions. 2. **Visually inspect the property** to determine the number of sections needed for composite sampling. 3. **Photograph** and document the pre-removal condition of the specific areas (e.g., east, west, south, or north yards) of each park, play area, or residential yard identified for soils removal. 4. **Verify utility locates** have been performed and adjust sampling locations to avoid conflicts.
3.Sampling	1. Crew members will wear Level D PPE (hard hat, high visibility vest, hard toe boots, safety glasses). 2. Prepare and label a sample bag with the unique sample identification number for each composite sample. 3. **Collect the samples from a minimum of 3 depth intervals (placing each sample in the corresponding bag).** Use the soil probe equipped with the core collection tube to advance the sampling tube to 12 inches below ground surface (bgs). If unable to advance to 12 inches (hit refusal) or obtain poor core recovery, discard and move to a new location within close proximity to original location. Obtain a complete core from that subsample location. It is important to note that some soil types may become compressed, and you might not obtain a

complete 12 inch core. Ensure that the core tube was advanced to total depth of 12 inches bgs (can have a marker on the side of the core tube to visually see when depth has been achieved). Adjust the bgs depth accordingly for the sod mat. Sod mat thickness can vary, but generally ranges from 1" to 1.5" in well maintained lawn areas. Sampler will have a better gauge once the first subsample location has been cored.

Ensure that you have the appropriate nitrile gloves, or equivalent when handling samples for this step and the remaining steps requiring handling of samples. Each sample represents a depth interval below ground surface (bgs). The first depth interval being 0-2 inches bgs, the second being 2-6 inches bgs, the third being 6-12 inches bgs. Use the Core Correlation equation to split the core into the appropriate depth intervals:

R = L/H

R = Recovery

L = Length of Sample (inches)

H = Depth of Sample Interval (Inches)

i.e., Cored from 0"-12" and obtained a soil core of 9".

L = 9"; H = 12"

R = 9/12 = 0.75

How much to take for 0"-2" Sample Interval?

L = R*H = 2*0.75 = 1.5"

How much to take for 2"-12" Sample Interval?

L =R*H = 10*0.75 = 7.5"

Total Length = 9"

1.5+7.5 = 9 check (ok)

Collect subsamples from 5 subsample locations within each sample polygon (refer to QAPP for additional details) in an X pattern (if possible).

	a. The first composite sample will consist of subsamples from the 0-to-2 inch depth interval, b. The second composite sample will consist of subsamples from the 2-to-6 inch depth interval, and c. The third composite sample will consist of subsamples from the 6-to-12 inch depth interval. d. For Flower/Vegetable Garden components only, fourth and fifth composite samples will be collected from the 12-to-18 inch and 18-to-24 inch depth intervals. 4. Show the location of each subsample on the map. 5. **Homogenizing subsample in prep for collecting Hg split**. Close the Ziplock bag and hand knead to homogenize the sample collected as consistent with the QAPP. 6. **Collect subsample for Hg split**. Collect a split from the Ziplock sample bag and place in an appropriately labeled 4oz amber glass jar for Hg analysis. To further ensure homogenization and representativeness, the aliquots for the mercury subsample will be obtained from several areas of the homogenized sample bag using a clean scoop. 7. **Field Preservation of samples collected**. Place the labeled 4oz amber jar Hg sample in the bubble wrap and place on ice in the cooler while in the field. The As and Pb sample can be shipped at ambient temperature. Mercury samples can be held in the temperature monitored temporary sample storage refrigerator until shipment.
4. Sampling areas covered with grass	1. Collect the initial composite sub-sample from immediately beneath the vegetative mat (sod), or in the absence of vegetation 0 to 2 inches below ground surface (bgs). 2. If a vegetative mat is present, separate it from the soil surface with a stainless steel knife or equivalent, shake and scrape the removed vegetative mat over the sample collection bowl to dislodge any mineral soil particles. Include all dislodged soil particles in the composite sample. 3. Collect the remaining subsamples as described above.
5.Chain of Custody	**Prepare a chain-of-custody** for samples transferred to laboratory for analysis. Follow procedures outlined in SOP SA-04.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOP's / WORK PLANS	SOP SA-01 SOP SA-05 SOP SA-04 SOP S-01

	SOP DE-02 Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)
FORMS/CHECKLIST	RMAP Access Agreement Sample Request Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
CREW LEAD	**DATE**
CREW MEMBERS	**DATE**

Revisions:

Rev.	Description	Date	Approval

RMAP SOP-2

EARTHEN BASEMENT SOIL SAMPLING

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform site sampling tasks at residences under the Residential Metals Abatement Program (RMAP) within Butte Priority Soils Operating Unit (BPSOU) and the expanded area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sampling collection and handling, and chain of custody protocol required to complete RMAP soil sampling within residential earthen basements.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Pre-Sampling Tasks	1. Schedule time to conduct sampling with the property owner. 2. Have the property owner fill out the Sample Request Form. 3. Conduct a pre-sampling walk through of the residence with the property owner to determine a site-specific sampling plan including identifying the safest way to access the basement. **4.** Record documentation per SOP SA-05.
2.Visual inspection and map	1. **Visually inspect the basement area** to identify obvious hazards prior to sampling. 2. **Draw a scaled map and photograph** the area.
3.Sampling	1. Crew members will wear Level D PPE (hard hat, high visibility vest, hard toe boots, safety glasses). 2. Prepare and label a sample bag with the unique sample identification number for the basement composite sample. 3. Collect five surficial (0 to 2-inch) subsamples, composite them into 1 representative composite sample for the basement, and place it in the respective labeled bag. See QAPP for additional details.
4. Chain of Custody	1. **Prepare a chain-of-custody** for samples transferred to laboratory for analysis. Follow procedures outlined in SOP-SA-04.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT

The following documents should be referenced to assist in completing the associated task.

DRAWINGS	NA
RELATED SOP's / WORK PLANS	SOP SA-01 SOP SA-05 SOP SA-04 SOP S-01 SOP DE-02 Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)

FORMS/CHECKLIST	RMAP Access Agreement Sample Request Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
CREW LEAD	**DATE**
CREW MEMBERS	**DATE**

Revisions:

Rev.	Description	Date	Approval

RMAP SOP-3

ATTIC/CRAWL SPACE DUST SAMPLING

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform site sampling tasks at residences under the Residential Metals Abatement Program (RMAP) within Butte Priority Soils Operating Unit (BPSOU) and the expanded area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, and sample collection within RMAP attics/crawl spaces.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Pre-Sampling Tasks	1. Schedule time to conduct sampling with the property owner. 2. Have the property owner fill out the Sample Request Form. 3. Conduct a pre-sampling walk through of the residence with the property owner to determine a site-specific sampling plan including identifying the safest way to access the attic/crawl space. 4. Record documentation per SOP SA-05.
2. Visual Inspection / Documentation of Work Area	1. Open the access point to make access into the sampling area. 2. Visually inspect the area to identify obvious hazards prior to sampling. 3. Take notes and photographs to document site conditions prior to sampling and potential remedial action cleanup activities.
3. Sampling	1. Crew members will wear Level D PPE (hard hat, high visibility vest, hard toe boots, safety glasses). 2. Label an amber sampling jar for the sample with the appropriate sample identification number. 3. Move insulation and/or debris to find the dust. 4. Attic dust composite sampling (based on a minimum of 2 subsample locations within the attic) will be conducted using a scoop and brush as appropriate for the location. See QAPP for additional details. 5. The amount of dust and insulation present in the attic space will determine the sampling method used. Each sample will consist of at least the minimum amount of material required for laboratory analysis (minimum mass of 0.2 grams, ideally closer to 0.4 grams if site conditions allow). 6. Samples will be collected in 4-ounce amber glass sampling jars labeled using a unique sample identification number for tracking. 7. Special care will be taken to ensure enough sample volume is collected for both arsenic/lead and Mercury analysis. Ziplock bags will not be utilized due to static issues that could lead to lost sample volume. 8. Replace any disturbed insulation and close access point.

4. Chain of Custody	1. Prepare a chain-of-custody for samples transferred to laboratory for analysis. Follow procedures outlined in SOP-SA-04.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOP's / WORK PLANS	SOP SA-01 SOP SA-05 SOP SA-04 Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)
FORMS/CHECKLIST	RMAP Access Agreement Sample Request Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
CREW LEADER	**DATE**
CREW MEMBERS	**DATE**

Revisions:

Rev.	Description	Date	Approval

RMAP SOP-04

RESIDENTIAL LIVING SPACE DUST SAMPLING

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively site sampling tasks at residences under the Residential Metals Abatement Program (RMAP) within Butte Priority Soils Operating Unit (BPSOU) and the expanded area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sampling collection and handling, and chain of custody protocol required to complete RMAP residential living space dust sampling tasks.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Pre-Sampling Tasks	1. Schedule time to conduct sampling with the property owner. 2. Have the property owner fill out the Sample Request Form. 3. Conduct a pre-sampling walk through of the residence with the property owner to determine a site-specific sampling plan. 4. Record documentation per SOP SA-05.
2.Sampling	1. Crew members will wear Level D PPE (hard hat, high visibility vest, hard toe boots, safety glasses). 2. Dust sampling will be completed using a high volume small surface sampler (HVS3) or Agency-approved equal. 3. The sampling equipment shall be decontaminated according to manufacturer's instructions prior to each use. 4. Prepare a clean sampling bottle for sample collection by labeling it with the appropriate unique sample identification number. 5. Collect samples from the following locations at each residence to provide a composite sample of the minimum amount of material required for laboratory analysis (minimum mass of 0.2 grams, ideally closer to 0.4 grams if site conditions allow): a. The floor area directly inside the main entries. b. The floor areas in the most frequently occupied rooms (normally the living room and/or kitchen). c. The floors in the children's bedrooms. d. The floor areas adjacent to or under attic pathways. 6. See QAPP for additional details.
3. Chain of Custody	1. Prepare a chain-of-custody for samples transferred to laboratory for analysis. Follow procedures outlined in SOP-SA-04.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOP's / WORK PLANS	SOP SA-01 SOP SA-05 SOP SA-04 Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Properties)
FORMS/CHECKLIST	RMAP Access Agreement Sample Request Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
CREW LEAD	**DATE**
CREW MEMBERS	**DATE**

Revisions:

Rev.	Description	Date	Approval

RMAP SOP-5

INTERIOR AIR MONITORING

PURPOSE	Establish a uniform procedure to safely, consistently, and effectively perform site sampling tasks at residences under the Residential Metals Abatement Program (RMAP) within Butte Priority Soils Operating Unit (BPSOU) and the expanded area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sampling collection, equipment operation, and interpretation of interior residential air monitoring data. Monitoring for mercury is completed using a mobile, active sampling device.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1.Pre-Sampling Tasks	1. Schedule time to conduct sampling with the property owner. 2. Have the property owner fill out the Sample Request Form. 3. Conduct a pre-sampling walk through of the residence with the property owner to determine a site-specific sampling plan. 4. Record documentation per SOP SA-05.
2. Equipment acquisition	1. RMAP Field Team Leader contact the preferred supplied of the specialty equipment to request unit rental, calibration, set-up, and delivery of the unit. Mercury Instruments USA 8392 S. Continental Divide Rd. Suite: 102 Littleton CO, 80127 Phone (303) 972-1493
3. Device receipt and start-up	1. Sampling parameters are pre-set and calibration and accompanying documentation is provided by the supplier. The device is field ready upon delivery. 2. Field personnel perform sampling device start-up as described in the manufacturer's operating instructions and verify pre-set parameters.
4. Visual inspection	1. **Visually inspect the area** to identify obvious hazards prior to sampling
5.Sampling	1. Crew members will wear Level D PPE (hard hat, high visibility vest, hard toe boots, safety glasses). 2. Refer to manufacturer's operating manual for specific instructions. 3. Perform air monitoring and sample collection from areas listed below. a.) Area directly inside the main entry to the residence, b.) Area in the most frequently occupied room (normally living room or kitchen). c.) Area in the child's bedroom or another frequently occupied room if no children are present in the home. d.) Additional samples collected from basement area of exposed earthen

	basement soils if the mercury action level is exceeded in soil samples. 4. Perform sampling along area of interest (entryway, bedrooms, play areas, etc.). 5. Sampling personnel will perform the sampling by systematically walking throughout the living space making sure to vary the height of the sampling instrument to ensure proper characterization of the mercury concentrations within the residence. 6. Download sampling data from the device to the field computer.
6.Data review	1. Compare sample results to action levels listed in RMAP QAPP, Table 1.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
DRAWINGS	NA
RELATED SOP's / WORK PLANS	SOP SA-05 SOP-SA-04 Final Multi-Pathway Residential Metals Abatement Program Plan Final Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP) (Residential Parcels)
FORMS/CHECKLIST	Manufacturer calibration certification RMAP Access Agreement Sample Request Form

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as acknowledgement that I have received training on the procedure and associated competency testing.	
MANAGER	**DATE**
CREW LEADER	**DATE**
CREW MEMBERS	**DATE**

APPROVALS/CONCURRENCE	
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Revisions:

Rev.	Description	Date	Approval

RMAP-SOP-DE-01.
PERSONAL DECONTAMINATION PROCEDURES - GENERAL

PURPOSE	To provide standard instructions for decontamination of all personnel leaving a contaminated area.
SCOPE	This practice has been prepared for task trained personnel conducting work on unreclaimed sites within the BPSOU area. The tasks are general and are to be used in conjunction with published manufacturer and internal practices.

WORK INSTRUCTIONS

The following instructions are intended to provide general guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made. All work carried out under this SOP will be consistent with procedures and policies described within appropriate internal policies.

TASK	INSTRUCTIONS
1. Wash/ Remove outer contaminated items.	Remove nitrile or latex gloves by grasping the outside of the opposite glove near the wrist. Pull and peel the glove away from the hand, turning the glove inside out with the contaminated side now on the inside. Hold the removed glove in the opposite gloved hand. Slide one or two fingers of the ungloved hand under the wrist of the remaining glove. Peel glove off from the inside, creating a bag for both gloves. If wearing protective coveralls such as Tyvec suites, brush built up material off the suit, only if in designated decontamination zone. Unzip the coverall and begin rolling that outwards, rolling it down over your shoulders. Place both hands behind your back and pull down each arm until completely removed. Sit down and remove each shoe then roll the coveralls down (ensuring the contaminated side is not touched or comes into contact with clothing) over your knees until completely removed. Decontamination zones will be evaluated on a site specific basis by the field team leader. These zones will be utilized by crew members to remove and dispose of personal protective equipment (PPE). For instructions to remove additional PPE not described in this document, refer to the project's HASP. Wash with soap (nonphosphate) and tap water the outer, more heavily contaminated items, such as boots. Rinse the items in tap water.
2. Wash inner contaminated items.	If necessary, wash with soap (nonphosphate) and tap water the inner, less contaminated items. Rinse the items in tap water.
3. Store/ transport items.	Store/transport contaminated items in a separate designated area to prevent cross contamination prior to disposal.
4. Dispose of contaminated items.	Dispose of contaminated clothing and equipment in accordance with site/project, client, and/or federal and state requirements.
5. Contact the Safety and Health Manager.	For contaminants other than those found typically at uncontrolled hazardous waste sites, such as asbestos, PCB, PCE, etc. see the Safety and Health Manager.

Information about Emergency Decontamination	
1. During life-saving process.	If the decontamination procedure is essential to the life-saving process, decontamination must be performed immediately.
2. During heat-related illness.	If heat-related illness develops, protective clothing should be removed as soon as possible. Wash, rinse, and/or cut off protective clothing/equipment.
3. When medical treatment is needed.	If medical treatment is required to save a life, decontamination should be delayed until the victim is stabilized. Wrap the victim to reduce contamination of others.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	In general, the following items will be needed: soap, tap water, tarps, decontamination tubs, brushes, and sprayer. The Sampling and Analysis Plan (SAP) will describe additional items needed for decontamination, if required.
Forms/Checklist	

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as an acknowledgement that I have received training on the procedure and associated competency training	
Manager	**Date**
Lead Operator	**Date**
Operator	**Date**

RMAP-SOP-DE-02. SOIL SAMPLING EQUIPMENT DECONTAMINATION - GENERAL	REVISION: 0 PAGE 1 of 2

PURPOSE	To provide standard instructions for routine decontamination of direct sample, non-disposable soil sampling equipment (i.e., soil probes).
SCOPE	This practice has been prepared for general equipment decontamination to be used in conjunction with individual agency or company procedures.

WORK INSTRUCTIONS

The following instructions are intended to provide general guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made. All work carried out under this SOP will be consistent with procedures and policies described within appropriate internal policies.

TASK	INSTRUCTIONS
1.	Physically remove gross contamination from equipment by abrasive scraping and/or brushing.
2.	Wash equipment with non-phosphate detergent wipe with tap water and a stiff brush (as appropriate).
3.	Triple rinse with tap water.
4.	Place equipment on plastic sheeting or foil to air dry. Wrap equipment in foil or plastic wrap to transport or store.
Notes	
1.	The decontamination procedure described above is performed on the soil sampling probe prior to conducting soil sampling and at the conclusion of all soil sampling activities. The procedure is also used to decontaminate the soil probe between each residential property. Only gross contaminant removal occurs between different sampling sections of an individual residential property (such as between sampling of North Yard and South Yard).
2.	Location of decontamination activities will be determined by crew leader in the field based on site specific conditions. Decontamination solutions may be disposed of to the ground surface, in the same general area in which soil sampling occurred. Disposable supplies will be collected by the field team leader and disposed of at the BPSOU Mine Waste Repository or local landfill as appropriate.
3.	All equipment leaving the contaminated area of a site must be decontaminated. Decontamination methods include removal of contaminants through physical, chemical, or a combination of both methods. Decontamination procedures should be used in conjunction with other methods to prevent contamination of sampling and monitoring equipment.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	In general, the following items will be needed: scrapers, brushes, detergent wipes, water.
Forms/Checklist	

APPROVALS/CONCURRENCE By signing this document, all parties acknowledge the completeness and applicability of this SOP for its intended purpose. Also, by signing this document, it serves as an acknowledgement that I have received training on the procedure and associated competency training	
Manager	**Date**
Crew Lead	**Date**
Crew Members	**Date**

PURPOSE	To provide standard instructions for surface soil sampling for unreclaimed sites in the BPSOU area.
SCOPE	Work described in this procedure includes visual assessment and site documentation, sample collection and handling, and chain of custody protocol required to complete routine soil sampling tasks.
DEFINITIONS	Surface Sample: a surface sample is defined as a mineral soil sample collected from immediately beneath the vegetative mat. It generally includes some interval from the upper six inches of soil. Surface sampling under biased conditions may be selected after considering factors such as type of contaminant, length of time the area has been contaminated, the type of soil, and the past use of the area.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Grab/Opportunistic Sample	
Visual Inspection and map	1. Verify utility locates have been performed and adjust sampling sites to avoid conflicts. 2. Inspect the area for possible hazards prior to sampling. 3. Visually inspect the site to determine the number test areas for composite sampling 4. Photograph and document the existing site conditions. 5. Draw a scaled map of the site if a pre-sampling map hasn't been completed
	Note: Sample collection devices include stainless steel scoops or trowels, stainless steel probes, and disposable Teflon trowels. For inorganic contaminants, disposable plastic scoops will be used. These procedures may be modified in the field based on field and site conditions after appropriate annotations have been made in the field log book.
	Identify site-specific hazards and verify utility locates. 1. Crew members will wear Level D PPE (hard hat, high visibility vest, hard toe boots, safety glasses). 2. Perform utility locates or verify utility locates have been performed. 3. Walk through the site and determine any site-specific hazards associated with the sampling area. Discuss findings with sampling crew and note in the field logbook. 4. Verify the utility locate information by identifying where natural gas pipes or other utilities enter any structures on the property or if yard lights or street lights are present with no overhead lines. Determine if an underground sprinkling system is present, where applicable. If sample locations have not been assigned in the Sampling Analysis Plan (SAP), note the already marked and/or probable locations of underground utilities and try to avoid those areas when choosing sample locations. Also, note the location of overhead lines and overhead

	hazards and avoid those areas, if possible. 5. If sample locations are identified in the SAP, use the appropriate survey method to locate and mark the sample locations.
Test Pit Sampling	
1. Dig a 6 to 12-inch square pit.	Dig a 6 to 12-inch square pit to a depth of approximately 6 inches. The size and depth of the sample pit required depends on the amount of material needed for sample analysis and the interval to be sampled. If a sod mat is present, separate the sod mat from the mineral soil surface with the chosen sampling tool. Shake and scrape the removed sod mat over the sample collection bowl to dislodge any mineral soil particles. Place all dislodged particles in the sample. If the surface material is coarse-grained material, free of intermixed materials (i.e., graveled driveway), collect the sample from the appropriate layer below the protective barrier. However, if the graveled driveway, alley or lot contains soil/dust material on the surface, collect the sample from the appropriate interval. If the sample area is unvegetated, collect the sample material from the designated interval inches below ground surface.
2. Measure and mark the interval to be sampled.	Measure the interval to be sampled (e.g., 0-2 inches or 0-6 inches) with a stainless steel tape measure or a ruler and mark the appropriate interval.
3. Scrape the walls of the sample pit.	Scrape the walls of the sample pit within the marked interval with a decontaminated stainless steel trowel or scoop, a Teflon scoop, or a disposable plastic scoop to expose a clean surface.
4. Collect the sample.	Once the wall of the test pit has been cleaned, collect the sample by scraping the appropriate interval on the cleaned face of the pit with the sampling tool and placing the material in a decontaminated stainless steel bowl, or a new cleaned foil pan.
5. Remove coarse fragments from the bowl.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
6. Pack the samples.	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping and store in a cooler at 4°C or less. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with additional soil left over to be archived.
7. Record sampling information.	Record appropriate information about the sample collection in the field logbook.
8. Return all the removed dirt into	Return all the removed dirt into the hole and return the sample area to pre-sampling conditions.

the hole.	
9. Decontaminate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Stainless steel probe opportunistic sampling	
1. Collect the sample	Collect sample as per probe manufacturers instructions
2. Pack the sample	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less.
3. Record the sample	Record appropriate information about the sample collection in the field logbook.
4. Decontaminate sampling equipment	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Composite Sampling/ Test Pits	
Note	In many situations, a composite sample is more appropriate for sample collection than a grab sample. Several types of composite samples can be collected. A sampler can collect a biased composite sample by identifying specific spots within the sample area that appear to be contaminated or not contaminated and digging sample pits in those locations. Composite samples can also be collected randomly as defined in a SAP. Sub samples shall be collected in a three-point (triangular) pattern. At each point, a subsample of predetermined depth is collected. The diagonal distance between the points is commonly ten feet, depending on the area of soil homogeneity. The precise method for compositing the sample will be discussed in the SAP. Each sub sample test hole will be prepared and sampled in the manner discussed above under the Grab Sample section.
1. Collect composite samples.	Composite samples will consist of discrete aliquots of equal amounts of soil from each subsample location. The soil aliquots will be collected into a stainless steel bowl and thoroughly mixed. The sampler may also “eyeball” an equal amount of sample material from each hole into a resealable plastic bag (i.e., Ziploc®). The sample material would be thoroughly mixed between each sub sample pit and prior to placing in the appropriate sample containers.
2. Remove coarse fragments.	Remove all coarse fragments greater than 0.5 inches from the bowl. Mix the remaining material in the bowl with the sampling tool.
3. Pack the samples.	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less. Any remaining sample material will be returned to the sample holes. A sufficient quantity of soil will be collected in each sample container to provide for analysis with

	additional soil left over to be archived.
4. Record sampling information.	Record appropriate information about the sample collection in the field logbook.
5. Return all the removed dirt into the hole.	Return all the removed dirt into the hole and return the sample area to pre-sampling conditions.
6. Decontaminate the equipment.	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.
Composite Sampling Stainless Steel Probe	
1. Collect composite samples	Collect in the same triangular pattern and mix as described above. Collect samples as per probe manufacturers instructions
1. Pack the samples	Transfer the soil sample directly into the appropriate sample container according to SOP-SA-01 Soil and Water Sample Packaging and Shipping, label the samples, and store in a cooler at 4°C or less.
2. Record sampling information	Record appropriate information about the sample collection in the field logbook.
3. Decontaminate the equipment	Decontaminate sampling tools according to procedures outlined in SOP-DE-02 Equipment Decontamination.

ADDITIONAL HSSE CONSIDERATIONS This section to be completed with concurrence from the Safety and Health Manager.	
Required PPE	**Personnel Protection Equipment** (PPE): Hard hat, safety glasses, high-visibility work shirt or vest, long pants, work boots, nitrile gloves, and leather gloves.
Applicable SDS	**Safety Data Sheets** (SDSs) will be maintained based on-site characterization and contaminants.
Required Permits/Forms	Per site/project requirements.
Additional Training	Per site/project requirements.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	Map with site location and sample locations.
Related SOPs/ Procedures/ Work Plans	SOP-SA-01 Soil and Water Sample Packaging and Shipping and SOP-DE-02 Equipment Decontamination.
Tools	Sampling tools: stainless steel scoops or trowels, stainless steel probes, disposable Teflon trowels, disposable plastic scoops (for inorganic contaminants), stainless steel tape measure or a ruler, decontaminated stainless steel bowl or cleaned foil pan, one-

	quart plastic bag, sampling containers, and cooler. Field logbook.
Forms/Checklists	

PURPOSE	To provide standard instructions for soil and water sample packaging and shipping for unreclaimed sites in the BPSOU area.
SCOPE	Work described in this procedure includes instruction on the correct methods to package, ship and Chain of Custody documentation.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
1. Place the sample containers in Ziploc bags.	Based on the analytes requested (e.g., low level mercury, low level chromium, etc.), it may be necessary to place each filled sample container in separate Ziploc bags to prevent cross contamination; keep the container clean, dry, and isolated; and protect the sample label. In most cases, all sample containers collected from a specific sample location are placed in a large Ziploc bag and shipped together.
2. Package the samples.	Place samples in a cooler, which has been previously lined with a plastic bag. Surround the samples with non-contaminating packaging materials to reduce movement and absorb any leakage. Double bag the ice and place it in the cooler. Seal the plastic bag in the cooler to contain the samples, packing material, and ice.
3. Review and sign Chain of Custody forms.	The Field Team Leader or their designated representative will double check the Chain-of-Custody (CoC) forms to assure those samples recorded on the CoC forms are in the cooler. The Field Team Leader or the designated representative will then sign the CoC form to relinquish custody. One copy of the signed CoC form will remain with the Field Team Leader. Make a photocopy of the completed forms, if there are no carbon copies available.
4. Tape paperwork to cooler.	Place paperwork in a sealed Ziploc bag and tape it to the inside of the cooler lid.
5. Bag samples for separate analytical batches.	If the shipping cooler contains more samples than can be analyzed in one analytical batch, the laboratory may request that the samples in the cooler be bagged for separate analytical batches. This may be necessary so that the appropriate Quality Control/ Quality Assurance samples are included in each analytical batch. In this case, fill out separate COC forms for each batch and include the forms in the appropriate plastic bags. Place the COC forms for each batch in a sealed Ziploc bag. The COC forms for each batch should be placed at the top of the plastic bag so that they are clearly visible to laboratory personnel when they open the plastic bags.
6. Label the cooler.	Label the cooler with the appropriate labels to describe the content of the cooler (e.g., NOS, flammable liquids, flammable solids, this side up, fragile, etc.). Close the cooler and place the appropriate shipping labels (e.g., overnight shipping from Federal Express, UPS, or the U.S. Postal Service or equivalent) on the lid of the cooler.
7. Sign CoC seals.	The Field Team Leader or the designated representative will sign CoC seals and place the signed seals over the opening edge of the cooler.

8. Tape the cooler.	Place tape over the custody seals and around the cooler.
9. Transport the cooler.	Transport the cooler(s) to a secure storage, to the shipping agent, or directly to the laboratory. If shipping the cooler, follow established federal and state regulations depending on cooler content.
Note:	Bagging of samples and lining of coolers is not necessary, if samplers transport the samples directly to the laboratory.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT **The following documents should be referenced to assist in completing the associated task**.	
P&IDS	
Drawings	
Related SOPs/ Procedures/ Work Plans	As per individual site SAPs.
Tools	Plastic bags, Ziploc bags, non-contaminating packaging materials, tape, COC seals, ice, and cooler
Forms/Checklist	Chain of Custody forms.

PURPOSE	This SOP establishes the requirements for documenting and maintaining environmental sample chain of custody from point of origin to receipt of sample at the analytical laboratory. It is applicable from the time of sample acquisition until custody of the sample is transferred to an analytical laboratory.
SCOPE	This practice describes the responsibilities of the sampling crew and the required forms to ensure proper custody procedures have been completed.
DEFINITIONS	Chain of Custody: is an unbroken trail of accountability that ensures the physical security of samples, data, and records. Custody refers to the physical responsibility for sample integrity, handling, and/or transportation. Custody responsibilities are effectively met, if the samples are: • In the responsible individual's physical possession; • In the responsible individual's visual range after having taken possession; • Secured by the responsible individual so that no tampering can occur; or • Secured or locked by the responsible individual in an area in which access is restricted to authorized personnel only.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Project Manager's Responsibilities	The Project Manager is responsible for overall management of environmental sampling activities, designating sampling responsibilities to qualified personnel, and reviewing any changes to the sampling plan.
Field Team Leader's Responsibilities	The Project Manager may act as the Field Team Leader or may choose to appoint a Field Team Leader. The Field Team Leader is responsible for general supervision of field sampling activities and ensuring proper storage/transportation of samples from the field to the analytical laboratory. Chain of Custody forms will be reviewed for accuracy and completeness to preserve sample integrity from collection to receipt by an analytical lab by the Field Team Leader. The review of Chain of Custody forms may be delegated to qualified personnel. The Field Team Leader is responsible for sample custody until the sample has been properly relinquished as documented on the chain of custody form.
Field Sampler's Responsibilities	The Field Sampler is responsible for sample acquisition in compliance with technical procedures, initiating the Chain of Custody, and checking sample integrity and documentation prior to transfer. Field samplers are also responsible for initial transfer of samples consisting of physical transfer of samples directly to the internal laboratory or transferred to a shipping carrier, (e.g., United Parcel Service or Federal Express) for delivery.

Laboratory Technician's Responsibilities	The receiving Laboratory Technician is responsible for inspection of transferred samples to ensure proper labeling and satisfactory sample condition. Unacceptable samples will be identified and segregated. The Laboratory Project Manager will be notified. The Laboratory Technician will review the Chain of Custody for completeness and file as part of the project's permanent record.
Samples Handling and Chain of Custody Forms	All samples will be collected and handled in accordance with SOP-SA-01 Soil Sample Packaging and shipping, or methods described in the Sampling and Analysis Plan (SAP). Samples will be transported in insulated coolers with ice ('blue ice' is acceptable) as necessary to maintain temperature at 4 °C+/- 2 °C until receipt by the analytical laboratory. The Field Team Leader or designated Field Sampler shall initiate the Chain of Custody form for the initial transfer of samples. A Chain of Custody form will be completed and accompany every sample. The form includes the following information: • Project code; • Project name; • Samplers signature; • Sample identification; • Date sampled; • Time sampled; • Analysis requested; • Remarks; • Relinquishing signature, data, and time; and • Receiving signature, date, and time. The Field Sampler relinquishing custody and the responsible individual accepting custody shall sign, date, and note the time of transfer on the Chain of Custody form. The Field Sampler may identify the carrier and reference the bill of lading number in lieu of the transporter's signature. One copy of the Chain of Custody form shall be filed as a temporary record of sample transfer by the Field Sampler. The original form shall accompany the samples and shall be returned to the sampling entity as part of the contracted laboratory Quality Assurance/Quality Control (QA/QC) requirements. The original form will be filed as part of the project's permanent records. The Project Manager (or designee) shall track the Chain of Custody to ensure timely receipt of samples by an analytical laboratory.

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
Drawings	
Related SOPs/ Procedures/ Work Plans	SOP-SA-01 Soil and Water Sample Packaging and Shipping
Tools	Seals and labels; chain of custody forms; chain of custody seals (provided by contracted laboratory); packing and shipping materials; and cooler and ice.
Forms/Checklist	Chain of Custody Forms.

PURPOSE	This SOP establishes the requirements for documenting and maintaining field logbooks and photographs. These procedures apply from the time field work begins until site activities are completed.
SCOPE	This practice has been prepared as a basic guide for project documentation.

WORK INSTRUCTIONS

The following instructions are intended to provide sufficient guidance to perform the task in a safe, accurate, and reliable manner. Should these instructions present information that is inaccurate or unsafe, operations personnel must bring the issue to the attention of the Project Manager and the appropriate revisions made.

TASK	INSTRUCTIONS
Logbooks	A designated field logbook will be used for each field project. If requested by the Project Manager, use a separate field logbook for each field task within a larger project. Label each logbook with the project name, dates that it covers, and logbook number. Use a waterproof marker, such as a Sharpie©, to write down the information. The logbooks will be bound and have consecutively numbered pages. The information recorded in these logbooks shall be written in ink. Begin a new page for each days notes. Write on every line of the logbook. If a blank space is necessary for clarity, such as a change of subject, skip one line before beginning the new subject. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page. Draw a diagonal line on any blank spaces of four lines or more to prevent unauthorized entries. The author will initial and date entries at the end of each day. All corrections will consist of a single line-out deletion in ink, followed by the author's initials and the date. Information not related to the project should not be entered in the logbook. The language used in the logbook should be factual and objective. These bound logbooks shall include the following entries: 1. A description of the field task. 2. Time and date fieldwork started. 3. Location and/or a description of the work areas including sketches, if needed, any maps or references needed to identify locations, and sketches of construction activities. If the location has been documented in the logbook during/prior visits, only changes in conditions should be noted. 4. Names and company affiliations of field personnel. 5. Name, company affiliation or address, and phone number of any field contacts or official site visitors. 6. Meteorological conditions at the beginning of fieldwork and any ensuing changes in these conditions. 7. Details of the fieldwork performed and reference to field data sheets, if used. 8. Deviation from the task-specific Sampling and Analysis Plan (SAP), Work Plan (WP), or Standard Operating Procedures (SOP). 9. All field measurements made.

	10. Any field laboratory analytical results. 11. Personnel and equipment decontamination procedures, if appropriate. For any field sampling work, the following entries should be made: 1. Sample location and number. 2. Sample type and amount collected. 3. Date and time of sample collection. 4. Type of sample preservation. 5. Split samples taken by other parties. Note the type of sample, sample location, time/date, name of person for whom the split was collected, that person's company, and any other pertinent information. 6. Sampling method, particularly any deviations from the SOP. 7. Documentation or reference of preparation procedures for reagents or supplies that will become an integral part of the sample, if available. This information may not be available for water or soil sampling bottles that come preserved from the laboratory or for preservatives provided by the laboratory. Bottle blanks will need to be used to evaluate the provided reagents. 8. The laboratory where the samples will be sent. No bound field logbooks will be destroyed or thrown away even if they are illegible or contain inaccuracies that require a replacement document.
Photographs	Take photographs of field activities using a digital camera. Photographs should include a scale in the picture when practical. Telephoto or wide-angle shots will not be used, since they cannot be used in enforcement meetings. The following items shall be recorded in the bound field logbook or on a field data sheet for each photograph taken: 1. The photographer's name, the date, the time of the photograph, and the general direction faced. 2. A brief description of the subject and the fieldwork portrayed in the picture. 3. Sequential number of the photograph. An electronic copy and/or a hard copy of the photographs shall be placed in task files in the field office after each day of field activities. Supporting documentation from the bound field logbooks or field data sheets shall be photocopied and placed in the task files to accompany the photographs once the field activities are complete

DRAWINGS, DOCUMENTS, AND TOOLS/EQUIPMENT The following documents should be referenced to assist in completing the associated task.	
P&IDS	
Drawings	
Related SOPS/ Procedures/ Work Plans	
Tools	Field logbook, Sharpie©, black pen, digital camera, and field data sheets.
Forms/Checklist	

ATTACHMENT B-2

SOIL LABORATORY SOPs

Attachment B-2
Soil Laboratory SOPs
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3	Pace	ENV-SOP-MIN4-0056	4	10/06/21	Metals Preperation of Solid Samples for Analysis by ICP and ICP-MS by 3050B	11
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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-GBAY-0164 **Revision:** 00

Document Title: Soil Sieve

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-GBAY-0164 **Revision:** 00
Title: Soil Sieve

All dates and times are in Central Time Zone.

ENV-SOP-GBAY-0164-Rev.00 Soil Sieve

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Elizabeth Turner (007857)	Manager - Quality Program	09 Apr 2021, 02:09:58 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Chad Rusch (007163)	General Manager 2	08 Apr 2021, 09:50:26 AM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
TEST METHOD ENV-SOP-GBAY-0164
ISSUER: Pace ENV – Green Bay Quality – GBAY

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure for drying and sieving soil samples to obtain a portion of soil for analysis.

1.1 Target Analyte List and Limits of Quantitation (LOQ) - Not applicable to this SOP.

1.2 Applicable Matrices: Soils and sediments.

1.3 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method or non-analytical process.

2.0 Summary of Method

A sample is homogenized and air dried. After air-drying, the sample is then sieved through a selected sieve size. The portion that passes the sieve is then ready for analysis.

3.0 Interferences

Not applicable to this SOP.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of

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solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Hg Samples	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	28 Days
All Other Metals	Ziplock Bag	2 cups	Thermal: ≤ 6°C Chemical: None	6 Months
Organic Parameters	16 oz glass jar	2 cups	Thermal: ≤ 6°C Chemical: None	VOA 14 Days SVOA 7 Days

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP ENV-SOP-GBAY-0006 *Sample Management* (current revision or replacement). Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored at ambient temperature until sample preparation. Prepared samples (extracts, digestates, distillates, other) are stored at ambient temperature until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

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7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment*	Manufacturer / Vendor*	Catalog #*
Sieve Shaker	RO-TAP®	RX-29
Sieve Shaker	Gilson	SS-15
Sieve Shaker	Endecotts	Minor 200
Sieve Shaker	Endecotts	Octagon 200
Sieve	Gilson or equivalent	Stainless steel, #10, #60, or other as needed
Sieve catch pans and lids	Gilson or equivalent	Stainless steel
Bakers' racks	Restaurant Supply	To hold 18" x 26" trays
Drying fan	Various	Local Store
Mortar ceramic/porcelain	Cole-Parmer	60322
Pestle ceramic/porcelain	Cole-Parmer	60323

*Or Equivalent

7.2 Supplies

Supplies	Vendor	Model/Version
Aluminum Foil Cake Pan	Durable Packaging / Webstaurant	612604245
8x8 Ziploc Bags	Fisher Scientific	23700218
12x12 Ziploc Bags	Uline	S-14416
Freezer Paper	Fisher Scientific or equivalent	50-200-5215
Wooden Rolling Pin	Restaurant Supply	Local Store
Rubber Mallet	Various	Local Store
Scissors	Various	Local Store

*Or Equivalent

8.0 REAGENTS AND STANDARDS

Not applicable to this SOP.

9.0 PROCEDURE

9.1 Balance calibration must be verified daily prior to use. Refer to SOP ENV-SOP-GBAY-0115 *Support Equipment* (current revision or replacement).

9.2 For any USDA marked samples, refer to SOP ENV-SOP-GBAY-0121 *Regulated Soil Handling* (current revision or replacement). Containers will be labeled with a pink Regulated Soil sticker.

9.3 Pulling Samples

9.3.1 Batch the samples in the LIMS.

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9.3.2 Pull the samples from either the soil Walk-In Cooler or from the ambient storage area in the Physical Testing Lab and organize them in the order to be processed. Their location will be dependent on the analytical work, if any, that will be done after the sieving.

9.4 Create a new Dry Sieve Worksheet File.

9.4.1 Use the Dry Sieve Template in the Dry Sieve folder, and make sure to "Save As", using the Horizon Batch Number (HBN).

9.4.2 Fill in the drying information for each sample on the Worksheet.

9.5 Air Dry Samples

9.5.1 Wearing gloves, line a tray with freezer paper wax side down. Fold the sides of freezer paper up about 1- 1 1/2" on each side to form a "boat".

9.5.2 Label the freezer paper with the sample number. Place the entire sample on the freezer paper. Multiple trays may be used for drying if a large sample volume was received.

9.5.3 Entire sample does not need to be dried if excess volume was received. Sample must be homogenized before splitting. Return undried portion to original container.

9.5.4 Weigh and document remaining sample mass. Some projects may require this to be labeled as "Archive".

9.5.5 Spread the soil evenly. Break up all clumps into about 1/2" or less size pieces. This will speed the drying process and ease the disaggregation process prior to sieving. Continue this process for all samples in the set. Change gloves between each sample.

9.5.6 In the drying logbook record the sample numbers, date, time, temperature, and humidity when the samples are placed in the drying cabinet. Place the entire set in a drying cabinet to air dry overnight. Longer drying may be required for wetter samples.

9.6 Soil Disaggregation

9.6.1 After the samples are dried remove them from the drying cabinets. Record the date, time, temperature, and humidity in the drying logbook.

9.6.2 Place a tray on the counter. Pick any rocks, twigs or other foreign matter and set to the side of the freezer paper boat.

9.6.3 Disaggregate the soil. Disaggregation is the process of loosening the clumped soil. It is not meant to crush or reduce the natural particle size of the soil. Place a sheet of paper, wax side up, over the sample. Using a rolling pin, roll over the dried soil for 1-2 minutes. A rubber mallet or pestle may be used to disaggregate soil clumps. Take care that the sample remains on the freezer paper. If sample is hard clay, a porcelain pestle may be used to break up chunks, being careful not to crush rocks.

9.7 Soil Sieve Procedure Using #10 Sieve

9.7.1 Place sieve on catch pan or clean freezer paper, wax side down. Pour sample into #10 sieve and sift. Gently rub the sample remaining on the sieve to break up clumps. When no more sample passes through sieve, dump all remaining sample on top of sieve onto

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a separate sheet of freezer paper. If large clumps are still present, repeat disaggregation and sieve until no clumps remain.

9.7.2 The sample portion remaining in the #10 sieve is then weighed, documented, and bagged with the sample number and a "Coarse Fragments" label on it.

9.7.3 Weigh, document, and place all the sample passing through the #10 sieve into a labeled Ziploc bag with the sample number and a "Fines" label on it. Add any organic matter that had been removed previously. This Organic matter may need to be cut up into smaller pieces using clean scissors.

9.7.4 Change gloves between samples.

9.8 Soil Sieving Procedure using sieves other than #10

9.8.1 Determine the sieve sizes and process to be used to meet project specifications.

9.8.1.1 Check with the project manager or lab manager for project specifications.

9.8.1.2 If multiple sieve portions are to be obtained, stack the set of sieves in the with the largest size openings on top to the smallest on the bottom, with a catch pan at the base.

9.8.1.3 If sieve sizes smaller than a #10 sieve are being used, the #10 sieve can be used to not plug up the smaller sieve. Anything retained by the #10 sieve must be considered part of the biggest sieve portion.

9.8.2 Pour the dried and disaggregated soil onto top sieve.

9.8.3 Record the sample number on the side of the catch pan. An abbreviated number may be used such as 407-1.

9.8.4 All dried contents are poured onto the sieve including the rocks and foreign matter that had been set to the side. The organic foreign matter may need to be cut up into smaller pieces using clean scissors.

9.8.5 Place the set of sieves on a mechanical shaker. Tighten the mechanical shaker adjustments so that the sieves fit tightly and securely in the mechanical shaker. Set the timer for 10 minutes and begin the sieve shaking.

9.8.6 After 10 minutes remove the sieves from the mechanical shaker.

9.8.7 Weigh, document, and place all the sample contents in the catch pan into a labeled Ziploc bag with the sample number and a "Fines" label on it.

9.8.8 Great care should be taken in matching the sample number written on the catch pans to the sample numbers on the labeled container.

9.8.9 Certain projects may require that the portion of sample above the sieve be retained. If this is required pour the sample remaining on top of the sieve(s) into a second bag, label with the lab number and mark "Coarse Fragments". Zero the balance with the same bags used, then weigh and document the mass of this portion.

9.8.10 Record the sieve date, analyst, shaker ID, and sieve size used on the Soil Sieve Prep Log. Note if coarse fragments were retained.

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9.9 Pulverization - Some projects or methods may require that the sieved sample be further pulverized prior to analysis. The sample may be pulverized with a motor and pestle or other method.

9.10 Cleaning Sieves – the sieves must be washed and dried between each use.

9.10.1 Place the sieves in the sink and scrub with a brush or green scrubbie and running hot water to remove any soil particles embedded in the mesh. Rinse well with tap water then rinse with deionized water. Soap is not used as it is very difficult to rinse from the sieves.

9.10.2 Place the sieves and catch pans in an oven to dry. Alternatively allow to air dry overnight on the counter.

10.0 Data Analysis And Calculations

Not applicable to this SOP.

11.0 Quality Control and Method Performance

11.1 Quality Control - Not applicable to this SOP.

11.2 Instrument QC - Not applicable to this SOP.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits - Not applicable to this SOP.

12.0 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-GBAY-0094 *Training and Employee Orientation* (current revision or replacement) for more information.

13.0 Data Review And Corrective Action

Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

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The review steps and checks that occur as employees complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-GBAY-0120 *Data Review and Final Report Processes* (current revision or replacement) for specific instructions and requirements for each step of the data review process.

13.1 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

There is no QC performed with this analysis.

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TEST METHOD STANDARD OPERATING PROCEDURE
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14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

15.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

16.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

17.0 ATTACHMENTS

Attachment I: Sieve prep log (Example)

Attachment II: Dry Sieve Flow Chart

18.0 REFERENCES

18.1 Pace Quality Assurance Manual - most current version.

18.2 The NELAC Institute (TNI); Volume 1, "Management and Technical Requirement for Laboratories Performing Environmental Analysis" - most current version.

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19.0 REVISION HISTORY

This Version: ENV-SOP-GBAY-0164-Rev.00

Section	Description of Change
All	First Issue of SOP.

This document supersedes the following document(s):

Document Number	Title	Version

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Soil Sieve
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Attachment I: Sieve Prep Log

Work Order		Date/Time In		Humidity In (%)		Temp In (°C)		Reviewed by	
Batch		Date/Time Out		Humidity Out (%)		Temp Out (°C)			

Samples		Sieve Date	Shaker ID	Analyst	Archive Weight (g) Balance ID: 40BALW	Analyst	Weight of >60 Mesh (g) Balance ID: 40BALX	Analyst	Weight of <60 Mesh (g) Balance ID: 40BALX	Analyst
	-001		40SKR3							
	-002		40SKR4							
	-003		40SKR5							
	-004		40SKR3							
	-005		40SKR4							
	-006		40SKR5							
	-007		40SKR4							
	-008		40SKR6							
	-009		40SKR3							
	-010		40SKR4							
	-011		40SKR6							
	-012		40SKR7							
	-013		40SKR8							
	-014		40SKR3							
	-015		40SKR4							
	-016		40SKR5							
	-017		40SKR6							
	-018		40SKR7							
	-019		40SKR8							
	-020		40SKR4							

A similar version including the same information may be used.

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TEST METHOD STANDARD OPERATING PROCEDURE
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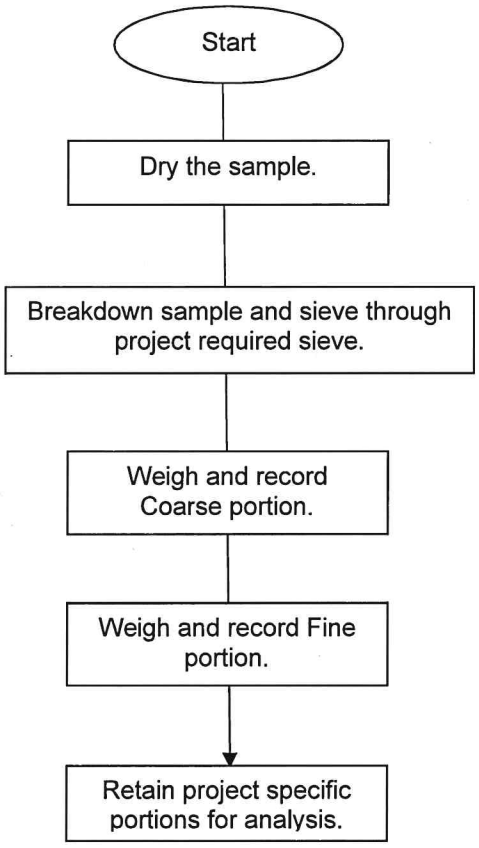
Attachment II: Dry Sieve Flow Chart



	ENV-SOP-MIN4-0055 v04_Percent Solids Moisture by ASTM D2974-07	
Pace ANALYTICAL SERVICES	**Effective Date: 08/23/2022**	COPYRIGHT© 2019, 2021, 2022 Pace®

Management Approval:
Andrew Mickelson Approved on 8/17/2022 12:09:12 PM
Aileen Stacks Approved on 8/23/2022 3:55:53 PM

1.0 Scope and Application

This standard operating procedure describes the gravimetric determination of the percent moisture by measuring the solids content of soils, peats, organic clays, silts, etc.

1.1 Applicable Matrices

This SOP is applicable to most moisture bearing solids including but not limited to soils, peats, organic clays, and silts.

Dry weights are automatically assigned to samples having a solid matrix listed in LIMS. Certain determinative methods do not require moisture corrected results and this test should not be conducted for the following procedures: Toxicity characteristic leachate procedure, 8280 Low Resolution Dioxin, PH, paint filter, and flashpoint.

2.0 Summary of Method

A representative portion of a soil sample is dried in an oven and the solids content is determined by the weight loss. The percent moisture content is calculated from the solids content.

3.0 Interferences

Not applicable to this SOP.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- **Dry Weight** – The weight of a sample based on percent solids after drying in an oven at a 105°C ±5°C.
- **Sample Delivery Group (SDG)** – A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently. A Sample Delivery Group is generally defined by one of the following, whichever occurs first:
    1) All Samples within a project; or
    2) Every set of 20 field samples within a project; or
    3) All samples received within a 14-day calendar period.
    4) Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soil samples in one SDG, all water samples in another), at the discretion of the laboratory. Clients may establish different SDG classifications to meet project specific requirements.

5.0 Health And Safety

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure

The following sections provide general health and safety information about chemicals and materials

that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.
- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001)
- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.
- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 Sample Collection, Preservation, Holding Time & Storage

The laboratory provides containers for the collection of samples upon client request. Refer to laboratory SOP ENV-SOP-MIN4-0009 *Bottle Preparation* (current version or equivalent replacement) for procedures related to preparation of bottle kits for the test method(s) associated with this SOP.

The laboratory does not perform sample collection or field measurements for this test method. Samples should be collected in accordance with a sampling plan and sampling procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

Container Type, Minimum Sample Amount, Preservation, and Holding Time Requirements:

Matrix	Container Size & Type	Required Sample Amount[1]	Preservation	Holding Time
Solid	8 oz glass jar	10 grams	<6°C, but above freezing	There is no specified holding time in ASTM2974. The LIMS is set to 30 days from collection for the sake of an acode requirement, but data will not be qualified for holding time exceedances

[1]*Amount of sample required for each discrete test.*

Thermal preservation is checked and recorded on receipt in accordance with laboratory SOP ENV-SOP-MIN4-0008 *Sample Management* (current version or equivalent replacement).

After receipt, samples are stored at <6°C until sample preparation.

After analysis, samples are retained as stated in the Pace® standard terms and conditions, unless otherwise specified in the analytical services contract. Samples are then disposed of in accordance with Federal, State, and Local regulations.

7.0 Equipment & Supplies

7.1 Equipment

Equipment	Description	Vendor/ Item # / Description
Metal Spatula	Metal spatula, knife, or spoon	N/A
Oven	Gravity Convection Oven	Fisher 650G, or equivalent
Balance	Analytical with a minimum sensitivity of 0.01g	SN H47315, SN 1126423468, or equivalent
Desiccator	With drierite in a metal tray	Fisher Scientific, or equivalent
Pace Workbench	Sample Preparation Logbook and Data Transmission Software	See master list for most current version
LIMS	Data Reporting Software	See master list for most current version

7.2 Supplies

Supply	Description	Vendor/ Item # / Description
Weighing dish	Disposable aluminum foil	Fisher Scientific #08-732-101, or equivalent
Aluminum foil	Novelis Foil, or equivalent	Fisher Scientific 1217, or equivalent

8.0 REAGENTS & STANDARDS

Not applicable to this procedure.

9.0 PROCEDURE

9.1 Equipment Preparation

9.1.1 Balance calibration verification

9.1.1.1 Daily calibration verification of the balance using one high, one medium, and one low Class I Standard weight.

9.1.1.2 Each day verify the balance that will be used for the moisture analysis with 50.0 g, 10.0 g and 1.0 g weights that are traceable to the National Institute of Standards. Record the appropriate information in a calibration logbook.

NOTE: All balances and weights are calibrated by an outside agency on an annual basis.

9.1.2 Temperature Monitoring

9.1.2.1 Calibrate thermometer in oven on an annual basis. Document calibration using Thermometer Calibration Benchsheet F-MN-L-218 (current version or equivalent replacement).

9.1.2.2 Read the temperature of the oven on a daily basis. Document in the Oven Temperature logbook. The acceptable temperature is 105°C ±5°C. Initial and final temperatures will also be recorded for each batch of samples.

9.1.3 Desiccator Verification

9.1.3.1 Note in comments section of logbook if the Drierite is to be replaced. This is

determined by color. If pink, Drierite it no longer anhydrous and must be replaced with anhydrous blue colored Drierite. See laboratory SOP ENV-SOP-MIN4-0146 *Drierite Regeneration* (current version or equivalent replacement), for procedure.

9.2 Sample Preparation

9.2.1 Batch Setup

9.2.1.1 Determine whether a specific container was collected for dry weight (normally a 60mL plastic container). If not, a metals container should be utilized for dry weight. Moisture samples cannot be obtained from WIDRO, GRO or VOC sample container.

NOTE: If only one container is sent for multiple tests that include VOA tests, VOA must take their sample out of the container first to keep the integrity of the sample. These containers will be delivered to the VOA lab with a "VOA FIRST" sticker attached to the cap. When VOA has taken a sub sample from the container, they will affix a black dot sticker over the "VOA FIRST" sticker. This indicates that the sample can now be used by other lab areas.

9.2.1.2 Create the electronic preplog file using template F-MN-I-348-Rev.03 "*ASTM D2974 | Percent Moisture / Percent Total Solids*"

9.2.1.3 Arrange physical samples in the order of which they appear in the preplog batch. Observe the sample position number that the prep log associates with each EPIC PRO sample number. Use a black marker to write the EPIC Pro batch number on the first tray (empty tray).

9.2.1.4 Order the trays in the exact numerical order that is displayed on the prep log template. The tare masses MUST be obtained in this order.

9.2.2 Tare and Wet Weight Determination

9.2.2.1 Click on the Balance icon to the left of the AutoPost button on the tool bar to connect to the balance.

9.2.2.2 Double click under "Dish Weight" in the preplog, in the bottom, middle pane, for the first sample.

9.2.2.3 Place a tin on the balance, wait for the balance to stabilize and press the print button to send the weight to prep log. You should now see the tare mass displayed in the "Dish Weight" field for your sample. Tare all the subsequent trays in this manner.

9.2.2.4 Place the tray on the balance. The same tare mass that is recorded on the prep log template should be displayed on the balance. Confirm the lab ID with the one in the template.

9.2.2.5 Obtain a representative sample by stirring. Make a comment in the 'Sample Notes' field if a sample or its DUP is not homogenous. Do not remove any rocks that are smaller that pea size. Add 5.0-10.0g of sample to the tray.

9.2.2.6 Place tray on the balance and close both balance doors. Double click in the "Wet Weight w/Dish" field for the first sample. Press the print button on the balance to transfer the data, you should now see the wet mass that is displayed on the balance also displayed in the correct cell of the prep log template. Obtain the wet

mass for all of the subsequent samples in this manner.

9.2.3 Sample Drying

9.2.3.1 Place samples in the oven. Dry the sample overnight (minimum of 16 hours). Record the initial time and temperature.

NOTE: The correction factor of the thermometer ID associated with the oven will calculate and display the corrected temperature based on the observed temperature you recorded.

9.2.3.2 Samples should not be dried longer than 24hours. Remove the sample from the oven, record the date and time the samples were removed from the oven.

9.2.3.3 Place samples in a desiccator and record the time. Allow samples to cool in the desiccator for at least 30 minutes.

9.2.4 Final Weight

9.2.4.1 Remove samples from desiccator and record the time.

9.2.4.2 Ensure the order of the trays are in the exact numerical order that is displayed on the prep log template. The dry masses MUST be obtained in this order.

9.2.4.3 Using the "Dry Weight 1" field in the prep-log, begin determining the final weight.

9.2.4.4 Tare the balance, place sample tray on the balance. Close balance doors.

9.2.4.5 Press the print button that is located next to the balance. You should now see the dry mass that is displayed on the balance also displayed in the correct cell of the prep log template.

9.2.4.6 Obtain the dry mass for all the remaining samples in this manner.

NOTE: If a sample was dried for less than 16 hours, it must be documented that constant weight was attained. To do this, record data for a minimum of two weigh/dry/desiccate weigh cycles with a minimum of 1 hour drying time in each cycle. Constant weight is defined as a loss in weight of <0.01 g between the start weight and final weight of the last cycle.

9.2.4.7 The TS Posted (%) and the Percent Moisture data will auto-populate based on the dry weight entered.

9.2.5 Documentation

9.2.5.1 Record the necessary information in the electronic preplog using template version F-MN-I-348-Rev.03 (or equivalent replacement). Information will include batch and sample ID, tin weight, initial and final weight, drying cycle time and temperature, desiccator cycle time, prep date, prep analyst, and supporting equipment. Also include any additional comments if needed.

10.0 Data Analysis & Calculations

10.1 Percent Solid Calculation

This calculation will be performed and reported in EPIC via the Workbench. This value will be used for calculating analytical concentration on a dry weight basis.

$$\%Solids = \left(\frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight}\right) \times 100$$

11.0 Quality Control & Method Performance

11.1 Quality Control

Prepare the following QC samples with each batch of samples.

QC Check	Acronym	Acceptance Criteria	Frequency
Duplicate	Dup	The RPD should be ≤ 30%.	One sample must be prepared and analyzed in duplicate at a frequency of 1 in 10 samples or 1 per analytical batch, whichever is more frequent.

11.2 Instrument QC

Not applicable to this SOP.

11.3 Method Performance

11.3.1 Method Validation

Refer to corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* (current version or equivalent replacement) for general requirements and procedures for method validation.

Establish detection limits (DL) and limits of quantitation (LOQ) at initial method set up and verify the DL and LOQ on an on-going basis thereafter. Refer to corporate policy and/or SOP for DL and LOQ requirements and procedures.

12.0 Data Review & Corrective Action

12.1 Data Review

The data review process of Pace® Analytical Services includes a series of checks performed at different stages of the process by different people to ensure that SOPs were followed, the analytical record is complete, and properly documented, QC criteria were met, proper corrective actions were taken for QC failure and other nonconformance(s), and test results are reported with proper qualification, when necessary.

The review and checks that are performed by the employee performing the task is called primary review.

All data and test results are also peer reviewed.

This process, known as secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented, and approved in accordance with the Pace® Analytical Services SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

Lastly, a third-level review, called a completeness check, is performed by reporting or project

management staff to verify the test report is complete.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (current version or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is required when QC or sample results are not within acceptance criteria.

If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 Pollution Prevention & Waste Management

Pace® proactively seeks ways to minimize waste generated during work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practices comply with all applicable federal and state laws and regulations. Excess reagents, samples, and method process wastes are characterized and disposed of in an acceptable manner in accordance with the Pace® Chemical Hygiene Plan / Safety Manual. Refer to this manual for these procedures.

14.0 Modifications

The procedures in this SOP have been modified from the reference test method as follows:

Modification	Test Method Procedure	Justification for Modification
Pace uses well homogenized aliquot of 5-10 g placed in 42mL disposable aluminum tin.	The method specifies a test specimen of at least 50 g while using a porcelain evaporative dish with a capacity of no less than 100 mL.	Reduced weight implemented for reduced drying time and minimizing waste stream volumes.

When applicable, comparability and/or equivalency studies necessary to validate the modification as required per corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*

(current version or equivalent replacement) are retained by local quality personnel for historical reference.

15.0 RESPONSIBILITIES

- All employees of Pace® Analytical Services that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- Local quality personnel are responsible for tracking the currency of the R&A on this SOP for employees at the locations they are assigned to and for notifying the General Manager (GM), however named, when R&A are overdue or outstanding. The GM and the employee's direct supervisor are responsible for ensuring the employee completes the R&A assignments as required.
- The supervisors and managers of Pace® Analytical Services, however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP the work area(s) they oversee.
- All employees of Pace® Analytical Services are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local Quality Manager and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual change to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015 *Document Management* (current version or equivalent replacement) and ENV-SOP-CORQ-0016 *SOP for Creation of SOP and SWI* (current version or equivalent replacement).
- Local quality personnel are responsible for monitoring conformity to this SOP during routine internal audits of work areas that utilize this SOP and for communicating gaps and deviations found during monitoring to the work area supervisor, who is responsible for correction of the situation.

16.0 ATTACHMENTS

Not applicable to this SOP.

17.0 REFERENCES

- ASTM D 2974-07, Standard Test Methods for Moisture, Ash, and Organic Matter of Peat and Other Organic Soils.
- TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-V1-2009.
- TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.
- ENV-SOP-CORQ-0011, *Method Validation*, current version.
- ENV-SOP-CORQ-0015, *Document Management*, current version.

- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-TMP-CORQ-0007, *Quality Manual Template*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- COR-MAN-001, *Pace® Safety Manual*, current version.

18.0 REVISION HISTORY

Authorship

Primary Author[1]	Job Title	Date Complete
Andrew Mickelson	Department Manager – Inorganics	8/15/22

[1]The primary author is the individual / role responsible for the content of this SOP. Send questions or suggestions for content to the primary author. See the Quality Manager for questions or concerns related to implementation of this SOP.

Revisions Made from Prior Version

Section	Description of Change
All	Converted SOP to latest corporate SOP template; and Updated to Qualtrax EDMS format.

Document Succession: This version replaces the following documents:

Document Number & Version	Document Title	Effective Date:
ENV-SOP-MIN4-0055 v03	Percent Solids Moisture by ASTM D2974-07	05/22/2022

Pace Analytical®
ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0056 **Revision:** 04

Document Title: Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B

Department(s): Metals

Date Information

Effective Date: 06 Oct 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0056 **Revision:** 04

Title: Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0056

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Sep 2021, 12:40:17 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	General Manager 2	01 Oct 2021, 05:17:47 PM	Approved
Andrew Mickelson (009792)	Manager	06 Oct 2021, 02:22:12 PM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Preparation of Solid Samples for Analysis by ICP and ICPMS
TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the preparation of solid samples using hot block digestion as described in EPA Method 3050B.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in the associated analytical SOP; SOP ENV-SOP-MIN4-0052 *Metals Analysis by ICP - Method 6010 and 200.7* or ENV-SOP-MIN4-0043 *Metals Analysis by ICP/MS - Method 6020 and 200.8* (or equivalent replacements).

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to sediments, sludges and soil samples.

2.0 SUMMARY OF METHOD

A one-gram aliquot sample is digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion, samples are brought to a final volume of 50mL. Digestates are then analyzed using Inductively Coupled Plasma (ICP) technologies for the determination of metals in solution.

3.0 INTERFERENCES

Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed in accordance with the quality control requirements given in SW-846 Sec. 8.0 to aid in determining whether Method 3050B is applicable to a given waste.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

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The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

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TEST METHOD STANDARD OPERATING PROCEDURE
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TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

7.1 Equipment

Equipment	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Hot Block ™	54 Place Hot Block	Environmental Express
Analytical Balance	Ability to weigh to the nearest 0.01g	Fisher Scientific or equivalent

7.2 Supplies

Supply	Description	Vendor/Item #/Description
Digestion Cups	50 mL verified to class A specification	Environmental Express or equivalent
Vapor Recovery Device	Reflux cap or Watch glass	Environmental Express or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent

8.0 Reagents And Standards

8.1 Reagents

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II	Verify that background levels of volatile compounds are acceptable by analysis
Hydrogen Peroxide	30% ACS Grade	Fisher brand
Hydrogen Peroxide	30%, Optima Grade for tin only	Fisher brand
Concentrated nitric acid (HNO_3)	Trace Metal grade	Fisher brand
Concentrated hydrochloric acid (HCl)	Trace Metal grade	Fisher brand

8.2 Standards

Standard	Concentration/Description	Requirements/Vendor/Item #
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are METALS-STK1 and METALS-STK2. See Appendix A for composition	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.
Mercury Spike – Stock solution standards for LCS and MS/MSD	10 µg/mL Hg-STK Stock	Purchased from Spex Certiprep. Store at room temperature. Expires as specified by manufacturer.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

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Verify the calibration of variable and fixed volume pipettes as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

Verify the calibration for the thermometer as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

9.1.2 Equipment

The hot block digestors are set to maintain a digestion temperature of 95 +/- 5°C. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature should be checked in different wells of the hot blocks such that all wells are evaluated over a period of time. Record the temperature of each hot block daily in the temperature logbook.

Balances shall be checked prior to use on each working day with a NIST traceable reference in the expected range of use. Balances must be verified with weights of a class appropriate for the accuracy of the balance being calibrated. Verify the calibration for the balance as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Record the measurements of each weight in the daily balance verification logbook.

9.2 Sample Preparation

9.2.1 Obtain and label digestion tubes in the order for which samples will be weighed out.

9.2.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh a 1-1.1g portion of sample (to the nearest 0.01g) and transfer to a 50 mL digestion cup. Alternative sample volume may be used based on sample matrix. Weigh out 3 aliquots for the batch QC sample (background, matrix spike (MS), and matrix spike duplicate (MSD) being sure to weigh them as close to the same weight as possible.

9.2.2.1 Create a method blank and a laboratory control sample (LCS) by weighing out 1 gram of resin beads for each.

9.2.2.2 Spike the LCS, MS/MSD each of METALS-STK1 and METALS-STK2. If mercury is requested spike 0.25 mL of Hg-SPK stock.

9.2.3 Add DI to the 10mL marking for each sample.

9.2.4 Add 7.5mL of concentrated HNO3, mix the slurry, and cover with a reflux cap. Heat the sample to 95 +/- 5°C and reflux for 70 minutes without boiling. Record initial Hot Block temperature in the digestion log. Observe the sample during heating for brown fumes indicating oxidation of the sample. If this occurs, add up to an additional 5 mL HNO3 and re-heat. Repeat this process until no fumes are given off during heating. Record on the digestion log to what samples and how much additional acid was added.

NOTE: When mercury is a requested analyte, watch glasses will be used rather than reflux caps.

9.2.5 Cool the sample 10 minutes. Add 2.5mL of 30% hydrogen peroxide. Cover with reflux cap and return to the Hot Block for warming which will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Heat until effervescence subsides for a total of 10 minutes. Cool the samples in the plastic cups.

NOTE: Use Optima grade hydrogen peroxide if the analysis of tin (Sn) is required. Tin is used as a stabilizer in the ACS grade of hydrogen peroxide.

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9.2.5.1 If effervescence does not subside, continue to add 30% hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note in the comments section of prep sheet the additional aliquots.

NOTE: Do NOT add more than a total of 10mL hydrogen peroxide.

9.2.6 Add 5mL of concentrated HCl, return the sample to the Hot Block and reflux for an additional 15 minutes without boiling.

9.2.7 Remove samples from Hot Block and record final temperature in digestion log. Allow samples to cool. Bring samples up to a final volume of 50 ml with DI water. Cap and invert several times for proper mixing.

9.2.8 Samples may be allowed to sit overnight while solid materials settle out or samples may be centrifuged for 15 minutes at a rate of 1000 rpm. If samples are centrifuged, all QC samples including the method blank and laboratory control sample (LCS) must also be centrifuged.

9.3 Documentation

9.3.1 Digestion Records

Record the necessary information in the electronic preplog using template version F-MN-I-330-Rev.01. Information includes batch and sample ID, initial and final volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. Save file in prep log with the naming convention;

"Queue HBN Method" Ie. MPRP 555222 6020A

10.0 Data Analysis And Calculations

10.1 Calculations

Refer to associated analytical SOP for equations and common calculations.

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to associated analytical SOP for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.

11.2 Method Performance

11.2.1 Method Validation

11.2.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

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Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to the associated analytical SOP for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 The preparation method has been modified in terms of the amounts of reagents used and the individual heating times. The chemistry is maintained. Reason for this modification is better performance for silver and antimony. PT samples are analyzed regularly to validate that the modifications are effective. Per the method, the nitric acid and peroxide amounts are varied based on the sample reaction and this is the case with the Pace method. Overall, the Pace digestion ends up with a higher total acid concentration.

14.2 The final volume for the Pace method is 50 mL, opposed to 100 mL for the reference method.

14.3 Samples are processed using the Hot Block digestion system employing metals free disposable plastic ware rather than glass beakers.

15.0 Responsibilities

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TEST METHOD STANDARD OPERATING PROCEDURE
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Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
8.2	Updated concentration description for the metals spike
9.1.2	Include balance calibration verification
9.2.2.2	Update spike sources and volumes
9.3.1	Provide greater detail for documentation procedure ie batch nomenclature.
Appendix A	Added/updated spike sources
9.1.2	Include balance calibration verification
9.3.1	Provide greater detail for documentation procedure ie batch nomenclature.
9.2.2.2	Update spike sources and volumes

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0056	Metals Preparation of Solid Samples for Analysis by ICP and ICPMS by EPA Method 3050B	03

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Preparation of Solid Samples for Analysis by ICP and ICPMS
TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

Appendix A: Metals Standard Reference

Stock standards used for solid sample preparation

METALS-STK1		METALS-STK2		Hg-SPK	
ZPACEMN-116		ZPACEMN-106		MERC-STK1 Stock	
Element	(mg/L)	Element	(µg/L)	Element	(µg/L)
Ca	2000	Si	500	Hg	10000
Fe	2000	Sb	100		
Mg	2000	Mo	100		
K	2000	Sn	100		
Na	2000	Ti	100		
Al	2000	S	2000		
Ba	100	As	100		
Be	100	Pd	20		
Bi	100	Pt	20		
B	100	Se	100		
Cd	100				
Cs	100				
Cr	100				
Co	100				
Cu	100				
Li	100				
P	100				
Mn	100				
Pb	100				
Ni	100				
Ag	50				
Sr	100				
Tl	100				
V	100				
Zn	100				
U	100				
Th	100				

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0052 **Revision:** 05

Document Title: Metals Analysis by ICP - Method 6010 and 200.7

Department(s): Metals

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Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0052 **Revision:** 05

Title: Metals Analysis by ICP - Method 6010 and 200.7

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0052 - ICP

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Jul 2020, 05:05:44 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Andrew Mickelson (009792)	Manager	20 Jul 2020, 02:32:20 PM	Approved
Krista Carlson (004514)	Project Coordinator 1	20 Jul 2020, 04:50:45 PM	Approved
Adam Haugerud (005828)	General Manager 2	31 Jul 2020, 11:01:55 AM	Approved

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved and total recoverable metals by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is verified daily by running a QC solution (CRDL) at the LOQ and evaluating against method specific limits.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to drinking water, ground water, aqueous samples, liquid samples, leachates, industrial wastes, soils, sludges, sediments, and other solid wastes.

2.0 SUMMARY OF METHOD

Prior to analysis, samples are solubilized or digested using appropriate sample preparation methods. This method describes the determination of elements by ICP-OES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a charge coupled device detector (CCD). All data is collected by simultaneous measurement. Software is used to measure and apply corrections due to background or inter-element interferences using a variety of techniques. Alternate wavelengths are also monitored for confirmation or to use in correction equations.

3.0 INTERFERENCES

3.1 Spectral Interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.2 Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate

wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

3.3 Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. A high solids nebulizer is used on all instruments. Internal standards are also used to monitor and correct for physical effects.

3.4 Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions, use of an ionization buffer, or by matrix matching of standards and samples.

3.5 Memory interferences result when analytes in a previous sample contribute to the signals measured in the new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from buildup of sample material in the plasma torch and spray chamber. Regular maintenance and awareness of samples with high concentrations minimize these interferences.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours if acidification is performed in the lab.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either stored at ambient or 6°C until sample preparation. Prepared sample digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPOES (Inductively Coupled Plasma Optical Emiison Spectrometer)	Agilent 720 or 5110 ICP instrumentation equipped with an CCD Detector, full wavelength region. Each instrument has an associated auto-sampler and recirculating chiller.
Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A or B volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Filters	Filtermate filters, 2 um PTFE, Environmental Express, SC0408
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type I – 18 megaohm
Nitric Acid (HNO_3), trace metals grade	Fisher Scientific, A-509-P212 or equivalent
Hydrochloric acid (HCl),trace metals grade	Fisher Scientific, A-508-P212 or equivalent
4% (v/v) Nitric Acid/5% (v/v) Hydrochloric Acid Solution	400 mL nitric acid (above) + 500 mL hydrochloric acid (above) to 10 liters with ASTM Type I water (18 megaohm). Used for all blanks and rinsing and preparation of standards.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards. Spex Certiprep or equivalent. See Appendix D for the standard information
Cesium Ionization Buffer for use with Agilent 720	50,000 PPM, High Purity Standards P/N 1B-CS-B5 or equivalent.
Wavelength Cal Solution - Agilent	Various analytes, Agilent P/N 6610030100
Internal Standards	Yttrium, Inorganic Ventures or equivalent

9.0 PROCEDURE

9.1 Equipment Preparation

Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following;

- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream
- Ar/O pressure - The argon supply pressure should be set at about 80-100psi. If the supply argon pressure falls below about 80psi, a safety interlock automatically shuts off the torch.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.

- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minute for break-in period
    - Adjust the pump-tubing in such a way to ensure proper flow prior to igniting the plasma. Decrease flow to where flow of bubble actually stops or barely moves. Turn knob 2 full turns.
- Ignite plasma while tubing is in a rinse solution, allow plasma to warm up at least 30 minutes and preferably 60-90 minutes.
- Use the warm up time to create the sequence and pour samples. Use Horizon Uploader to copy labels into the sequence.

9.1.1 Support Equipment

Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions

Instrument operating conditions vary by method and by instrument. All conditions are documented with each worksheet and cannot be modified after data has been generated. Instrument conditions are stored within a worksheet template. The analyst selects the appropriate Template for analysis. The analyst does not change operating conditions. Conditions are only changed during method development.

9.2 Initial Calibration

9.2.1 Calibration Design

A calibration curve consists of a single point standard and a calibration blank.

9.2.2 Calibration Sequence

Example Analytical Sequence

CAL0
CAL1
ICV
ICB
CRDLA
ICSA
ICAB
Fe 2000 SIC
Ca 2000 SIC/LDR
Al 1000 SIC/LDR
Mg 1000 SIC/LDR
Mn 100 SIC
CCV

CCB
Ba 20 SIC/LDR
Cr 50 SIC/LDR
Co 20 SIC/LDR
As 10 SIC
V 20 SIC/LDR
Cu 20 SIC/LDR
Ni 50 SIC/LDR
Ti 30 SIC/LDR
Mo 10 SIC/LDR
Zr 20 SIC
CCV
CCB
P 50 SIC
Ce 10 SIC
LDR A
LDR B
LDR C
CCV
CCB
CLIENT SAMPLES
CCV
CCB
CRDLA

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a single point calibration model, a linear regression curve is established using a calibration blank and one non-zero standard.

9.2.3.2 Relative Standard Error (RSE)

With a single point calibration model using a calibration blank and one non-zero standard, relative standard error evaluation is not applicable.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV, followed by an ICB, is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

9.3 Sample Preparation

9.3.1 Label all sample tubes so that each sample can be uniquely identified on the rack.

9.3.2 If any samples in a batch need to be filtered because of suspended material, use an Environmental Express Filtermate. The Method Blank and LCS must also be filtered if any samples are. Record the ID of the Filtermates used.

9.3.3 Centrifuge soil samples to minimize need for filtering.

9.3.4 Aqueous samples are poured without initial dilution unless historical data demonstrates otherwise.

9.3.5 Use Horizon Uploader to copy labels into the sequence.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Quantitative Identification

10.1.1 Monitor all initial QC checks. One re-analysis of QC checks is allowed. If initial QC fails twice, make instrument modifications and recalibrate using a new worksheet from template.

10.1.2 During the sample analysis or after the analysis is completed, transfer valid data into LIMS system using LIMS LINK.

10.1.2.1 Export data from instrument to CSV file.

10.1.2.2 Open LIMSLINK

10.1.2.3 Click open instrument, select CSV file from list, data will import

10.1.2.4 Highlight QC + samples, select “Get LIMS Info”

10.1.2.5 Run QC will prompt for Q-Batch # plus standard selection

10.1.2.6 Sample data will prompt for SD/PDS source sample.

10.1.2.7 Right click on samples to select/de-select elements

10.1.2.8 Highlight samples to upload and select “Export Run to Epic Pro”.

Note: Be sure to make the appropriate selections in LIMSLNK rather than post-editing in EPIC. This provides for a much smoother experience and minimizes chance for error. If edits must be done in EPIC be sure to make edits prior to uploading new data from LIMSLINK, as this, again minimizes error due to confusion.

10.1.3 When Complete, select “excel bench sheet”. Save the Excel Bench sheet to the instrument folder marked “LIMSLINK RAW DATA” Use convention of run date (e.g. 032917ICP5). Note discrepancies in the notes section of the run log (including dilutions, QC issues, re-runs, etc.).

10.1.4 In LIMS system make final adjustments and add any required footnotes. Complete checklist and turn data in for validation.

10.1.5 Documentation is a mix of electronic and paper files. Key data must be stored electronically so that data review may be performed from any location. Some documents are stored in the physical daily folder and archived for easy reference.

10.1.6 Label a physical file with the date. Record the file name, Q-Batch, and all prep batches on the folder for each run that day (example: 032917ICP5 and 032917ICP5B.

10.1.7 Store printed copies of batch worklist reports, prep bench sheets, the original checklist, a printed copy of the IEC Form 10-IN generated from Gandolf, and a printed copy of the run log from LIMSLINK file in this folder. If the data reviewer requests additional printed information they may print it themselves. Note, if data is validated remotely print a copy of the validation verification e-mail and include with each checklist.

10.1.8 Generate a copy of the raw data and print to the X:Drive.

10.2 Calculations

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations*, or equivalent replacement, for equations for common calculations.

10.2.1 Inter-element Correction Factor (IEC) = Concentration of apparent concentration (observed) in mg/L / Concentration of Interferent in mg/L.

10.2.2 The percent recovery of the spike is calculated from the following equation:

$$\%\ \text{Recovery} = \frac{(SSR-SR)\ X\ 100}{ST}$$

Where:
SSR = Spiked Sample Result, ug/L or mg/kg dry
SR = Sample Result, ug/L or mg/kg dry
ST = Spike Target, ug/L or mg/kg dry

10.2.3 The relative percent difference between the MS/MSD can be calculated as follows

$$RPD = \frac{|(S-D)|\ X\ (100)}{(S+D)/2}$$

Where:
RPD = Relative Percent Difference
S = Original Spiked Sample Value, ug/L or mg/kg dry
D = Second Spiked Sample Value, ug/L or mg/kg dry

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.

Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6010B/C/D. 1 per batch of 10 or fewer samples for 200.7
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples for 6010B/C/D.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6010B/C/D.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification (ICV)	Immediately after each initial calibration.
Spectral Interference Check Solutions (SIC)	Immediately after each ICV/ICB.
Initial Calibration Blank	Immediately after each ICV.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6010B/C/D/200.7 and at a minimum of once at the end of each run for 6010C.
ICSA verification	At the beginning of each sample run sequence after the CRDL.
ICSAB verification	This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP.
Internal Standard	An appropriate internal standard is required.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-NW-0018 *Determination of LOD and LOQ* for these procedures.

11.3.2 Linear Dynamic Range (LDR)

Method 6010D requires that a LDR check sample be analyzed daily. Because of this requirement for 6010D, the LDR is established daily for all methods. For some elements a single element standard is used to establish the LDR while in other cases a mixed standard is used to establish the LDR. If an LDR standard is not analyzed for a particular analyte then the LDR defaults to the highest calibration point in the calibration curve.

Data is reported up to 90% of the LDR. When evaluating interferences use values up to the full LDR for the interferent. The LDR may be established at higher or lower levels on a daily basis based on expected levels of samples being tested that day. The LDR may vary daily depending on slight changes in instrument performance (things like pump tubing wear, etc.). Refer to Attachment VII for default linear ranges and the typical standards used to establish them

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-NW-0025 *Training and Orientation Procedures* for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action

when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

Appendix E – Check Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-V1-2009.

TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Water and Solid Waste, SW-846 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

Test Methods for Evaluating Water and Solid Waste, SW-846, Update IV, Feb. 2007. Method 6010C.

Test Methods for Evaluating Water and Solid Waste, SW-846, Update V, July 2018. Method 6010D.

Method 200.7 Revision 4.4, *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry,* 1994.

US EPA Contract Laboratory Program Statement of Work ILM05.3, March 2004.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
17.0	Added years to 6010B & 200.7 references, updated formatting.
Appendix B	Updated MB Acceptance Criteria and Corrective Action for all methods. Updated Post Digestion Spike Acceptance Criteria for 6010B and 6010D.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0052	Metals Analysis by ICP – Method 6010 and 200.7	04

Appendix A: Target Analyte List and Routine LOQ

Table 1: Routine Analyte List and Limits of Quantitation (LOQ)[1]

Element	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	200	10
Antimony	20	1.0
Arsenic	20	1.0
Barium	10	0.50
Beryllium	5.0	0.25
Boron	150	7.5
Cadmium	3.0	0.15
Calcium	500	25
Chromium	10	0.50
Cobalt	10	0.50
Copper	10	0.50
Iron	50	2.5
Lead	10	0.5
Lithium	10	1.0
Magnesium	500	25
Manganese	5.0	0.25
Molybdenum	15	0.75
Nickel	20	1.0
Phosphorus	20	5
Potassium	2500	125
Selenium	20	1.0
Silicon	50	5
Silver	10	0.50
Sodium	1000	50
Strontium	5.0	0.5
Sulfur	500	25
Thallium	20	1.0
Tin	75	3.75
Titanium	25	1.25
Uranium	50	2.5
Vanadium	15	0.75
Zinc	20	1.0
Hardness	3300	N/A

[1] Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.

Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	A calibration curve must consist of a blank and at least one calibration standard.	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	± 10% for method 6010B, 6010C and 6010D or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010B, 6010C and 6010D and below 3% for 200.7.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criteria of +/- ½ of the RL for method 6010D. All elements of interest must be evaluated to +/- the RL for method 6010B,6010C and 200.7. Criteria to be evaluated to method criteria unless otherwise specified by client.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDLA / LLCCV	The CRDLA must be analyzed at the beginning of each run for every analyte of interest. The CRDLA is analyzed at or below the RL. Additionally, the CRDLA must be analyzed after samples to bracket method 6010C samples.	± 40% (or specified by the client) For method 6010C, must be within ± 30% . For method 6010D, must be within.± 20%.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the CRDL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	For method 6010B, 6010C, 6010D and 200.7, the CCV must be within ± 10% of the true value. The RSD of the CCV must be below 5% for 6010B.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every	All elements of interest must be evaluated to a criteria of +/- the	Identify source of problem, re-analyze. Analysis may proceed if	Qualify analytes with

	10, and at end of analytical window	RL for 200.7, 6010B, 6010C and 6010D. Depending on the data quality objective of individual clients different criteria may apply.	it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	70-125% of its true concentration	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solution (ICSA)	A mixed solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM is analyzed at the beginning of each sample run sequence. In some specific client requirements the ICSA must bracket the run or the analytical batch.	Acceptance criteria for the spiked analytes are 80-120%. Unspiked analytes must have an absolute value less than the RL.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSA can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Interference check solution (ICSAB)	A solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM with low to mid-range concentrations of target analytes as outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	The acceptance criteria are 80-120% for all spiked analytes.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSAB can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Spectral Interference Check Solutions (SIC)	SIC solutions are single-element solutions used to evaluate and correct IEC factors. Specific elements evaluated are listed in specific instrument methods.	Unspiked analytes must have an absolute value less than the RL.	If SIC fails, re-calculate IEC and re-process data. If a sample level exceeds an SIC level and the interfering element affects target analytes, then: a) run a higher SIC or b) dilute the sample.	None. Do not proceed with analysis for elements that cannot be verified.
Method Blank	One per 20 samples	Method 200.7: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed.	Qualify outages and explain in case narrative.

		Method 6010B, 6010C and 6010D: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	
LCS	One per 20 samples	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 6010B,6010C and 6010D 85-115% for 200.7 %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: 1:5 dilution of sample, SD RPD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL. 6010D: 1:5 Dilution of sample or MS, for concentrations 25x > LLOQ in parent sample, resultant RPD should agree within +/- 20%.	Data is qualified.	Qualify outages.
Post Digestion Spike	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	80-120% for 6010C 75-125% for 6010B and 6010D.	Data is qualified.	Qualify outages.

Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of +/- ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of +/- the RL for method 60106010B,6010C and 200.7. If the FB does not contain target analytes at a level that interferes with project-specific DQOs, then the FB would be considered acceptable.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range	If a SIC/LDR standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range. See Appendix C.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the data reporting range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved.	N/A

Note: In the absence of method specified recovery limits, results will be evaluated based on specifications outlined by the MPCA guidelines for Inorganic Analysis.

Appendix C: Linear Range Reference Table

Wavelength	LDR (PPM)	Standard	Type
Ag 328	2	CAL1	LDR
Al 237	1000	Al 1000 SIC/LDR	SIC/LDR
As 188	10	As 10 SIC	SIC
As 188	20	LDR B	LDR
B 249	20	LDR A	LDR
Ba 455***/Ba 585**	20	Ba 20 SIC/LDR	SIC/LDR
Ba 585*	50 0	Ba 50 SIC	SIC/
Be 234	4	CAL1	LDR
Ca 370	2000	Ca 2000 SIC/LDR	SIC/LDR
Cd 214	20	LDR B	LDR
Co 228	50	Co 50 SIC/LDR	SIC/LDR
Cr 267	20	Cr 20 SIC/LDR	SIC/LDR
Cr 267	50	Cr SIC/LDR	50
Cu 327	20	Cu 20 SIC/LDR	SIC/LDR
Cu 327	50	Cu 50 SIC/LDR	SIC
Fe 261	200	LDR C	LDR
Fe 273*	2000	Fe 2000 SIC	SIC
K 766***	200	LDR C	LDR
K 766**	20	CAL1	LDR
Li 670	4	CAL1	LDR
Mg 383	1000	Mg 1000 SIC/LDR	SIC/LDR
Mn 257	20	LDR B	LDR
Mn 293*	100	Mn 100 SIC	SIC
Mo 204	10	Mo 10 SIC/LDR	SIC/LDR
Na 589***	200	LDR C	LDR
Na 589**	20	CAL1	LDR
Ni 231	50	Ni 50 SICLDR	SIC/LDR
P 213	20	LDR B	LDR
Pb 220	100	LDR A	LDR
S 181	200	LDR C	LDR
Sb 206	20	LDR A	LDR
Se 196	20	LDR B	LDR
Si 251	20	CAL1	LDR
Sn 189	20	LDR A	LDR
Sr 421	4	CAL1	LDR
Ti 334	20	LDRA	LDR
Ti 334	30	Ti 30 SIC	SIC
Tl 190	20	LDR B	LDR
U	4	CAL1	LDR
V 292	20	V 20 SIC/LDR	SIC/LDR
Zn 206	50	LDR A	LDR

*Used for Interference Correction Only
** ICP4 Only
*** ICP5 Only

Appendix D: Standard Reference Tables

ICP Working Calibration Standard					ICP Calibration Verification Standard			
Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	100	1.0	50	2	50	1.0	50	1
Al	2,000	0.5	50	20	1000	0.5	50	10
As	200	1.0	50	4	100	1.0	50	2
Ba	200	1.0	50	4	100	1.0	50	2
Be	200	1.0	50	4	100	1.0	50	2
Ca	2000	0.5	50	20	1000	0.5	50	10
Cd	200	1.0	50	4	100	1.0	50	2
Co	200	1.0	50	4	100	1.0	50	2
Cr	200	1.0	50	4	100	1.0	50	2
Cu	200	1.0	50	4	100	1.0	50	2
Fe	2000	0.5	50	20	1000	0.5	50	10
K	2000	0.5	50	20	1000	0.5	50	10
Mg	2000	0.5	50	20	1000	0.5	50	10
Mn	200	1.0	50	4	100	1.0	50	2
Na	2000	0.5	50	20	1000	0.5	50	10
Ni	200	1.0	50	4	100	1.0	50	2
Pb	200	1.0	50	4	100	1.0	50	2
S	10000	0.1	50	20	10000	0.05	50	10
Sb	200	1.0	50	4	100	1.0	50	2
Se	200	1.0	50	4	100	1.0	50	2
Tl	200	1.0	50	4	100	1.0	50	2
V	200	1.0	50	4	100	1.0	50	2
Zn	200	1.0	50	4	100	1.0	50	2
Mo	200	1.0	50	4	100	1.0	50	2
B	200	1.0	50	4	100	1.0	50	2
Sn	200	1.0	50	4	100	1.0	50	2
Ti	200	1.0	50	4	100	1.0	50	2
Si	1000	1	50	20	500	1	50	10
Li	200	1	50	4	100	1	50	2
P	200	1	50	4	100	1	50	2
Sr	200	1	50	4	100	1	50	2
U	1000	0.2	50	4	1000	0.1	50	2

Appendix E: Interference Check Standard Reference Tables

ICSA				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	10	100	500000
Ca	5000	10	100	500000
Fe	2000	10	100	200000
Mg	5000	10	100	500000

ICSAB				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Ag	20	1.0	100	200
Al	5000	5.0	100	500000
As	10	1.0	100	100
Ba	50	1.0	100	500
Be	50	1.0	100	500
Ca	5000	5.0	100	500000
Cd	100	1.0	100	1000
Co	50	1.0	100	500
Cr	50	1.0	100	500
Cu	50	1.0	100	500
Fe	2000	5.0	100	200000
Mg	5000	5.0	100	500000
Mn	50	1.0	100	500
Ni	100	1.0	100	1000
Pb	5	1.0	100	50
Sb	60	1.0	100	600
Se	5	1.0	100	50
Tl	10	1.0	100	100
V	50	1.0	100	500
Zn	100	1.0	100	1000

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ENVIRONMENTAL SCIENCES

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QM Approval

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Management Approval

Name/Signature	Title	Date	Meaning/Reason
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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved and total recoverable metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

2.0 SUMMARY OF METHOD

Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. For the total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, mixed, and allowed to equilibrate for the required time prior to analysis.

Sample solutions are introduced by pneumatic nebulization into a plasma, in which desolvation, atomization and ionization occurs. Ions are extracted from the plasma through a differentially pumped vacuum interface and sorted on the basis of their mass-to-charge ratio. The ions transmitted through the quadrupole are detected by an electron multiplier. Ion intensities at each mass are recorded and compared to those obtained from external calibration standards to generate concentration values for the samples. Results are corrected for instrument drift and matrix effects using internal standards.

3.0 INTERFERENCES

Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instruments spectrometer. One way to solve this problem is to measure a different isotope for which there is no

interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. An example includes ClO+ (mass 51), which interferes with V, and must be corrected by measuring ClO+ at mass 53. When possible an interference free isotope should be chosen for measurement.

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of

solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours following acidification. Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are either stored at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Agilent 7700, 7800 7900 ICPMS instrumentation equipped with interference reduction technology. Each instrument has an associated auto-sampler, rough pump and recirculating chiller.

Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Collision Gas	Praxair or equivalent, Ultra high purity He, Ultra high purity H_2,
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent replacement
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent replacement
2% (v/v) Nitric Acid/1% (v/v) Hydrochloric Acid Solution	Used for instrument blanks, standards and dilutions. Prepared in 1 L increments utilizing a volumetric flask and transferring into a C&G narrow mouth storage bottle. This is measured by mixing 20 mL of HNO_3 trace metals grade acid and 10 mL of HCl trace metals grade acid and DI H2O, and bringing to volume of 1 L.
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Combine76 mL of HNO_3 trace metals grade acid and 38 mL of HCl trace metals grade and DI H2O, and bringing to volume of 1 G.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Agilent Tune Solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
EPA Tune solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
Internal Standard Stock Solution	Various suppliers; single element standards to be mixed prior to use with concentrations of 1,000 and 10,000 ug/mL
Working Standards	See Appendix C

9.0 PROCEDURE

9.1 Equipment Preparation

Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following:

9.1.1 Support Equipment

- Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide.
- Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream.
- Ar/O pressure - The argon supply pressure should be set at about 80psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- Helium / Hydrogen pressure - The helium and hydrogen supply pressure should be set at about 15 and 9 psi respectively.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.
- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minutes for break-in period.
- Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure: Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing sufficient concentrations of major cations. The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly degraded tip.

9.1.2 Instrument

Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:

- Torch Ignition - Click on the Plasma icon to open the Instrument window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide.)
- Warm-up- Instrument is allowed to warm-up 30 minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
- Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

- Start-up Configuration - Once the analysis tubing is placed in the Agilent tune solution and stable signal is achieved, the start-up configuration can be initiated. See section 9.1.2.1 for Agilent tune performance monitoring and criteria.
- Create New Experiment File – Open template from the drive. Apply the proper run name for the day (MMDDYYICPMS#). Introduce EPA tune solution and allow signal to stabilize. Initiate performance verification for each mode of analysis. Save each performance report to the network drive. See section 9.1.2.1 for EPA tune acceptance criteria.

9.1.2.1 Routine Instrument Operating Conditions

The instrument is configured to go through the manufacturer recommended startup tune procedure which includes; Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and standard mode performance verification. The measured ratios of oxides 156/140 and doubly charged 70/140 should be <3%. The measured masses of ^{7}Li, ^{89}Y, ^{205}Tl are monitored for initial resolution/axis tuning. EPA Performance verification is later performed for each cell condition used for sample analysis.

EPA Tune Verification - The EPA tuning standard must be analyzed in each mode of analysis to verify resolution and mass calibration are within the required specifications. The tuning standard is analyzed in each mode of analysis at least five times and the relative standard deviation (RSD) must be <5% for all analytes contained in the tuning standard. Conduct mass calibration and resolution checks in the mass regions of interest. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be <0.9 amu full width at 5% peak height.

Pace Minneapolis maintains approval for the analysis of up to 35 elements by the EPA Methods 200.8, 6020, 6020A, 6020B for water and soil matrices. All target analytes are analyzed either in a Helium mode (Collision Cell), hydrogen (Collision Cell), or No gas mode on the Agilent instruments depending on the sample matrix type. The use of interference reduction technologies (Collision Cell) is not allowed for drinking water analysis. Separate calibrations are performed for samples reporting by regulation of the SDWA.

9.2 Initial Calibration

9.2.1 Calibration Design

The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of at least three integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The working range varies with each analyte, see appendix C for summary. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Additional calibration specifications may be referenced in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing*, or equivalent replacement.

9.2.2 Calibration Sequence

Calibration Blank (CAL0)

CAL1
CAL2
CAL3
CAL4
CAL5
CAL6 (optional)
CAL7 (optional)
ICV
ICB
CRDL
ICSA
ICSAB
CCV
CCB
Client samples
CCV
CCB
CRDL (Optional)

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.998.

9.2.3.2 Relative Standard Error (RSE)

%RE is measured at the lowest calibration level and at a point near the mid-level of the calibration (the continuing calibration verification level is recommended). In order for a standard curve to be acceptable, the correlation coefficient/coefficient of determination criterion specified in the method must be met **and** both the low-level and mid-level %RE measures must meet the acceptance criteria. The low-level %RE acceptance criteria is 60%-140% and the mid-level is 90-110%.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

9.3 Digestate Preparation

9.3.1 Homogenization and Subsampling

All solid matrices are subject to centrifuge at a rate of 1000 rpm for 15 minutes or allowed to settle overnight prior to analysis. Once samples have been centrifuged or allowed to settle, an initial dilution of 20 fold is performed on each sample. This is completed by taking 4.75mL of 2% HNO3 / 1% HCL diluent and mixing with a 0.25mL aliquot of sample by means of vortex.

Aqueous samples are inverted multiple times and poured without initial dilution unless historical data demonstrates otherwise.

9.4 Analysis

The instrument performs sample analysis by executing 100 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.

The instrument(s) have been setup and configured in conjunction with manufacturer specifications. Masses were carefully selected to avoid and/or minimize interferences. Internal standard selection was based on performance for the appropriate mass range. Internal standard association must remain within 50 amu of targeted analyte.

The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volumes of well mixed sample aliquots must be prepared until the analysis solution contains < 0.1 mg/L silver.

10.0 Data Analysis And Calculations

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations*, or equivalent replacement, for equations for common calculations.

10.1 Hardness as CaCO3 in mg/L = 2.497 * [Ca in mg/L] + 4.118 * [Mg in mg/L]

10.2 Concentration of lead = summation of signals at 206, 207, and 208 m/z.

10.3 Silica (SiO2) (µg/L) = Silicon (Si) (µg/L) * DF * 60.09 amu (SiO2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

10.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L

v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \text{x}100$$

10.5 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 1:

Equation 1

$$\%RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where, S = Sample result, mg/L or mg/kg

D = Duplicate sample result, mg/L or mg/kg

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6020 (A)(B). 1 per batch of 10 or fewer samples for 200.8
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6020(A)(B).
Internal Standard	An appropriate internal standard is required for each analyte and sample determined by ICP-MS.

Internal Standard	Associated element
Scandium 45	Li, Be, B, Na, Mg, Al, Si, K, Ca, Ti, V, Cr, Mn, Fe, Se
Germanium 72	Co, Ni, Cu, Zn, As, Sr
Indium 115	Mo, Pd, Ag, Cd, Sn, Sb
Terbium 159	Ba, Pt, Hg, Tl, Pb, Bi
Iridium 193	U Th

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Tune	Daily prior to any calibration
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.
ICSA verification	At the beginning of each sample run sequence after the CRDL. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.
ICSAB verification	At the beginning of each sample run sequence after the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be near the midpoint of the calibration range. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or

extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Tuning criteria observed is more stringent than required by the SW846 methods so that the same criteria can be used for both methods 6020 and 200.8.

14.2 The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, thorium, and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.3 The following elements are not listed in the method 200.8 recommended analyte list: bismuth, boron, calcium, iron, lithium, magnesium, palladium, platinum, potassium, silica, silicon, sodium, strontium, tin, and titanium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.4 The following elements are not listed in the method 6020B recommended analyte list: bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 02/2007.

U.S. Environmental Protection Agency. SW846 Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, 7/2014.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

40 CFR Appendix B to Part 136, Definition and Procedure for the Determination of the Method Detection Limit - Rev 2, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
6.0	Updated sample retention from 45 to 21 days.
8.2	Internal Standard Stock Solution – added “1,000 and”
9.2.1	Updated 3 to 5 non-zero standards. Added “The working range…C for summary.”
9.2.2	Added “(optional)” to CAL6. Added “CAL7 (optional)”.
10.0	Added sections 10.4 and 10.5.
11.1	Updated Thoridium 232 to Iridium 193.
14.0	14.2 & 14.4: removed “-238” from uranium. 14.2: added thorium.
17.0	Removed references for Fisions and Region 9 Laboratory SOP.
Appendix A	Added Thorium. Updated Silica and Silicon entries. Removed Mercury NPW and potable water entries.
Appendix B	Updated ICAL Acceptance Criteria. Updated methods referenced in MB Acceptance Criteria. Added LDR acronym to QC Item.
Appendix C & D	Re-formatted tables.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0043	Metals Analysis by ICP/MS – Method 6020 and 200.8	03

Appendix A: Target Analyte List and Routine LOQ[1]

Analyte	Non-Potable Water (ug/L)	Potable Water (ug/L)	Soil (mg/kg)
Aluminum	20.00	20.0	20.00
Antimony	0.50	0.50	0.50
Arsenic	0.50	0.50	0.50
Barium	0.30	0.30	0.30
Beryllium	0.20	0.20	0.20
Bismuth	0.50	-	0.50
Boron	10.00	-	10.00
Cadmium	0.08	0.08	0.08
Calcium	40.00	-	40.00
Chromium	0.50	0.50	0.50
Cobalt	0.50	-	0.50
Copper	1.00	1.00	1.00
Iron	50.00	-	50.00
Lead	0.10	0.10	0.20
Lithium	0.50	-	0.50
Magnesium	10.00	-	10.00
Manganese	0.50	0.50	0.50
Mercury	-	-	0.20
Molybdenum	0.50	-	0.50
Nickel	0.50	0.50	0.50
Palladium	0.50	-	-
Platinum	0.50	-	-
Potassium	100.00	-	100.00
Selenium	0.50	0.50	0.50
Silica	214.00	-	214.0
Silicon	100.00	-	100.00
Silver	0.50	0.50	0.50
Sodium	50.00	-	50.00
Strontium	0.50	-	0.50
Thallium	0.10	0.10	0.10
Thorium	0.50	-	0.50
Tin	0.50	-	2.000
Titanium	1.00	-	1.00
Vanadium	1.00	1.00	1.00
Uranium-238	0.50	0.50	0.50
Zinc	5.00	5.00	5.00

[1] *Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.*

Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Tune	Daily prior to any calibration	Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. This must be completed using 5 replicates with a resulting RSD of <5%.	Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis.
ICAL	Daily	r ≥ 0.998 a Midlevel (recommended near ICV/CCV concentrations) %RE 90-110% Low-Level (Cal1) %RE 60-140%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	All analytes must be within ± 10% of the true value. (%R)	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A)(B) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.	For 6020/200.8: The acceptance criteria are ± 40% (or specified by the client). For 6020A: The acceptance criteria are ± 30% (or specified by the client). 6020B: The acceptance criteria is ± 20% (or specified by the client).	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve).	Qualify outages and explain in case narrative.

			If the CRDL is biased low, no data can be reported for the target elements failing criteria.	
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R): %RSD between multiple integrations must be ≤ 5%	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020 (B). WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	For method 6020, the intensity of internal standard in the ICB/CCB and ICS (ICSA/AB) standards must not deviate more than 80-120% from its original intensity in the associated calibration blank. The intensity of internal standard in the samples and remaining QC must not deviate more than 30-120%. For method 6020A/B, the intensity of the internal standard must not fall below 70% and not exceed 130% from its original intensity in the associated calibration blank. For Method 200.8 the intensity of internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solutions	ICSA containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti is analyzed at the beginning of each sample run sequence after the CRDL. ICSAB containing high concentrations of	ICSA all spiked elements are to be within 20% of the expected true value. The non-spiked elements are to be below the RL. ICSAB all spiked elements are to be within 20% of the expected true value.	Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis for elements that cannot be verified.

	C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.			
Method Blank (MB)	One per 20 samples	Method 200.8: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs. Method 6020, 6020A and 6020B: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	6020/6020A/6020B: 80-120% 200.8: 85-115%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	6020/6020A/6020B: 80-120% 200.8: 85-115% %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	6020/6020A/6020B: 75-125% 200.8: 70-130%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution[1]	One per batch of 20 samples or less		If criteria is not met, original sample and dilution shall be	Qualify outages.

		6020/6020A fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL. 6020B 1:5 dilution of sample 25x > LLOQ or 1:5 dilution of MS since reasonable concentrations are present, results to agree to ± 20%.	reanalyzed. If reanalysis fails, it is determined to be matrix interference.	
Post Digestion Spike[2]	One per batch if there is a MS failure.	6020/ 6020A 80-120% 6020B applicable to elements failing MS, results to agree to +/- 25%. Recommended if high concentration sample not available for dilution test.	If the element fails to meet the recovery criteria, reanalyze. If reanalysis fails, it is determined to be matrix interference.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Target analytes must be less than reporting limit. NC samples are required to be < ½ RL for target analytes. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range (LDR)	For method 6020B: Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the linear range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved as well as maintaining the minimum number of calibration standard requirements.	N/A

[1]To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of diluent. Note: this is a typical process for 200.8 and 6020W. It can be replicated for the preparation of highly concentrated samples by starting with a diluted "parent" sample and then performing the stepwise dilution process.
[2]To Prepare a Post Digestion Spike: An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. The spike addition should produce a minimum level of 10 times the lower limit of quantitation; routine spike volume is 0.020 mL of 20/250 mg/L and 1mg/L mercury stock concentration(s).

Appendix C: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Diluent Volume (mL)	Final Total Volume[1] (mL)	Final Concentration (ug/L)
Internal Standard	6020-Ge	1	See table 8.1	495	500	2000
	6020-Sc	1				
	6020-Tb	1				
	6020-In	1				
	6020-Ir	1				
Bi/Th primary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Bi/Th secondary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Hg 10ppb (intermediate)	HG-LL Stock	0.05		49.95	50	10
6020 Hg-SPK	MERC-STK1	0.05		49.95	50	1000
Hg (Intermediate) C	MERC-STK2	0.25		249.75	250	1000
6020-SPK (intermediate)	Bi-STK	0.2		4.6	10	20,000 / 250,000 / 500,000
	Th-STK	0.2				
	HP7375	5				
6020-SPK2 (intermediate)	HP7376	1		9	10	20,000
6020-SPK3 (intermediate)	HP7379	1		9	10	20,000 / 10,000
CAL-SPK1 (intermediate)	HP7375	0.25		9.5	10	25000/12500/1000/500/10
	HP7379	0.05				
	HP7376	0.05				
	6020Hg-SPK	0.1				
	Bi/Th Intermediate	0.05				
Cal 0	N/A	N/A		50	50	0
Cal 1	ZPACEMN103	0.1		9.7	10	Varied
	ZPACEMN104	0.1				
	Hg 10ppb (intermediate)	0.1				0.1
Cal 2	CAL-SPK1	0.1		9.9	10	250/125/10/5/0.1
Cal 3	CA:L-SPK1	0.5		9.5	10	1250/625/50/25/0.5
Cal 4	CAL-SPK1	1		9	10	2500/1250/100/50/1

Cal 5	CAL-SPK1	2.5		7.5	10	6250/3120/250/125/2.5
Cal 6	CAL-SPK1 (intermediate)	5		-	5	25000/12500/1000/500/10
CRDL	ZPACEMN-103	0.1		9.6	10	varied
	ZPACEMN-104	0.1				
	6020 Hg-SPK	0.2				0.2
ICS-A	ICS-ICPMS	0.25		9.75	10	25000/500
ICS-AB	ICS-ICPMS	0.25		9.56	10	27500/26200/1250/600/100/50/4
	6020-SPK	0.05				
	6020-SPK2	0.05				
	6020-SPK3	0.05				
	6020Hg-SPK	0.04				
ICV / CCV add Hg	XPACEMN-75	0.05		49.31	50	4/80/1000
	XPACEMN-76	0.02				
	Bi/Th Intermediate	0.4				
	XPACEMN-77	0.02				
	Hg Intermediate C	0.2				

[1]Alternate final volumes may be prepared at the discretion of the scientist, so long as the concentrations specified above are maintained.

Appendix D: Stock Standard Summary

Stock Standard Concentrations

	HP7379	HP7376	HP7375	XPACEMN 77	XPACEMN 76	XPACEMN 75	ZPACEMN 103	ZPACEMN 104	ICS-ICPMS	Agilent Tune	EPA Tune
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Aluminum	-		1000			1000	2		1,000		
Antimony		200		200				0.005			
Arsenic	200				200			0.05			
Barium	200				200		0.03				10
Beryllium	200				200		0.02				10
Bismuth							0.05				
Boron		200		200			1				
Cadmium	200				200		0.008				
Calcium			1000			1000	4		1,000		
Chromium	200				200		0.05				
Cobalt	200				200		0.05			10	10
Copper	200				200		0.1				
Iron			500			500	5		1,000		
Lead	200				200		0.01				
Lithium	200				200		0.05			10	10
Magnesium			1000			1000	1		1,000		10
Manganese	200				200		0.05				
Molybdenum		200		200				0.05	20		
Nickel	200				200		0.05				
Palladium		200		200				0.05			
Platinum		200		200				0.05			
Potassium			1000			1000	10		1,000		
Selenium	200				200			0.05			
Silicon			500			500		10			
Silver	100				100		0.05				
Sodium			1000			1000	5		1,000		
Strontium	200				200		0.05				
Thallium					100		0.01			10	10

Tin		200		200		20		0.05			
Titanium		200		200		20		0.1	20		
Vanadium	200				200		0.1				
Zinc	200				200		0.5				
Uranium	200						0.05				10
Indium											10
Cesium					200						10
Cerium										10	
Yttrium										10	10
Rhodium											10
Thorium							0.05				

Single Element Stock Standard Concentrations

	Bi-STK (Spex)	Bi-STK (Agilent)	6020-Th (Spex)	6020-Th (Agilent)	MERC-STK1	MERC-STK2	HG-LL Stock	6020-Ge	6020-Sc	6020-Tb	6020-In	6020-Ir
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Bismuth	1000											
Bismuth		1000										
Thorium			1000									
thorium				10000								
Mercury					1000							
Mercury						1000						
Mercury							10					
Germanium								1000				
Scandium									10000			
Terbium										1000		
Indium											1000	
Iridium												1000

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0054 **Revision:** 04

Document Title: Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1

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ENV-SOP-MIN4-0054 - Mercury

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Jul 2020, 05:04:19 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Krista Carlson (004514)	Project Coordinator 1	20 Jul 2020, 11:18:09 AM	Approved
Andrew Mickelson (009792)	Manager	20 Jul 2020, 11:31:19 AM	Approved
Adam Haugerud (005828)	General Manager 2	31 Jul 2020, 10:38:58 AM	Approved

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of mercury in mobility procedure extracts, aqueous wastes, ground waters, soils, sediments, bottom deposits, and sludge-type materials using cold vapor atomic absorption (CVAA).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The default reporting limit (RL) or Limit of Quantitation (LOQ) for mercury in liquid is 0.2 µg/L. The default reporting limit for mercury in soil is 0.02 mg/kg. Reporting limits may vary based on the nature of the individual sample matrix. For certain applications, a lower level method optimized for sensitivity in which the reporting limit is 0.010 µg/L is available. This is for aqueous samples only.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

2.0 SUMMARY OF METHOD

2.1 The method, a CVAA technique, is based on the absorption of radiation at the characteristic wavelength of 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

2.2 Chemical Reactions - Organic mercury compounds are decomposed by digestion with potassium permanganate in acid solution. The mercuric ions are then reduced to the elemental state with stannous chloride and mercury vapor is produced.

3.0 INTERFERENCES

3.1 Potassium permanganate is added during digestion of samples to break down organo-mercury compounds which would otherwise not respond to the cold vapor technique. A heating step is required for methyl mercuric chloride when present in or spiked to a natural system. Possible sulfide interferences are also eliminated by the addition of potassium permanganate. EPA studies indicate concentrations as high as 20 mg/L of sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, EPA studies indicate copper concentrations as high as 10 mg/L had no effect on recovery of mercury from reagent water.

3.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. The design of the dedicated mercury analyzer assures that this does not occur.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the

laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	30 mL	Acidified with nitric acid to pH<2, stored ambient	Must be analyzed within 28 days of collection.
Solid	8 oz glass jar	0.3 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either stored at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
Mercury analyzer, computer controlled	Cold Vapor Atomic Adsorption (CVAA), Cetac M-7600 or equivalent. Each instrument has an associated auto-sampler, Cetac ASX 520 or equivalent
Hot Block™ digester	54 place block or equivalent, Environmental Express SC154 or equivalent
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Peristaltic pump tubing	Fisher Scientific or equivalent
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Resin Pellets	Environmental Express SC400 or equivalent
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent
Sulfuric acid	Fisher Scientific P/N A510-P212 or equivalent
Potassium permanganate solution	Dissolve 100 g potassium permanganate in a minimum volume of reagent water and dilute to 2000 mL with reagent water. Store the reagent at room temperature in either a plastic or glass container. This solution expires 3 months from preparation date. Fisher Scientific brand reagents or equivalent.
Sodium chloride - Hydroxylamine hydrochloride solution	Dissolve 240 g sodium chloride and 240 g hydroxylamine hydrochloride in reagent water and dilute to 2000 mL with reagent water. Store the standard at room temperature in either a plastic or glass container. Solution expires 1 month from preparation date. Fisher Scientific brand reagents or equivalent.
Potassium persulfate solution (5%)	Dissolve 100 g of potassium persulfate in reagent grade water and dilute to 2000 mL. This solution expires 3 months from the preparation date. Fisher Scientific brand reagents or equivalent.
Rinse solution	Add 48 mL concentrated hydrochloric acid to 800 mL water, add 24 mL concentrated nitric acid and dilute to 1 L with reagent water. Store in 5L Nalgene container at room temperature. The solution expires 1 week from preparation date.
Stannous Chloride	Add 140 mL concentrated hydrochloric acid and 200 grams SNCl2-2H20 to 2000 mL reagent water. Different amounts may be made based on need. Store in bottle marked “Stannous Chloride” at the instrument. Fisher Scientific brand reagents or equivalent.
Aqua Regia	Mix 3 parts concentrated hydrochloric acid with 1 part concentrated nitric acid. Use fresh daily, expires within 24 hours.

8.2 Standards

Standard	Description
Mercury Calibration Stock Solution	1000 mg/mL, NIST traceable standard. Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
Intermediate Working Calibration Solution[1]	50 ug/L intermediate final concentration. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed. See appendix B for composition.
ICV/CCV Mercury Stock Solution	1 ug/mL, NIST traceable standard. Must be from a separate source than the mercury calibration stock source. Spex-Certiprep or equivalent.
Low Level Mercury Calibration Stock Solution	10 mg/L, NIST traceable standard. Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
Low Level ICV/CCV Mercury Stock Solution	10 mg/L, NIST traceable standard. Must be from a separate source than the mercury calibration stock source. Inorganic Ventures or equivalent.
Low Level Mercury Calibration Intermediate Standard[1]	1 ug/L final concentration. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed.

	See appendix B for composition.

8.2.1 Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed.

8.2.2 SW-846 series methods for mercury require that calibration standards are processed like samples including heating while EPA 245.1 specifically prohibits the calibration standards from being heated. Daily calibration records are documented in the electronic Prep Log.

9.0 PROCEDURE

9.1 Water

9.1.1 Sample Preparation

9.1.1.1 Prepare a method blank (MB) by transferring 30 mL of reagent grade water to a new 50 mL digestion cup. Label with the LIMS batch number and sample number.

9.1.1.2 Prepare a laboratory control sample (LCS) by transferring a 0.15 mL aliquot of the stock mercury standard to a 50 mL cup. For low level mercury samples, transfer 0.15 mL aliquot of the low level mercury intermediate standard. Bring the total volume to 30 mL with reagent water. Label with the LIMS batch number and sample number.

9.1.1.3 Shake sample to achieve homogeneity. Maximum sample volume is 30 mL. Use this or a smaller volume diluted to 30 mL. Place the sample into the 50 mL cup labeled with the corresponding LIMS sample number. Record sample volume in the Hg CVAA Sample Preparation Log.

9.1.1.4 Prepare an MS/MSD by transferring 0.15 mL aliquot of the stock mercury standard to 50 mL cups. For low level mercury samples, transfer 0.15 mL aliquot of the low level mercury intermediate standard. Bring the total volume of each to 30 mL with sample.

9.1.1.5 To all samples (including QC) add 1.5 mL concentrated sulfuric acid and 0.75 mL concentrated nitric acid, mixing well after each addition.

9.1.1.6 To all samples (including QC) add 5 mL potassium permanganate. If the purple color disappears, the sample is re-batched and re-prepped at a lower volume.

9.1.1.7 To all samples (including QC) add 2.5 mL of potassium persulfate solution and swirl to mix.

9.1.1.8 Loosely cap each cup and place into the digestion block, maintained at a temperature of 95°C ± 2°C and heat for two hours. Observe the initial temperature and time in the block.

9.1.1.9 After the two hour digestion, remove the samples from the block and cool. Observe the time the samples were removed from the block, as well as the final temperature of the block.

9.1.1.10 To all samples (including QC) add 1.8 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine

hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.1.2 Documentation – Digestion Records

Record the observations and necessary information in the electronic preplog using template version F-MN-I-342-Rev.02. Information includes batch and sample ID, initial and final times, temperatures, volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. The initial and final times and temperatures will be representative of the elapsed time for the batch.

9.2 Solid/Semi-Solid

9.2.1 Sample Preparation

9.2.1.1 Prepare a MB by weighing 0.3 g of resin pellets in a 50 mL cup.

9.2.1.2 Prepare a LCS by weighing 0.3 g of resin pellets in a 50 mL cup and spiking with a 0.15 mL aliquot of the ICV/CCV working mercury standard.

9.2.1.3 Weigh a representative 0.3-0.36 g portion of sample in a 50 mL cup.

9.2.1.4 Weigh two additional samples for matrix spike/matrix spike duplicate (MS/MSD) and spike carefully to get these samples as close to the weight of the unspiked sample used for QC, as possible. Spike both the MS and MSD with 0.15 mL of the mercury ICV/CCV working standard.

9.2.1.5 To all samples (including QC) add 3 mL DI water.

9.2.1.6 To all samples (including QC) add 3 mL aqua regia (see 10.1 above).

9.2.1.7 Place in hot block, maintained at 95°C ± 2°C and heat for 2 minutes. Record this time and temperature as the initial start time.

9.2.1.8 Remove from hot block and allow to cool.

9.2.1.9 Bring all samples (including QC) up to a volume of 30 mL with DI water.

9.2.1.10 To all samples (including QC) add 9 mL potassium permanganate. If the purple color disappears, re-prepare the sample, MB, and LCS with less DI and the corresponding amount of potassium permanganate added so that final volume does not exceed 30 mL. Additional permanganate is noted as a comment on the prep form.

9.2.1.11 Loosely cap each cup and return samples to hot block digester, maintained at a temperature of 95°C ± 2°C and heat for 30 minutes.

9.2.1.12 Remove the samples from the block and record the final time and the temperature. Allow the samples to cool.

9.2.1.13 To all samples (including QC) add 3.6 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.2.2 Documentation – Digestion Records

Record the necessary information in the electronic preplog using template version F-MN-I-343-Rev.03. Information includes batch and sample ID, initial and final times, temperatures, volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. The initial and final times and temperatures will be representative of the elapsed time for the batch.

9.3 Equipment Preparation & Analysis

9.3.1 Turn on the computer and load the software. Turn on, or 'wake up' the instrument and allow the lamp to warm up for about 90 minutes from a cold shut down (lamp off, main power off and gas off) and 5 minutes from standby (lamp off, main power on and gas off). Check the following:

9.3.2 Prepare any necessary reagents and record the appropriate information (volumes, manufacturer, lot numbers, etc.) in the standard solution log.

9.3.3 Check instrument waste and empty as needed.

9.3.4 Perform any routine maintenance as needed and record in maintenance log.

9.3.5 Check the $KMnO_4$ trap at the back of the instrument to make sure it is filled with crystalline $KMnO_4$ and not wet or spent (the brown MnO_2 color approaches the open end of the trap).

9.3.6 Fill the rinse solution container with rinse solution, if needed, and move the probe down into the rinse well.

9.3.7 Check peristaltic pump tubing installation, make sure tension is adjusted if needed, and turn pump on.

9.3.8 Place the $SnCl_2$ line in DI water.

9.3.9 Initialize the wetting of the GLS by selecting 'wet the gas liquid separator post' option in the software. This increases the gas flow to 300-350 mL/min and ramps the pump speed to 100%. Pinch the waste line tubing shut with your fingers. Watch the bubbles and ensure that 1-2 bubbles completely propels to the top of the chamber, wetting the entire post and the top. As soon as this happens, open the waste line tubing so the GLS can drain.

9.3.10 Inspect the GLS to make sure it is draining completely and liquid is not pooling.

9.3.11 Attach the sample gas line to the nafion dryer cartridge.

9.3.12 Fill the stannous chloride bottle with stannous chloride.

9.3.13 Place the $SnCl_2$ line into the $SnCl_2$ solution bottle.

9.3.14 Create a worksheet for analysis by selecting 'new from' in the file menu. Enter the name, ie 20Aug15 (DDMMMYY), a, b, c etc. (if more than one run is performed that day) soil or water to indicate sample matrix, and instrument ID number. The program will then go to the Method Editor page.

9.3.14.1 In the conditions page in the Method Editor, check the instrument settings including the time profile (baseline correction and read time delays). To do this, read a standard and move the baseline correction window and read time window accordingly if needed.

9.3.14.2 Check the Standards page to ensure the correct calibration parameters and standards are entered.

9.3.14.3 Check the QC tests page to make sure the correct test solutions and parameters are entered if the software is to calculate recoveries during analysis.

9.3.15 Create a sequence in the sequence editor tab and enter sample IDs or import them from LimsLink.

9.3.16 Start analysis, monitor all initial QC checks. If initial QC fails, make adjustments if needed and re-calibrate. If checks pass criteria, continue with sample analysis.

9.3.17 After analysis, print out a report and transfer valid data into LIMS system via LimsLink.

9.3.18 After completing sample analysis for the day, shut down the instrument.

9.3.18.1 Place the $SnCl_2$ line in 10% HNO_3 and run for ~10 minutes. After this move the probe up out of the rinse well and place the $SnCl_2$ line in DI water and run for 2-5 minutes. Remove from DI and allow the line to run dry. Turn off pump, disconnect the clamps, and loosen pump tubing.

9.3.18.2 Disconnect the sample gas line from the nafion dryer cartridge.

9.3.18.3 Turn off the gas and the lamp.

9.3.18.4 If the instrument will be used in the next day or two, leave it in the stand-by mode. If not, do a cold shut down and turn off the software, instrument, auto sampler and auto diluter.

9.4 Routine Instrument Operating Conditions

Parameter	Setting
Sample Probe Depth (mm)	145
ASX Rinse Pump Speed (%)	50
Sample Uptake Time (s)	45
Rinse Time (s)	95
Gas Flow (mL/min)	100
Pump speed (%)	50
Read Delay time (s)	55.50
Replicate read time (s)	1.50
Replicates	4

9.5 Initial Calibration

9.5.1 Calibration Design

9.5.1.1 The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of four integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Instruments must be calibrated at a minimum of once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data.

9.5.1.2 Additional calibration specifications may be referenced in ENV-SOP-NW-0027 *Calibration Procedures*, or equivalent replacement.

9.5.2 Calibration Sequence

Calibration Blank (CAL0)
CAL1
CAL2
CAL3
CAL4
CAL5
ICV
ICB
CRDL
CCV
CCB
Client samples
CRDL
CCV
CCB

9.5.3 ICAL Evaluation

9.5.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.995.

9.5.3.2 Relative Standard Error (RSE)

%RSE is evaluated after all calibration points have been measured. In order for a standard curve to be acceptable, the %RSE acceptance criteria is 80%-120% must be observed.

Note: %RSE is analogous to %RSD. 40CFR Part 136 allow %RSE to be used in place of correlation coefficient (R) or coefficient of determination (r^2) for the acceptability determination of the curve.

9.5.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.5.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 The percent recovery in the LCS is calculated using Equation 1:

Equation 1

$$\%\,\mathrm{Recovery} = \frac{SR}{SA} \mathrm{x}100$$

Where, SR = LCS result (ug/L or mg/kg)
SA = spike added, ug/L or mg/kg

10.2 The percent recovery of mercury in the matrix spike and matrix spike duplicate is calculated using Equation 2:

Equation 2

$$\%\,\mathrm{Recovery} = \frac{(SSR - SR)}{SA} \mathrm{x}100$$

Where, SSR = Spiked sample result, mg/L or mg/kg
SR = Sample result, mg/L or mg/kg
SA = Spike added, mg/L or mg/kg

10.3 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 3:

Equation 3

$$\%RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where, S = Sample result, mg/L or mg/kg
D = Duplicate sample result, mg/L or mg/kg

10.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L
v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \mathrm{x}100$$

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 7470/7471. 1 per batch of 10 or fewer samples for 245.1
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	Performed at client request.
Post Digestion Spike	Performed at client request.
Filter Blank (FB)	1 per batch of 20 or fewer samples when applicable.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run. May be run more frequently per state or client requirement.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-NW-0018 *Determination of LOD and LOQ* for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-NW-0025 *Training and Orientation Procedures* for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Use of Block Digestor- Heating is conducted with hot block digestion as the heating equivalent mentioned in SW 846 7471B (section 6.10) and SW 846 7470. This is also compliant with method 245.1 under the Clean Water Act method flexibility in 40CFR section 136.6 (b) (4) (iii).

14.2 The lab utilizes a 30 mL final volume, all solid weights and reagent ratios are conducted based on the 0.3 g versus the 0.5 g initial weight accordingly.

14.3 Mercury calibration standards are prepared and digested weekly for SW-846 analysis of soils and waters. The stability and performance of standards prepared weekly has been evaluated and documented.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – QC Summary

Appendix B – Working Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7470A, 1994.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471A, 1994.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7000a, Revision 1, July 1992.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471B, Revision 2, Feb 2011.

Methods for Chemical Analysis of Water and Wastes, Method 245.1. Rev.3.0, 1994.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

Minnesota Pollution Control Agency, Laboratory Quality Control and Data Policies, July 2011.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
Appendix A	Updated MB Acceptance Criteria and Corrective Action.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	03

Appendix A: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	r ≥ 0.995 RSE < 20%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	± 10% for SW-846 7000 series methods and ± 5% for 245.1	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR and West Virginia require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV[4]	At the beginning of each run. Depending on data quality objectives it may be required that a CRDL bracket samples.	± 30% (or specified by the client)	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the CRDL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.
CCV[5]	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R):	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.

CCB	Daily, before sample analysis, after every 10, and at end of analytical window	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR and West Virginia require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Method Blank	One per 20 samples	Method 7470/7471: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. Method 245.1: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1.	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD[1]	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1 % RPD ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD[2,3]	One per 20 samples for 7470/7470A and 7471/7471B. One per 10 samples for 200.8	80-120% for 7470/7470A[3] and 7471/74/1B. 245.1: 70-130% %RPD: 20%	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria,	Qualify analytes with MS out of criteria.

			the MSD sample and associated parent sample need to be flagged. If MS or MSD fails and spike amount is less than 4 times the native concentration in the sample, remove M1 flag and replace with P6 flag. If the RPD is outside the limit, report the data and footnote the samples with precision outliers. The footnote only applies to samples within the same batch containing the sample used for the MS and MSD analyses.	
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Post Digestion Spike	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x FB detections, report the data.	Qualify outages and explain in case narrative.

[1]WIDNR requires the use of a lab created matrix solution from unused samples.

[2]In the event that only samples identified as Equipment Blanks and/or Field Blanks are available, and LCS/LCSD will be prepared in place of MS/MSD.

[3]In the absence of method specified recovery limits, results will be evaluated based on specifications outlined by the MPCA guidelines for Inorganic Analysis.

[4]A reporting limit verification is performed by analyzing a CRDL at ± 30% while the method has no low end criteria.

[5] ICV/CCV criteria is ± 10% while the 7000 series indicates ± 20%, the tighter criteria is applied to allow for instrumentation to be utilized for any mercury method throughout an analytical shift.

Appendix B: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Solvent	Solvent Volume (mL)	Final Total Volume (mL)	Final Concentration (µg/L)
Mercury Calibration Intermediate.	Mercury Stock (10 ug/mL)	5	Reagent water	985	1000	50
	Concentrated nitric acid	10				
Standard 0	Intermediate Standard (50 µg/L)	0	Reagent water	30	30	0
Standard 1		0.12		29.88		0.2
Standard 2		0.6		29.4		1.0
Standard 3		1.8		28.2		3.0
Standard 4		3.0		27		5.0
Standard 5		6.0		24		10
CRDL		0.12		29.88		0.2
ICV/CCV	Mercury Stock 1000 mg/mL	0.15	Reagent water	29.85	30	5.0
ICB/CCB	N/A	N/A	Reagent water	30	30	0
Low Level Mercury Calibration Intermediate Standard; Prepare every 6 months.	Calibration Mercury Stock (10 mg/L)	0.100	Reagent water	984.9	1000	1.0
	Concentrated nitric acid	5.0				
	Concentrated hydrochloric acid	10				
Standard 0	Intermediate Standard (1.0 µg/L)	0	Reagent Water	30	30	0
Standard 1		0.30		29.7		0.010
Standard 2		0.75		29.25		0.025
Standard 3		1.5		28.5		0.050
Standard 4		3.0		27		0.100
Standard 5		6.0		24		0.200
CRDL		0.30		29.7		0.01
Low Level Mercury ICV/CCV **Intermediate** Standard. Prepare every 6 months	ICV/CCV Mercury Stock (10 mg/L)	0.4	Reagent water	184.6	200	20
	Concentrated nitric acid	5.0				
	Concentrated hydrochloric acid	10				
Low Level Mercury ICV/CCV	Low Level Mercury ICV/CCV Intermediate (75 µg/L)	0.15	Reagent water	29.85	30	0.10
Lower Level Mercury ICB/CCB	N/A	N/A	Reagent water	30	30	0

ATTACHMENT B-3

DUST LABORATORY SOPs

Attachment B-3
Dust Laboratory SOPs
Index

Laboratory	SOP Number	Revision #	Effective Date	SOP Title	# Pages
Pace	ENV-SOP-MIN4-0056	4	10/06/21	Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B	11
Pace	ENV-SOP-MIN4-0052	5	07/31/20	Metals Analysis by ICP - Method 6010 and 200.7	22
Pace	ENV-SOP-MIN4-0043	4	02/22/21	Metals Analysis by ICP/MS - Method 6020 and 200.8	24
Pace	ENV-SOP-MIN4-0054	4	07/31/20	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	20

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Document Information

Document Number: ENV-SOP-MIN4-0056 **Revision:** 04

Document Title: Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B

Department(s): Metals

Date Information

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Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0056 **Revision:** 04

Title: Metals Preparation of Solid Samples for Analysis by ICP and ICP-MS by 3050B

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0056

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Sep 2021, 12:40:17 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	General Manager 2	01 Oct 2021, 05:17:47 PM	Approved
Andrew Mickelson (009792)	Manager	06 Oct 2021, 02:22:12 PM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Preparation of Solid Samples for Analysis by ICP and ICPMS
TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the preparation of solid samples using hot block digestion as described in EPA Method 3050B.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in the associated analytical SOP; SOP ENV-SOP-MIN4-0052 *Metals Analysis by ICP - Method 6010 and 200.7* or ENV-SOP-MIN4-0043 *Metals Analysis by ICP/MS - Method 6020 and 200.8* (or equivalent replacements).

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to sediments, sludges and soil samples.

2.0 SUMMARY OF METHOD

A one-gram aliquot sample is digested in concentrated nitric acid, hydrochloric acid and hydrogen peroxide. After digestion, samples are brought to a final volume of 50mL. Digestates are then analyzed using Inductively Coupled Plasma (ICP) technologies for the determination of metals in solution.

3.0 INTERFERENCES

Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed in accordance with the quality control requirements given in SW-846 Sec. 8.0 to aid in determining whether Method 3050B is applicable to a given waste.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

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The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

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7.1 Equipment

Equipment	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Hot Block ™	54 Place Hot Block	Environmental Express
Analytical Balance	Ability to weigh to the nearest 0.01g	Fisher Scientific or equivalent

7.2 Supplies

Supply	Description	Vendor/Item #/Description
Digestion Cups	50 mL verified to class A specification	Environmental Express or equivalent
Vapor Recovery Device	Reflux cap or Watch glass	Environmental Express or equivalent
Resin beads	For solid matrix QC	Environmental Express or equivalent

8.0 Reagents And Standards

8.1 Reagents

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II	Verify that background levels of volatile compounds are acceptable by analysis
Hydrogen Peroxide	30% ACS Grade	Fisher brand
Hydrogen Peroxide	30%, Optima Grade for tin only	Fisher brand
Concentrated nitric acid (HNO_3)	Trace Metal grade	Fisher brand
Concentrated hydrochloric acid (HCl)	Trace Metal grade	Fisher brand

8.2 Standards

Standard	Concentration/Description	Requirements/Vendor/Item #
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are METALS-STK1 and METALS-STK2. See Appendix A for composition	Purchased from Inorganic Ventures (or equivalent). Store at room temperature. Expires as specified by manufacturer.
Mercury Spike – Stock solution standards for LCS and MS/MSD	10 µg/mL Hg-STK Stock	Purchased from Spex Certiprep. Store at room temperature. Expires as specified by manufacturer.

9.0 Procedure

9.1 Equipment Preparation

9.1.1 Support Equipment

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Verify the calibration of variable and fixed volume pipettes as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

Verify the calibration for the thermometer as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

9.1.2 Equipment

The hot block digestors are set to maintain a digestion temperature of 95 +/- 5°C. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature should be checked in different wells of the hot blocks such that all wells are evaluated over a period of time. Record the temperature of each hot block daily in the temperature logbook.

Balances shall be checked prior to use on each working day with a NIST traceable reference in the expected range of use. Balances must be verified with weights of a class appropriate for the accuracy of the balance being calibrated. Verify the calibration for the balance as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Record the measurements of each weight in the daily balance verification logbook.

9.2 Sample Preparation

9.2.1 Obtain and label digestion tubes in the order for which samples will be weighed out.

9.2.2 Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh a 1-1.1g portion of sample (to the nearest 0.01g) and transfer to a 50 mL digestion cup. Alternative sample volume may be used based on sample matrix. Weigh out 3 aliquots for the batch QC sample (background, matrix spike (MS), and matrix spike duplicate (MSD) being sure to weigh them as close to the same weight as possible.

9.2.2.1 Create a method blank and a laboratory control sample (LCS) by weighing out 1 gram of resin beads for each.

9.2.2.2 Spike the LCS, MS/MSD each of METALS-STK1 and METALS-STK2. If mercury is requested spike 0.25 mL of Hg-SPK stock.

9.2.3 Add DI to the 10mL marking for each sample.

9.2.4 Add 7.5mL of concentrated HNO3, mix the slurry, and cover with a reflux cap. Heat the sample to 95 +/- 5°C and reflux for 70 minutes without boiling. Record initial Hot Block temperature in the digestion log. Observe the sample during heating for brown fumes indicating oxidation of the sample. If this occurs, add up to an additional 5 mL HNO3 and re-heat. Repeat this process until no fumes are given off during heating. Record on the digestion log to what samples and how much additional acid was added.

NOTE: When mercury is a requested analyte, watch glasses will be used rather than reflux caps.

9.2.5 Cool the sample 10 minutes. Add 2.5mL of 30% hydrogen peroxide. Cover with reflux cap and return to the Hot Block for warming which will start the peroxide reaction. Care must be taken to ensure that losses do not occur due to vigorous effervescence. Heat until effervescence subsides for a total of 10 minutes. Cool the samples in the plastic cups.

NOTE: Use Optima grade hydrogen peroxide if the analysis of tin (Sn) is required. Tin is used as a stabilizer in the ACS grade of hydrogen peroxide.

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9.2.5.1 If effervescence does not subside, continue to add 30% hydrogen peroxide in 1mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Note in the comments section of prep sheet the additional aliquots.

NOTE: Do NOT add more than a total of 10mL hydrogen peroxide.

9.2.6 Add 5mL of concentrated HCl, return the sample to the Hot Block and reflux for an additional 15 minutes without boiling.

9.2.7 Remove samples from Hot Block and record final temperature in digestion log. Allow samples to cool. Bring samples up to a final volume of 50 ml with DI water. Cap and invert several times for proper mixing.

9.2.8 Samples may be allowed to sit overnight while solid materials settle out or samples may be centrifuged for 15 minutes at a rate of 1000 rpm. If samples are centrifuged, all QC samples including the method blank and laboratory control sample (LCS) must also be centrifuged.

9.3 Documentation

9.3.1 Digestion Records

Record the necessary information in the electronic preplog using template version F-MN-I-330-Rev.01. Information includes batch and sample ID, initial and final volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. Save file in prep log with the naming convention;

"Queue HBN Method" Ie. MPRP 555222 6020A

10.0 Data Analysis And Calculations

10.1 Calculations

Refer to associated analytical SOP for equations and common calculations.

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to associated analytical SOP for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.

11.2 Method Performance

11.2.1 Method Validation

11.2.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

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Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to the associated analytical SOP for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 The preparation method has been modified in terms of the amounts of reagents used and the individual heating times. The chemistry is maintained. Reason for this modification is better performance for silver and antimony. PT samples are analyzed regularly to validate that the modifications are effective. Per the method, the nitric acid and peroxide amounts are varied based on the sample reaction and this is the case with the Pace method. Overall, the Pace digestion ends up with a higher total acid concentration.

14.2 The final volume for the Pace method is 50 mL, opposed to 100 mL for the reference method.

14.3 Samples are processed using the Hot Block digestion system employing metals free disposable plastic ware rather than glass beakers.

15.0 Responsibilities

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Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
8.2	Updated concentration description for the metals spike
9.1.2	Include balance calibration verification
9.2.2.2	Update spike sources and volumes
9.3.1	Provide greater detail for documentation procedure ie batch nomenclature.
Appendix A	Added/updated spike sources
9.1.2	Include balance calibration verification
9.3.1	Provide greater detail for documentation procedure ie batch nomenclature.
9.2.2.2	Update spike sources and volumes

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0056	Metals Preparation of Solid Samples for Analysis by ICP and ICPMS by EPA Method 3050B	03

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Metals Preparation of Solid Samples for Analysis by ICP and ICPMS
TEST METHOD EPA Method 3050B
ISSUER: Pace ENV – Minneapolis – MIN4

Appendix A: Metals Standard Reference

Stock standards used for solid sample preparation

METALS-STK1		METALS-STK2		Hg-SPK	
ZPACEMN-116		ZPACEMN-106		MERC-STK1 Stock	
Element	(mg/L)	Element	(µg/L)	Element	(µg/L)
Ca	2000	Si	500	Hg	10000
Fe	2000	Sb	100		
Mg	2000	Mo	100		
K	2000	Sn	100		
Na	2000	Ti	100		
Al	2000	S	2000		
Ba	100	As	100		
Be	100	Pd	20		
Bi	100	Pt	20		
B	100	Se	100		
Cd	100				
Cs	100				
Cr	100				
Co	100				
Cu	100				
Li	100				
P	100				
Mn	100				
Pb	100				
Ni	100				
Ag	50				
Sr	100				
Tl	100				
V	100				
Zn	100				
U	100				
Th	100				

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ENVIRONMENTAL SCIENCES

Document Information

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ENV-SOP-MIN4-0052 - ICP

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	30 Jul 2020, 05:05:44 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Andrew Mickelson (009792)	Manager	20 Jul 2020, 02:32:20 PM	Approved
Krista Carlson (004514)	Project Coordinator 1	20 Jul 2020, 04:50:45 PM	Approved
Adam Haugerud (005828)	General Manager 2	31 Jul 2020, 11:01:55 AM	Approved

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved and total recoverable metals by Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is verified daily by running a QC solution (CRDL) at the LOQ and evaluating against method specific limits.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to drinking water, ground water, aqueous samples, liquid samples, leachates, industrial wastes, soils, sludges, sediments, and other solid wastes.

2.0 SUMMARY OF METHOD

Prior to analysis, samples are solubilized or digested using appropriate sample preparation methods. This method describes the determination of elements by ICP-OES. The method measures element-emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by a charge coupled device detector (CCD). All data is collected by simultaneous measurement. Software is used to measure and apply corrections due to background or inter-element interferences using a variety of techniques. Alternate wavelengths are also monitored for confirmation or to use in correction equations.

3.0 INTERFERENCES

3.1 Spectral Interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

3.2 Spectral overlap can be compensated by computer-correcting the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate

wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

3.3 Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. A high solids nebulizer is used on all instruments. Internal standards are also used to monitor and correct for physical effects.

3.4 Chemical interferences include molecular compound formation, ionization effects and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions, use of an ionization buffer, or by matrix matching of standards and samples.

3.5 Memory interferences result when analytes in a previous sample contribute to the signals measured in the new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from buildup of sample material in the plasma torch and spray chamber. Regular maintenance and awareness of samples with high concentrations minimize these interferences.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours if acidification is performed in the lab.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either stored at ambient or 6°C until sample preparation. Prepared sample digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPOES (Inductively Coupled Plasma Optical Emiison Spectrometer)	Agilent 720 or 5110 ICP instrumentation equipped with an CCD Detector, full wavelength region. Each instrument has an associated auto-sampler and recirculating chiller.
Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A or B volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Filters	Filtermate filters, 2 um PTFE, Environmental Express, SC0408
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type I – 18 megaohm
Nitric Acid (HNO_3), trace metals grade	Fisher Scientific, A-509-P212 or equivalent
Hydrochloric acid (HCl),trace metals grade	Fisher Scientific, A-508-P212 or equivalent
4% (v/v) Nitric Acid/5% (v/v) Hydrochloric Acid Solution	400 mL nitric acid (above) + 500 mL hydrochloric acid (above) to 10 liters with ASTM Type I water (18 megaohm). Used for all blanks and rinsing and preparation of standards.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Initial Calibration Verification (ICV) Stock Standard solutions	Custom blend. Must be separate stock from the calibration standards. Spex Certiprep or equivalent. See Appendix D for the standard information
Cesium Ionization Buffer for use with Agilent 720	50,000 PPM, High Purity Standards P/N 1B-CS-B5 or equivalent.
Wavelength Cal Solution - Agilent	Various analytes, Agilent P/N 6610030100
Internal Standards	Yttrium, Inorganic Ventures or equivalent

9.0 PROCEDURE

9.1 Equipment Preparation

Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following;

- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream
- Ar/O pressure - The argon supply pressure should be set at about 80-100psi. If the supply argon pressure falls below about 80psi, a safety interlock automatically shuts off the torch.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.

- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minute for break-in period
    - Adjust the pump-tubing in such a way to ensure proper flow prior to igniting the plasma. Decrease flow to where flow of bubble actually stops or barely moves. Turn knob 2 full turns.
- Ignite plasma while tubing is in a rinse solution, allow plasma to warm up at least 30 minutes and preferably 60-90 minutes.
- Use the warm up time to create the sequence and pour samples. Use Horizon Uploader to copy labels into the sequence.

9.1.1 Support Equipment

Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.

9.1.2 Instrument

9.1.2.1 Routine Instrument Operating Conditions

Instrument operating conditions vary by method and by instrument. All conditions are documented with each worksheet and cannot be modified after data has been generated. Instrument conditions are stored within a worksheet template. The analyst selects the appropriate Template for analysis. The analyst does not change operating conditions. Conditions are only changed during method development.

9.2 Initial Calibration

9.2.1 Calibration Design

A calibration curve consists of a single point standard and a calibration blank.

9.2.2 Calibration Sequence

Example Analytical Sequence

CAL0
CAL1
ICV
ICB
CRDLA
ICSA
ICAB
Fe 2000 SIC
Ca 2000 SIC/LDR
Al 1000 SIC/LDR
Mg 1000 SIC/LDR
Mn 100 SIC
CCV

CCB
Ba 20 SIC/LDR
Cr 50 SIC/LDR
Co 20 SIC/LDR
As 10 SIC
V 20 SIC/LDR
Cu 20 SIC/LDR
Ni 50 SIC/LDR
Ti 30 SIC/LDR
Mo 10 SIC/LDR
Zr 20 SIC
CCV
CCB
P 50 SIC
Ce 10 SIC
LDR A
LDR B
LDR C
CCV
CCB
CLIENT SAMPLES
CCV
CCB
CRDLA

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a single point calibration model, a linear regression curve is established using a calibration blank and one non-zero standard.

9.2.3.2 Relative Standard Error (RSE)

With a single point calibration model using a calibration blank and one non-zero standard, relative standard error evaluation is not applicable.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV, followed by an ICB, is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

9.3 Sample Preparation

9.3.1 Label all sample tubes so that each sample can be uniquely identified on the rack.

9.3.2 If any samples in a batch need to be filtered because of suspended material, use an Environmental Express Filtermate. The Method Blank and LCS must also be filtered if any samples are. Record the ID of the Filtermates used.

9.3.3 Centrifuge soil samples to minimize need for filtering.

9.3.4 Aqueous samples are poured without initial dilution unless historical data demonstrates otherwise.

9.3.5 Use Horizon Uploader to copy labels into the sequence.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 Quantitative Identification

10.1.1 Monitor all initial QC checks. One re-analysis of QC checks is allowed. If initial QC fails twice, make instrument modifications and recalibrate using a new worksheet from template.

10.1.2 During the sample analysis or after the analysis is completed, transfer valid data into LIMS system using LIMS LINK.

10.1.2.1 Export data from instrument to CSV file.

10.1.2.2 Open LIMSLINK

10.1.2.3 Click open instrument, select CSV file from list, data will import

10.1.2.4 Highlight QC + samples, select “Get LIMS Info”

10.1.2.5 Run QC will prompt for Q-Batch # plus standard selection

10.1.2.6 Sample data will prompt for SD/PDS source sample.

10.1.2.7 Right click on samples to select/de-select elements

10.1.2.8 Highlight samples to upload and select “Export Run to Epic Pro”.

Note: Be sure to make the appropriate selections in LIMSLNK rather than post-editing in EPIC. This provides for a much smoother experience and minimizes chance for error. If edits must be done in EPIC be sure to make edits prior to uploading new data from LIMSLINK, as this, again minimizes error due to confusion.

10.1.3 When Complete, select “excel bench sheet”. Save the Excel Bench sheet to the instrument folder marked “LIMSLINK RAW DATA” Use convention of run date (e.g. 032917ICP5). Note discrepancies in the notes section of the run log (including dilutions, QC issues, re-runs, etc.).

10.1.4 In LIMS system make final adjustments and add any required footnotes. Complete checklist and turn data in for validation.

10.1.5 Documentation is a mix of electronic and paper files. Key data must be stored electronically so that data review may be performed from any location. Some documents are stored in the physical daily folder and archived for easy reference.

10.1.6 Label a physical file with the date. Record the file name, Q-Batch, and all prep batches on the folder for each run that day (example: 032917ICP5 and 032917ICP5B.

10.1.7 Store printed copies of batch worklist reports, prep bench sheets, the original checklist, a printed copy of the IEC Form 10-IN generated from Gandolf, and a printed copy of the run log from LIMSLINK file in this folder. If the data reviewer requests additional printed information they may print it themselves. Note, if data is validated remotely print a copy of the validation verification e-mail and include with each checklist.

10.1.8 Generate a copy of the raw data and print to the X:Drive.

10.2 Calculations

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations*, or equivalent replacement, for equations for common calculations.

10.2.1 Inter-element Correction Factor (IEC) = Concentration of apparent concentration (observed) in mg/L / Concentration of Interferent in mg/L.

10.2.2 The percent recovery of the spike is calculated from the following equation:

$$\%\ \text{Recovery} = \frac{(SSR-SR)\ X\ 100}{ST}$$

Where:
SSR = Spiked Sample Result, ug/L or mg/kg dry
SR = Sample Result, ug/L or mg/kg dry
ST = Spike Target, ug/L or mg/kg dry

10.2.3 The relative percent difference between the MS/MSD can be calculated as follows

$$RPD = \frac{|(S-D)|\ X\ (100)}{(S+D)/2}$$

Where:
RPD = Relative Percent Difference
S = Original Spiked Sample Value, ug/L or mg/kg dry
D = Second Spiked Sample Value, ug/L or mg/kg dry

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.

Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6010B/C/D. 1 per batch of 10 or fewer samples for 200.7
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples for 6010B/C/D.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6010B/C/D.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification (ICV)	Immediately after each initial calibration.
Spectral Interference Check Solutions (SIC)	Immediately after each ICV/ICB.
Initial Calibration Blank	Immediately after each ICV.
Continuing Calibration Verification (CCV)	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6010B/C/D/200.7 and at a minimum of once at the end of each run for 6010C.
ICSA verification	At the beginning of each sample run sequence after the CRDL.
ICSAB verification	This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP.
Internal Standard	An appropriate internal standard is required.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-NW-0018 *Determination of LOD and LOQ* for these procedures.

11.3.2 Linear Dynamic Range (LDR)

Method 6010D requires that a LDR check sample be analyzed daily. Because of this requirement for 6010D, the LDR is established daily for all methods. For some elements a single element standard is used to establish the LDR while in other cases a mixed standard is used to establish the LDR. If an LDR standard is not analyzed for a particular analyte then the LDR defaults to the highest calibration point in the calibration curve.

Data is reported up to 90% of the LDR. When evaluating interferences use values up to the full LDR for the interferent. The LDR may be established at higher or lower levels on a daily basis based on expected levels of samples being tested that day. The LDR may vary daily depending on slight changes in instrument performance (things like pump tubing wear, etc.). Refer to Attachment VII for default linear ranges and the typical standards used to establish them

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-NW-0025 *Training and Orientation Procedures* for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action

when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

Appendix E – Check Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-V1-2009.

TNI Standard, Management and Technical Requirements for Laboratories Performing Environmental Analyses, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Water and Solid Waste, SW-846 3rd Edition, Final Update III, Revision 2, December 1996. Method 6010B.

Test Methods for Evaluating Water and Solid Waste, SW-846, Update IV, Feb. 2007. Method 6010C.

Test Methods for Evaluating Water and Solid Waste, SW-846, Update V, July 2018. Method 6010D.

Method 200.7 Revision 4.4, *Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry,* 1994.

US EPA Contract Laboratory Program Statement of Work ILM05.3, March 2004.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
17.0	Added years to 6010B & 200.7 references, updated formatting.
Appendix B	Updated MB Acceptance Criteria and Corrective Action for all methods. Updated Post Digestion Spike Acceptance Criteria for 6010B and 6010D.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0052	Metals Analysis by ICP – Method 6010 and 200.7	04

Appendix A: Target Analyte List and Routine LOQ

Table 1: Routine Analyte List and Limits of Quantitation (LOQ)[1]

Element	Water PRL (ug/L)	Soil PRL (mg/kg)
Aluminum	200	10
Antimony	20	1.0
Arsenic	20	1.0
Barium	10	0.50
Beryllium	5.0	0.25
Boron	150	7.5
Cadmium	3.0	0.15
Calcium	500	25
Chromium	10	0.50
Cobalt	10	0.50
Copper	10	0.50
Iron	50	2.5
Lead	10	0.5
Lithium	10	1.0
Magnesium	500	25
Manganese	5.0	0.25
Molybdenum	15	0.75
Nickel	20	1.0
Phosphorus	20	5
Potassium	2500	125
Selenium	20	1.0
Silicon	50	5
Silver	10	0.50
Sodium	1000	50
Strontium	5.0	0.5
Sulfur	500	25
Thallium	20	1.0
Tin	75	3.75
Titanium	25	1.25
Uranium	50	2.5
Vanadium	15	0.75
Zinc	20	1.0
Hardness	3300	N/A

[1] Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.

Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	A calibration curve must consist of a blank and at least one calibration standard.	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	± 10% for method 6010B, 6010C and 6010D or ± 5% for method 200.7 The RSD of the standards must be below 5% for 6010B, 6010C and 6010D and below 3% for 200.7.	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criteria of +/- ½ of the RL for method 6010D. All elements of interest must be evaluated to +/- the RL for method 6010B,6010C and 200.7. Criteria to be evaluated to method criteria unless otherwise specified by client.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDLA / LLCCV	The CRDLA must be analyzed at the beginning of each run for every analyte of interest. The CRDLA is analyzed at or below the RL. Additionally, the CRDLA must be analyzed after samples to bracket method 6010C samples.	± 40% (or specified by the client) For method 6010C, must be within ± 30% . For method 6010D, must be within.± 20%.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the CRDL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	For method 6010B, 6010C, 6010D and 200.7, the CCV must be within ± 10% of the true value. The RSD of the CCV must be below 5% for 6010B.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every	All elements of interest must be evaluated to a criteria of +/- the	Identify source of problem, re-analyze. Analysis may proceed if	Qualify analytes with

	10, and at end of analytical window	RL for 200.7, 6010B, 6010C and 6010D. Depending on the data quality objective of individual clients different criteria may apply.	it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	70-125% of its true concentration	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solution (ICSA)	A mixed solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM is analyzed at the beginning of each sample run sequence. In some specific client requirements the ICSA must bracket the run or the analytical batch.	Acceptance criteria for the spiked analytes are 80-120%. Unspiked analytes must have an absolute value less than the RL.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSA can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSA passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Interference check solution (ICSAB)	A solution containing concentrations of Al, Ca, and Mg at 500 PPM and Fe at 200 PPM with low to mid-range concentrations of target analytes as outlined in ILM5.3. This is analyzed following the ICSA when requested. This is required by certain clients. It is not a method requirement and need be analyzed only for clients specifying this in the QAPP	The acceptance criteria are 80-120% for all spiked analytes.	Identify and correct source of problem, repeat performance verification(s). Note: The ICSAB can be re-processed after appropriate SIC solutions are analyzed and the IECs are recalculated. If ICSAB passes, continue.	None. Do not proceed with analysis for elements that cannot be verified.
Spectral Interference Check Solutions (SIC)	SIC solutions are single-element solutions used to evaluate and correct IEC factors. Specific elements evaluated are listed in specific instrument methods.	Unspiked analytes must have an absolute value less than the RL.	If SIC fails, re-calculate IEC and re-process data. If a sample level exceeds an SIC level and the interfering element affects target analytes, then: a) run a higher SIC or b) dilute the sample.	None. Do not proceed with analysis for elements that cannot be verified.
Method Blank	One per 20 samples	Method 200.7: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed.	Qualify outages and explain in case narrative.

		Method 6010B, 6010C and 6010D: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	
LCS	One per 20 samples	80-120% for 6010B,6010C and 6010D 85-115% for 200.7	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 6010B,6010C and 6010D 85-115% for 200.7 %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	75-125% for 6010B, 6010C, and 6010D 70-130% for 200.7 % RPD: 20%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	One SD per batch. Method suggestion / Pace Policy, if reporting by 6010B, 6010C, or 6010D.	6010B/C: 1:5 dilution of sample, SD RPD should agree within +/- 10% of the original result when the original sample is greater than 10x the RL. 6010D: 1:5 Dilution of sample or MS, for concentrations 25x > LLOQ in parent sample, resultant RPD should agree within +/- 20%.	Data is qualified.	Qualify outages.
Post Digestion Spike	Method suggestion / Pace policy if reporting by 6010B, 6010C, 6010D and MS/MSD fail outside 75-125%	80-120% for 6010C 75-125% for 6010B and 6010D.	Data is qualified.	Qualify outages.

Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	All elements of interest must be evaluated to a criteria of +/- ½ the RL for method 6010D. All elements of interest must be evaluated to a criteria of +/- the RL for method 60106010B,6010C and 200.7. If the FB does not contain target analytes at a level that interferes with project-specific DQOs, then the FB would be considered acceptable.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range	If a SIC/LDR standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range. See Appendix C.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the data reporting range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved.	N/A

Note: In the absence of method specified recovery limits, results will be evaluated based on specifications outlined by the MPCA guidelines for Inorganic Analysis.

Appendix C: Linear Range Reference Table

Wavelength	LDR (PPM)	Standard	Type
Ag 328	2	CAL1	LDR
Al 237	1000	Al 1000 SIC/LDR	SIC/LDR
As 188	10	As 10 SIC	SIC
As 188	20	LDR B	LDR
B 249	20	LDR A	LDR
Ba 455***/Ba 585**	20	Ba 20 SIC/LDR	SIC/LDR
Ba 585*	50 0	Ba 50 SIC	SIC/
Be 234	4	CAL1	LDR
Ca 370	2000	Ca 2000 SIC/LDR	SIC/LDR
Cd 214	20	LDR B	LDR
Co 228	50	Co 50 SIC/LDR	SIC/LDR
Cr 267	20	Cr 20 SIC/LDR	SIC/LDR
Cr 267	50	Cr SIC/LDR	50
Cu 327	20	Cu 20 SIC/LDR	SIC/LDR
Cu 327	50	Cu 50 SIC/LDR	SIC
Fe 261	200	LDR C	LDR
Fe 273*	2000	Fe 2000 SIC	SIC
K 766***	200	LDR C	LDR
K 766**	20	CAL1	LDR
Li 670	4	CAL1	LDR
Mg 383	1000	Mg 1000 SIC/LDR	SIC/LDR
Mn 257	20	LDR B	LDR
Mn 293*	100	Mn 100 SIC	SIC
Mo 204	10	Mo 10 SIC/LDR	SIC/LDR
Na 589***	200	LDR C	LDR
Na 589**	20	CAL1	LDR
Ni 231	50	Ni 50 SICLDR	SIC/LDR
P 213	20	LDR B	LDR
Pb 220	100	LDR A	LDR
S 181	200	LDR C	LDR
Sb 206	20	LDR A	LDR
Se 196	20	LDR B	LDR
Si 251	20	CAL1	LDR
Sn 189	20	LDR A	LDR
Sr 421	4	CAL1	LDR
Ti 334	20	LDRA	LDR
Ti 334	30	Ti 30 SIC	SIC
Tl 190	20	LDR B	LDR
U	4	CAL1	LDR
V 292	20	V 20 SIC/LDR	SIC/LDR
Zn 206	50	LDR A	LDR

*Used for Interference Correction Only
** ICP4 Only
*** ICP5 Only

Appendix D: Standard Reference Tables

ICP Working Calibration Standard					ICP Calibration Verification Standard			
Element	Stock Conc. (mg/L)	Aliquot (mL)	Final Volume (mL)	Cal STD Final Conc. (mg/L)	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (mg/L)
Ag	100	1.0	50	2	50	1.0	50	1
Al	2,000	0.5	50	20	1000	0.5	50	10
As	200	1.0	50	4	100	1.0	50	2
Ba	200	1.0	50	4	100	1.0	50	2
Be	200	1.0	50	4	100	1.0	50	2
Ca	2000	0.5	50	20	1000	0.5	50	10
Cd	200	1.0	50	4	100	1.0	50	2
Co	200	1.0	50	4	100	1.0	50	2
Cr	200	1.0	50	4	100	1.0	50	2
Cu	200	1.0	50	4	100	1.0	50	2
Fe	2000	0.5	50	20	1000	0.5	50	10
K	2000	0.5	50	20	1000	0.5	50	10
Mg	2000	0.5	50	20	1000	0.5	50	10
Mn	200	1.0	50	4	100	1.0	50	2
Na	2000	0.5	50	20	1000	0.5	50	10
Ni	200	1.0	50	4	100	1.0	50	2
Pb	200	1.0	50	4	100	1.0	50	2
S	10000	0.1	50	20	10000	0.05	50	10
Sb	200	1.0	50	4	100	1.0	50	2
Se	200	1.0	50	4	100	1.0	50	2
Tl	200	1.0	50	4	100	1.0	50	2
V	200	1.0	50	4	100	1.0	50	2
Zn	200	1.0	50	4	100	1.0	50	2
Mo	200	1.0	50	4	100	1.0	50	2
B	200	1.0	50	4	100	1.0	50	2
Sn	200	1.0	50	4	100	1.0	50	2
Ti	200	1.0	50	4	100	1.0	50	2
Si	1000	1	50	20	500	1	50	10
Li	200	1	50	4	100	1	50	2
P	200	1	50	4	100	1	50	2
Sr	200	1	50	4	100	1	50	2
U	1000	0.2	50	4	1000	0.1	50	2

Appendix E: Interference Check Standard Reference Tables

ICSA				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Al	5000	10	100	500000
Ca	5000	10	100	500000
Fe	2000	10	100	200000
Mg	5000	10	100	500000

ICSAB				
Element	Stock Conc. (mg/L)	Aliquot in (mL)	Final Volume (mL)	Final Conc. (ug/L)
Ag	20	1.0	100	200
Al	5000	5.0	100	500000
As	10	1.0	100	100
Ba	50	1.0	100	500
Be	50	1.0	100	500
Ca	5000	5.0	100	500000
Cd	100	1.0	100	1000
Co	50	1.0	100	500
Cr	50	1.0	100	500
Cu	50	1.0	100	500
Fe	2000	5.0	100	200000
Mg	5000	5.0	100	500000
Mn	50	1.0	100	500
Ni	100	1.0	100	1000
Pb	5	1.0	100	50
Sb	60	1.0	100	600
Se	5	1.0	100	50
Tl	10	1.0	100	100
V	50	1.0	100	500
Zn	100	1.0	100	1000

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ENVIRONMENTAL SCIENCES

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QM Approval

Name/Signature	Title	Date	Meaning/Reason
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Management Approval

Name/Signature	Title	Date	Meaning/Reason
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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved and total recoverable metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

2.0 SUMMARY OF METHOD

Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. For the total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, mixed, and allowed to equilibrate for the required time prior to analysis.

Sample solutions are introduced by pneumatic nebulization into a plasma, in which desolvation, atomization and ionization occurs. Ions are extracted from the plasma through a differentially pumped vacuum interface and sorted on the basis of their mass-to-charge ratio. The ions transmitted through the quadrupole are detected by an electron multiplier. Ion intensities at each mass are recorded and compared to those obtained from external calibration standards to generate concentration values for the samples. Results are corrected for instrument drift and matrix effects using internal standards.

3.0 INTERFERENCES

Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instruments spectrometer. One way to solve this problem is to measure a different isotope for which there is no

interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. An example includes ClO+ (mass 51), which interferes with V, and must be corrected by measuring ClO+ at mass 53. When possible an interference free isotope should be chosen for measurement.

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of

solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours following acidification. Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are either stored at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Agilent 7700, 7800 7900 ICPMS instrumentation equipped with interference reduction technology. Each instrument has an associated auto-sampler, rough pump and recirculating chiller.

Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Collision Gas	Praxair or equivalent, Ultra high purity He, Ultra high purity H_2,
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent replacement
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent replacement
2% (v/v) Nitric Acid/1% (v/v) Hydrochloric Acid Solution	Used for instrument blanks, standards and dilutions. Prepared in 1 L increments utilizing a volumetric flask and transferring into a C&G narrow mouth storage bottle. This is measured by mixing 20 mL of HNO_3 trace metals grade acid and 10 mL of HCl trace metals grade acid and DI H2O, and bringing to volume of 1 L.
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Combine76 mL of HNO_3 trace metals grade acid and 38 mL of HCl trace metals grade and DI H2O, and bringing to volume of 1 G.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Agilent Tune Solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
EPA Tune solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
Internal Standard Stock Solution	Various suppliers; single element standards to be mixed prior to use with concentrations of 1,000 and 10,000 ug/mL
Working Standards	See Appendix C

9.0 PROCEDURE

9.1 Equipment Preparation

Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following:

9.1.1 Support Equipment

- Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide.
- Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream.
- Ar/O pressure - The argon supply pressure should be set at about 80psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- Helium / Hydrogen pressure - The helium and hydrogen supply pressure should be set at about 15 and 9 psi respectively.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.
- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minutes for break-in period.
- Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure: Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing sufficient concentrations of major cations. The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly degraded tip.

9.1.2 Instrument

Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:

- Torch Ignition - Click on the Plasma icon to open the Instrument window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide.)
- Warm-up- Instrument is allowed to warm-up 30 minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
- Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

- Start-up Configuration - Once the analysis tubing is placed in the Agilent tune solution and stable signal is achieved, the start-up configuration can be initiated. See section 9.1.2.1 for Agilent tune performance monitoring and criteria.
- Create New Experiment File – Open template from the drive. Apply the proper run name for the day (MMDDYYICPMS#). Introduce EPA tune solution and allow signal to stabilize. Initiate performance verification for each mode of analysis. Save each performance report to the network drive. See section 9.1.2.1 for EPA tune acceptance criteria.

9.1.2.1 Routine Instrument Operating Conditions

The instrument is configured to go through the manufacturer recommended startup tune procedure which includes; Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and standard mode performance verification. The measured ratios of oxides 156/140 and doubly charged 70/140 should be <3%. The measured masses of ^{7}Li, ^{89}Y, ^{205}Tl are monitored for initial resolution/axis tuning. EPA Performance verification is later performed for each cell condition used for sample analysis.

EPA Tune Verification - The EPA tuning standard must be analyzed in each mode of analysis to verify resolution and mass calibration are within the required specifications. The tuning standard is analyzed in each mode of analysis at least five times and the relative standard deviation (RSD) must be <5% for all analytes contained in the tuning standard. Conduct mass calibration and resolution checks in the mass regions of interest. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be <0.9 amu full width at 5% peak height.

Pace Minneapolis maintains approval for the analysis of up to 35 elements by the EPA Methods 200.8, 6020, 6020A, 6020B for water and soil matrices. All target analytes are analyzed either in a Helium mode (Collision Cell), hydrogen (Collision Cell), or No gas mode on the Agilent instruments depending on the sample matrix type. The use of interference reduction technologies (Collision Cell) is not allowed for drinking water analysis. Separate calibrations are performed for samples reporting by regulation of the SDWA.

9.2 Initial Calibration

9.2.1 Calibration Design

The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of at least three integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The working range varies with each analyte, see appendix C for summary. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Additional calibration specifications may be referenced in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing*, or equivalent replacement.

9.2.2 Calibration Sequence

Calibration Blank (CAL0)

CAL1
CAL2
CAL3
CAL4
CAL5
CAL6 (optional)
CAL7 (optional)
ICV
ICB
CRDL
ICSA
ICSAB
CCV
CCB
Client samples
CCV
CCB
CRDL (Optional)

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.998.

9.2.3.2 Relative Standard Error (RSE)

%RE is measured at the lowest calibration level and at a point near the mid-level of the calibration (the continuing calibration verification level is recommended). In order for a standard curve to be acceptable, the correlation coefficient/coefficient of determination criterion specified in the method must be met **and** both the low-level and mid-level %RE measures must meet the acceptance criteria. The low-level %RE acceptance criteria is 60%-140% and the mid-level is 90-110%.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

9.3 Digestate Preparation

9.3.1 Homogenization and Subsampling

All solid matrices are subject to centrifuge at a rate of 1000 rpm for 15 minutes or allowed to settle overnight prior to analysis. Once samples have been centrifuged or allowed to settle, an initial dilution of 20 fold is performed on each sample. This is completed by taking 4.75mL of 2% HNO3 / 1% HCL diluent and mixing with a 0.25mL aliquot of sample by means of vortex.

Aqueous samples are inverted multiple times and poured without initial dilution unless historical data demonstrates otherwise.

9.4 Analysis

The instrument performs sample analysis by executing 100 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.

The instrument(s) have been setup and configured in conjunction with manufacturer specifications. Masses were carefully selected to avoid and/or minimize interferences. Internal standard selection was based on performance for the appropriate mass range. Internal standard association must remain within 50 amu of targeted analyte.

The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volumes of well mixed sample aliquots must be prepared until the analysis solution contains < 0.1 mg/L silver.

10.0 Data Analysis And Calculations

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations*, or equivalent replacement, for equations for common calculations.

10.1 Hardness as CaCO3 in mg/L = 2.497 * [Ca in mg/L] + 4.118 * [Mg in mg/L]

10.2 Concentration of lead = summation of signals at 206, 207, and 208 m/z.

10.3 Silica (SiO2) (µg/L) = Silicon (Si) (µg/L) * DF * 60.09 amu (SiO2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

10.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L

v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \text{x}100$$

10.5 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 1:

Equation 1

$$\%RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where, S = Sample result, mg/L or mg/kg

D = Duplicate sample result, mg/L or mg/kg

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6020 (A)(B). 1 per batch of 10 or fewer samples for 200.8
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6020(A)(B).
Internal Standard	An appropriate internal standard is required for each analyte and sample determined by ICP-MS.

Internal Standard	Associated element
Scandium 45	Li, Be, B, Na, Mg, Al, Si, K, Ca, Ti, V, Cr, Mn, Fe, Se
Germanium 72	Co, Ni, Cu, Zn, As, Sr
Indium 115	Mo, Pd, Ag, Cd, Sn, Sb
Terbium 159	Ba, Pt, Hg, Tl, Pb, Bi
Iridium 193	U Th

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Tune	Daily prior to any calibration
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.
ICSA verification	At the beginning of each sample run sequence after the CRDL. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.
ICSAB verification	At the beginning of each sample run sequence after the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be near the midpoint of the calibration range. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or

extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Tuning criteria observed is more stringent than required by the SW846 methods so that the same criteria can be used for both methods 6020 and 200.8.

14.2 The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, thorium, and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.3 The following elements are not listed in the method 200.8 recommended analyte list: bismuth, boron, calcium, iron, lithium, magnesium, palladium, platinum, potassium, silica, silicon, sodium, strontium, tin, and titanium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.4 The following elements are not listed in the method 6020B recommended analyte list: bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 02/2007.

U.S. Environmental Protection Agency. SW846 Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, 7/2014.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

40 CFR Appendix B to Part 136, Definition and Procedure for the Determination of the Method Detection Limit - Rev 2, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
6.0	Updated sample retention from 45 to 21 days.
8.2	Internal Standard Stock Solution – added “1,000 and”
9.2.1	Updated 3 to 5 non-zero standards. Added “The working range…C for summary.”
9.2.2	Added “(optional)” to CAL6. Added “CAL7 (optional)”.
10.0	Added sections 10.4 and 10.5.
11.1	Updated Thoridium 232 to Iridium 193.
14.0	14.2 & 14.4: removed “-238” from uranium. 14.2: added thorium.
17.0	Removed references for Fisions and Region 9 Laboratory SOP.
Appendix A	Added Thorium. Updated Silica and Silicon entries. Removed Mercury NPW and potable water entries.
Appendix B	Updated ICAL Acceptance Criteria. Updated methods referenced in MB Acceptance Criteria. Added LDR acronym to QC Item.
Appendix C & D	Re-formatted tables.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0043	Metals Analysis by ICP/MS – Method 6020 and 200.8	03

Appendix A: Target Analyte List and Routine LOQ[1]

Analyte	Non-Potable Water (ug/L)	Potable Water (ug/L)	Soil (mg/kg)
Aluminum	20.00	20.0	20.00
Antimony	0.50	0.50	0.50
Arsenic	0.50	0.50	0.50
Barium	0.30	0.30	0.30
Beryllium	0.20	0.20	0.20
Bismuth	0.50	-	0.50
Boron	10.00	-	10.00
Cadmium	0.08	0.08	0.08
Calcium	40.00	-	40.00
Chromium	0.50	0.50	0.50
Cobalt	0.50	-	0.50
Copper	1.00	1.00	1.00
Iron	50.00	-	50.00
Lead	0.10	0.10	0.20
Lithium	0.50	-	0.50
Magnesium	10.00	-	10.00
Manganese	0.50	0.50	0.50
Mercury	-	-	0.20
Molybdenum	0.50	-	0.50
Nickel	0.50	0.50	0.50
Palladium	0.50	-	-
Platinum	0.50	-	-
Potassium	100.00	-	100.00
Selenium	0.50	0.50	0.50
Silica	214.00	-	214.0
Silicon	100.00	-	100.00
Silver	0.50	0.50	0.50
Sodium	50.00	-	50.00
Strontium	0.50	-	0.50
Thallium	0.10	0.10	0.10
Thorium	0.50	-	0.50
Tin	0.50	-	2.000
Titanium	1.00	-	1.00
Vanadium	1.00	1.00	1.00
Uranium-238	0.50	0.50	0.50
Zinc	5.00	5.00	5.00

[1] *Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.*

Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Tune	Daily prior to any calibration	Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. This must be completed using 5 replicates with a resulting RSD of <5%.	Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis.
ICAL	Daily	r ≥ 0.998 a Midlevel (recommended near ICV/CCV concentrations) %RE 90-110% Low-Level (Cal1) %RE 60-140%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	All analytes must be within ± 10% of the true value. (%R)	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A)(B) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.	For 6020/200.8: The acceptance criteria are ± 40% (or specified by the client). For 6020A: The acceptance criteria are ± 30% (or specified by the client). 6020B: The acceptance criteria is ± 20% (or specified by the client).	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve).	Qualify outages and explain in case narrative.

			If the CRDL is biased low, no data can be reported for the target elements failing criteria.	
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R): %RSD between multiple integrations must be ≤ 5%	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020 (B). WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	For method 6020, the intensity of internal standard in the ICB/CCB and ICS (ICSA/AB) standards must not deviate more than 80-120% from its original intensity in the associated calibration blank. The intensity of internal standard in the samples and remaining QC must not deviate more than 30-120%. For method 6020A/B, the intensity of the internal standard must not fall below 70% and not exceed 130% from its original intensity in the associated calibration blank. For Method 200.8 the intensity of internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solutions	ICSA containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti is analyzed at the beginning of each sample run sequence after the CRDL. ICSAB containing high concentrations of	ICSA all spiked elements are to be within 20% of the expected true value. The non-spiked elements are to be below the RL. ICSAB all spiked elements are to be within 20% of the expected true value.	Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis for elements that cannot be verified.

	C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.			
Method Blank (MB)	One per 20 samples	Method 200.8: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs. Method 6020, 6020A and 6020B: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	6020/6020A/6020B: 80-120% 200.8: 85-115%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	6020/6020A/6020B: 80-120% 200.8: 85-115% %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	6020/6020A/6020B: 75-125% 200.8: 70-130%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution[1]	One per batch of 20 samples or less		If criteria is not met, original sample and dilution shall be	Qualify outages.

		6020/6020A fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL. 6020B 1:5 dilution of sample 25x > LLOQ or 1:5 dilution of MS since reasonable concentrations are present, results to agree to ± 20%.	reanalyzed. If reanalysis fails, it is determined to be matrix interference.	
Post Digestion Spike[2]	One per batch if there is a MS failure.	6020/ 6020A 80-120% 6020B applicable to elements failing MS, results to agree to +/- 25%. Recommended if high concentration sample not available for dilution test.	If the element fails to meet the recovery criteria, reanalyze. If reanalysis fails, it is determined to be matrix interference.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Target analytes must be less than reporting limit. NC samples are required to be < ½ RL for target analytes. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range (LDR)	For method 6020B: Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the linear range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved as well as maintaining the minimum number of calibration standard requirements.	N/A

[1]To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of diluent. Note: this is a typical process for 200.8 and 6020W. It can be replicated for the preparation of highly concentrated samples by starting with a diluted "parent" sample and then performing the stepwise dilution process.
[2]To Prepare a Post Digestion Spike: An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. The spike addition should produce a minimum level of 10 times the lower limit of quantitation; routine spike volume is 0.020 mL of 20/250 mg/L and 1mg/L mercury stock concentration(s).

Appendix C: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Diluent Volume (mL)	Final Total Volume[1] (mL)	Final Concentration (ug/L)
Internal Standard	6020-Ge	1	See table 8.1	495	500	2000
	6020-Sc	1				
	6020-Tb	1				
	6020-In	1				
	6020-Ir	1				
Bi/Th primary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Bi/Th secondary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Hg 10ppb (intermediate)	HG-LL Stock	0.05		49.95	50	10
6020 Hg-SPK	MERC-STK1	0.05		49.95	50	1000
Hg (Intermediate) C	MERC-STK2	0.25		249.75	250	1000
6020-SPK (intermediate)	Bi-STK	0.2		4.6	10	20,000 / 250,000 / 500,000
	Th-STK	0.2				
	HP7375	5				
6020-SPK2 (intermediate)	HP7376	1		9	10	20,000
6020-SPK3 (intermediate)	HP7379	1		9	10	20,000 / 10,000
CAL-SPK1 (intermediate)	HP7375	0.25		9.5	10	25000/12500/1000/500/10
	HP7379	0.05				
	HP7376	0.05				
	6020Hg-SPK	0.1				
	Bi/Th Intermediate	0.05				
Cal 0	N/A	N/A		50	50	0
Cal 1	ZPACEMN103	0.1		9.7	10	Varied
	ZPACEMN104	0.1				
	Hg 10ppb (intermediate)	0.1				0.1
Cal 2	CAL-SPK1	0.1		9.9	10	250/125/10/5/0.1
Cal 3	CA:L-SPK1	0.5		9.5	10	1250/625/50/25/0.5
Cal 4	CAL-SPK1	1		9	10	2500/1250/100/50/1

Cal 5	CAL-SPK1	2.5		7.5	10	6250/3120/250/125/2.5
Cal 6	CAL-SPK1 (intermediate)	5		-	5	25000/12500/1000/500/10
CRDL	ZPACEMN-103	0.1		9.6	10	varied
	ZPACEMN-104	0.1				
	6020 Hg-SPK	0.2				0.2
ICS-A	ICS-ICPMS	0.25		9.75	10	25000/500
ICS-AB	ICS-ICPMS	0.25		9.56	10	27500/26200/1250/600/100/50/4
	6020-SPK	0.05				
	6020-SPK2	0.05				
	6020-SPK3	0.05				
	6020Hg-SPK	0.04				
ICV / CCV add Hg	XPACEMN-75	0.05		49.31	50	4/80/1000
	XPACEMN-76	0.02				
	Bi/Th Intermediate	0.4				
	XPACEMN-77	0.02				
	Hg Intermediate C	0.2				

[1]Alternate final volumes may be prepared at the discretion of the scientist, so long as the concentrations specified above are maintained.

Appendix D: Stock Standard Summary

Stock Standard Concentrations

	HP7379	HP7376	HP7375	XPACEMN 77	XPACEMN 76	XPACEMN 75	ZPACEMN 103	ZPACEMN 104	ICS-ICPMS	Agilent Tune	EPA Tune
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Aluminum	-		1000			1000	2		1,000		
Antimony		200		200				0.005			
Arsenic	200				200			0.05			
Barium	200				200		0.03				10
Beryllium	200				200		0.02				10
Bismuth							0.05				
Boron		200		200			1				
Cadmium	200				200		0.008				
Calcium			1000			1000	4		1,000		
Chromium	200				200		0.05				
Cobalt	200				200		0.05			10	10
Copper	200				200		0.1				
Iron			500			500	5		1,000		
Lead	200				200		0.01				
Lithium	200				200		0.05			10	10
Magnesium			1000			1000	1		1,000		10
Manganese	200				200		0.05				
Molybdenum		200		200				0.05	20		
Nickel	200				200		0.05				
Palladium		200		200				0.05			
Platinum		200		200				0.05			
Potassium			1000			1000	10		1,000		
Selenium	200				200			0.05			
Silicon			500			500		10			
Silver	100				100		0.05				
Sodium			1000			1000	5		1,000		
Strontium	200				200		0.05				
Thallium					100		0.01			10	10

Tin		200		200		20		0.05			
Titanium		200		200		20		0.1	20		
Vanadium	200				200		0.1				
Zinc	200				200		0.5				
Uranium	200						0.05				10
Indium											10
Cesium					200						10
Cerium										10	
Yttrium										10	10
Rhodium											10
Thorium							0.05				

Single Element Stock Standard Concentrations

	Bi-STK (Spex)	Bi-STK (Agilent)	6020-Th (Spex)	6020-Th (Agilent)	MERC-STK1	MERC-STK2	HG-LL Stock	6020-Ge	6020-Sc	6020-Tb	6020-In	6020-Ir
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Bismuth	1000											
Bismuth		1000										
Thorium			1000									
thorium				10000								
Mercury					1000							
Mercury						1000						
Mercury							10					
Germanium								1000				
Scandium									10000			
Terbium										1000		
Indium											1000	
Iridium												1000

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0054 **Revision:** 04

Document Title: Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1

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Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

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ENV-SOP-MIN4-0054 - Mercury

QM Approval

Name/Signature	Title	Date	Meaning/Reason
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Management Approval

Name/Signature	Title	Date	Meaning/Reason
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1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of mercury in mobility procedure extracts, aqueous wastes, ground waters, soils, sediments, bottom deposits, and sludge-type materials using cold vapor atomic absorption (CVAA).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The default reporting limit (RL) or Limit of Quantitation (LOQ) for mercury in liquid is 0.2 µg/L. The default reporting limit for mercury in soil is 0.02 mg/kg. Reporting limits may vary based on the nature of the individual sample matrix. For certain applications, a lower level method optimized for sensitivity in which the reporting limit is 0.010 µg/L is available. This is for aqueous samples only.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

2.0 SUMMARY OF METHOD

2.1 The method, a CVAA technique, is based on the absorption of radiation at the characteristic wavelength of 253.7 nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance is measured as a function of mercury concentration.

2.2 Chemical Reactions - Organic mercury compounds are decomposed by digestion with potassium permanganate in acid solution. The mercuric ions are then reduced to the elemental state with stannous chloride and mercury vapor is produced.

3.0 INTERFERENCES

3.1 Potassium permanganate is added during digestion of samples to break down organo-mercury compounds which would otherwise not respond to the cold vapor technique. A heating step is required for methyl mercuric chloride when present in or spiked to a natural system. Possible sulfide interferences are also eliminated by the addition of potassium permanganate. EPA studies indicate concentrations as high as 20 mg/L of sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, EPA studies indicate copper concentrations as high as 10 mg/L had no effect on recovery of mercury from reagent water.

3.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. The design of the dedicated mercury analyzer assures that this does not occur.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the

laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	30 mL	Acidified with nitric acid to pH<2, stored ambient	Must be analyzed within 28 days of collection.
Solid	8 oz glass jar	0.3 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either stored at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
Mercury analyzer, computer controlled	Cold Vapor Atomic Adsorption (CVAA), Cetac M-7600 or equivalent. Each instrument has an associated auto-sampler, Cetac ASX 520 or equivalent
Hot Block™ digester	54 place block or equivalent, Environmental Express SC154 or equivalent
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Peristaltic pump tubing	Fisher Scientific or equivalent
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Resin Pellets	Environmental Express SC400 or equivalent
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent
Sulfuric acid	Fisher Scientific P/N A510-P212 or equivalent
Potassium permanganate solution	Dissolve 100 g potassium permanganate in a minimum volume of reagent water and dilute to 2000 mL with reagent water. Store the reagent at room temperature in either a plastic or glass container. This solution expires 3 months from preparation date. Fisher Scientific brand reagents or equivalent.
Sodium chloride - Hydroxylamine hydrochloride solution	Dissolve 240 g sodium chloride and 240 g hydroxylamine hydrochloride in reagent water and dilute to 2000 mL with reagent water. Store the standard at room temperature in either a plastic or glass container. Solution expires 1 month from preparation date. Fisher Scientific brand reagents or equivalent.
Potassium persulfate solution (5%)	Dissolve 100 g of potassium persulfate in reagent grade water and dilute to 2000 mL. This solution expires 3 months from the preparation date. Fisher Scientific brand reagents or equivalent.
Rinse solution	Add 48 mL concentrated hydrochloric acid to 800 mL water, add 24 mL concentrated nitric acid and dilute to 1 L with reagent water. Store in 5L Nalgene container at room temperature. The solution expires 1 week from preparation date.
Stannous Chloride	Add 140 mL concentrated hydrochloric acid and 200 grams SNCl2-2H20 to 2000 mL reagent water. Different amounts may be made based on need. Store in bottle marked “Stannous Chloride” at the instrument. Fisher Scientific brand reagents or equivalent.
Aqua Regia	Mix 3 parts concentrated hydrochloric acid with 1 part concentrated nitric acid. Use fresh daily, expires within 24 hours.

8.2 Standards

Standard	Description
Mercury Calibration Stock Solution	1000 mg/mL, NIST traceable standard. Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
Intermediate Working Calibration Solution[1]	50 ug/L intermediate final concentration. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed. See appendix B for composition.
ICV/CCV Mercury Stock Solution	1 ug/mL, NIST traceable standard. Must be from a separate source than the mercury calibration stock source. Spex-Certiprep or equivalent.
Low Level Mercury Calibration Stock Solution	10 mg/L, NIST traceable standard. Store at room temperature. Expires as specified by manufacturer. Inorganic Ventures or equivalent.
Low Level ICV/CCV Mercury Stock Solution	10 mg/L, NIST traceable standard. Must be from a separate source than the mercury calibration stock source. Inorganic Ventures or equivalent.
Low Level Mercury Calibration Intermediate Standard[1]	1 ug/L final concentration. Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed.

	See appendix B for composition.

8.2.1 Mercury Calibration Intermediate Standard to be prepared every 6 months or as needed. The calibration standards are prepared using the same type of acid and reagents, at the same concentration range as the samples to be analyzed.

8.2.2 SW-846 series methods for mercury require that calibration standards are processed like samples including heating while EPA 245.1 specifically prohibits the calibration standards from being heated. Daily calibration records are documented in the electronic Prep Log.

9.0 PROCEDURE

9.1 Water

9.1.1 Sample Preparation

9.1.1.1 Prepare a method blank (MB) by transferring 30 mL of reagent grade water to a new 50 mL digestion cup. Label with the LIMS batch number and sample number.

9.1.1.2 Prepare a laboratory control sample (LCS) by transferring a 0.15 mL aliquot of the stock mercury standard to a 50 mL cup. For low level mercury samples, transfer 0.15 mL aliquot of the low level mercury intermediate standard. Bring the total volume to 30 mL with reagent water. Label with the LIMS batch number and sample number.

9.1.1.3 Shake sample to achieve homogeneity. Maximum sample volume is 30 mL. Use this or a smaller volume diluted to 30 mL. Place the sample into the 50 mL cup labeled with the corresponding LIMS sample number. Record sample volume in the Hg CVAA Sample Preparation Log.

9.1.1.4 Prepare an MS/MSD by transferring 0.15 mL aliquot of the stock mercury standard to 50 mL cups. For low level mercury samples, transfer 0.15 mL aliquot of the low level mercury intermediate standard. Bring the total volume of each to 30 mL with sample.

9.1.1.5 To all samples (including QC) add 1.5 mL concentrated sulfuric acid and 0.75 mL concentrated nitric acid, mixing well after each addition.

9.1.1.6 To all samples (including QC) add 5 mL potassium permanganate. If the purple color disappears, the sample is re-batched and re-prepped at a lower volume.

9.1.1.7 To all samples (including QC) add 2.5 mL of potassium persulfate solution and swirl to mix.

9.1.1.8 Loosely cap each cup and place into the digestion block, maintained at a temperature of 95°C ± 2°C and heat for two hours. Observe the initial temperature and time in the block.

9.1.1.9 After the two hour digestion, remove the samples from the block and cool. Observe the time the samples were removed from the block, as well as the final temperature of the block.

9.1.1.10 To all samples (including QC) add 1.8 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine

hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.1.2 Documentation – Digestion Records

Record the observations and necessary information in the electronic preplog using template version F-MN-I-342-Rev.02. Information includes batch and sample ID, initial and final times, temperatures, volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. The initial and final times and temperatures will be representative of the elapsed time for the batch.

9.2 Solid/Semi-Solid

9.2.1 Sample Preparation

9.2.1.1 Prepare a MB by weighing 0.3 g of resin pellets in a 50 mL cup.

9.2.1.2 Prepare a LCS by weighing 0.3 g of resin pellets in a 50 mL cup and spiking with a 0.15 mL aliquot of the ICV/CCV working mercury standard.

9.2.1.3 Weigh a representative 0.3-0.36 g portion of sample in a 50 mL cup.

9.2.1.4 Weigh two additional samples for matrix spike/matrix spike duplicate (MS/MSD) and spike carefully to get these samples as close to the weight of the unspiked sample used for QC, as possible. Spike both the MS and MSD with 0.15 mL of the mercury ICV/CCV working standard.

9.2.1.5 To all samples (including QC) add 3 mL DI water.

9.2.1.6 To all samples (including QC) add 3 mL aqua regia (see 10.1 above).

9.2.1.7 Place in hot block, maintained at 95°C ± 2°C and heat for 2 minutes. Record this time and temperature as the initial start time.

9.2.1.8 Remove from hot block and allow to cool.

9.2.1.9 Bring all samples (including QC) up to a volume of 30 mL with DI water.

9.2.1.10 To all samples (including QC) add 9 mL potassium permanganate. If the purple color disappears, re-prepare the sample, MB, and LCS with less DI and the corresponding amount of potassium permanganate added so that final volume does not exceed 30 mL. Additional permanganate is noted as a comment on the prep form.

9.2.1.11 Loosely cap each cup and return samples to hot block digester, maintained at a temperature of 95°C ± 2°C and heat for 30 minutes.

9.2.1.12 Remove the samples from the block and record the final time and the temperature. Allow the samples to cool.

9.2.1.13 To all samples (including QC) add 3.6 mL of hydroxylamine hydrochloride to reduce the excess permanganate. The permanganate is reduced when the purple color dissipates. If the purple color does not dissipate, add additional hydroxylamine hydrochloride until the color dissipates. Note this on the preparation log and adjust in LIMS. For example: if an additional mL is needed, then add 1 mL to the final volume.

9.2.2 Documentation – Digestion Records

Record the necessary information in the electronic preplog using template version F-MN-I-343-Rev.03. Information includes batch and sample ID, initial and final times, temperatures, volumes, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed. The initial and final times and temperatures will be representative of the elapsed time for the batch.

9.3 Equipment Preparation & Analysis

9.3.1 Turn on the computer and load the software. Turn on, or 'wake up' the instrument and allow the lamp to warm up for about 90 minutes from a cold shut down (lamp off, main power off and gas off) and 5 minutes from standby (lamp off, main power on and gas off). Check the following:

9.3.2 Prepare any necessary reagents and record the appropriate information (volumes, manufacturer, lot numbers, etc.) in the standard solution log.

9.3.3 Check instrument waste and empty as needed.

9.3.4 Perform any routine maintenance as needed and record in maintenance log.

9.3.5 Check the $KMnO_4$ trap at the back of the instrument to make sure it is filled with crystalline $KMnO_4$ and not wet or spent (the brown MnO_2 color approaches the open end of the trap).

9.3.6 Fill the rinse solution container with rinse solution, if needed, and move the probe down into the rinse well.

9.3.7 Check peristaltic pump tubing installation, make sure tension is adjusted if needed, and turn pump on.

9.3.8 Place the $SnCl_2$ line in DI water.

9.3.9 Initialize the wetting of the GLS by selecting 'wet the gas liquid separator post' option in the software. This increases the gas flow to 300-350 mL/min and ramps the pump speed to 100%. Pinch the waste line tubing shut with your fingers. Watch the bubbles and ensure that 1-2 bubbles completely propels to the top of the chamber, wetting the entire post and the top. As soon as this happens, open the waste line tubing so the GLS can drain.

9.3.10 Inspect the GLS to make sure it is draining completely and liquid is not pooling.

9.3.11 Attach the sample gas line to the nafion dryer cartridge.

9.3.12 Fill the stannous chloride bottle with stannous chloride.

9.3.13 Place the $SnCl_2$ line into the $SnCl_2$ solution bottle.

9.3.14 Create a worksheet for analysis by selecting 'new from' in the file menu. Enter the name, ie 20Aug15 (DDMMMYY), a, b, c etc. (if more than one run is performed that day) soil or water to indicate sample matrix, and instrument ID number. The program will then go to the Method Editor page.

9.3.14.1 In the conditions page in the Method Editor, check the instrument settings including the time profile (baseline correction and read time delays). To do this, read a standard and move the baseline correction window and read time window accordingly if needed.

9.3.14.2 Check the Standards page to ensure the correct calibration parameters and standards are entered.

9.3.14.3 Check the QC tests page to make sure the correct test solutions and parameters are entered if the software is to calculate recoveries during analysis.

9.3.15 Create a sequence in the sequence editor tab and enter sample IDs or import them from LimsLink.

9.3.16 Start analysis, monitor all initial QC checks. If initial QC fails, make adjustments if needed and re-calibrate. If checks pass criteria, continue with sample analysis.

9.3.17 After analysis, print out a report and transfer valid data into LIMS system via LimsLink.

9.3.18 After completing sample analysis for the day, shut down the instrument.

9.3.18.1 Place the $SnCl_2$ line in 10% HNO_3 and run for ~10 minutes. After this move the probe up out of the rinse well and place the $SnCl_2$ line in DI water and run for 2-5 minutes. Remove from DI and allow the line to run dry. Turn off pump, disconnect the clamps, and loosen pump tubing.

9.3.18.2 Disconnect the sample gas line from the nafion dryer cartridge.

9.3.18.3 Turn off the gas and the lamp.

9.3.18.4 If the instrument will be used in the next day or two, leave it in the stand-by mode. If not, do a cold shut down and turn off the software, instrument, auto sampler and auto diluter.

9.4 Routine Instrument Operating Conditions

Parameter	Setting
Sample Probe Depth (mm)	145
ASX Rinse Pump Speed (%)	50
Sample Uptake Time (s)	45
Rinse Time (s)	95
Gas Flow (mL/min)	100
Pump speed (%)	50
Read Delay time (s)	55.50
Replicate read time (s)	1.50
Replicates	4

9.5 Initial Calibration

9.5.1 Calibration Design

9.5.1.1 The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of four integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Instruments must be calibrated at a minimum of once every 24 hours or prior to use. The instrument standardization date and time must be included in the raw data.

9.5.1.2 Additional calibration specifications may be referenced in ENV-SOP-NW-0027 *Calibration Procedures*, or equivalent replacement.

9.5.2 Calibration Sequence

Calibration Blank (CAL0)
CAL1
CAL2
CAL3
CAL4
CAL5
ICV
ICB
CRDL
CCV
CCB
Client samples
CRDL
CCV
CCB

9.5.3 ICAL Evaluation

9.5.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.995.

9.5.3.2 Relative Standard Error (RSE)

%RSE is evaluated after all calibration points have been measured. In order for a standard curve to be acceptable, the %RSE acceptance criteria is 80%-120% must be observed.

Note: %RSE is analogous to %RSD. 40CFR Part 136 allow %RSE to be used in place of correlation coefficient (R) or coefficient of determination (r^2) for the acceptability determination of the curve.

9.5.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.5.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 The percent recovery in the LCS is calculated using Equation 1:

Equation 1

$$\%\,\mathrm{Recovery} = \frac{SR}{SA} \mathrm{x}100$$

Where, SR = LCS result (ug/L or mg/kg)
SA = spike added, ug/L or mg/kg

10.2 The percent recovery of mercury in the matrix spike and matrix spike duplicate is calculated using Equation 2:

Equation 2

$$\%\,\mathrm{Recovery} = \frac{(SSR - SR)}{SA} \mathrm{x}100$$

Where, SSR = Spiked sample result, mg/L or mg/kg
SR = Sample result, mg/L or mg/kg
SA = Spike added, mg/L or mg/kg

10.3 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 3:

Equation 3

$$\%RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where, S = Sample result, mg/L or mg/kg
D = Duplicate sample result, mg/L or mg/kg

10.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L
v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \mathrm{x}100$$

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 7470/7471. 1 per batch of 10 or fewer samples for 245.1
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	Performed at client request.
Post Digestion Spike	Performed at client request.
Filter Blank (FB)	1 per batch of 20 or fewer samples when applicable.

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run. May be run more frequently per state or client requirement.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-NW-0018 *Determination of LOD and LOQ* for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-NW-0025 *Training and Orientation Procedures* for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Use of Block Digestor- Heating is conducted with hot block digestion as the heating equivalent mentioned in SW 846 7471B (section 6.10) and SW 846 7470. This is also compliant with method 245.1 under the Clean Water Act method flexibility in 40CFR section 136.6 (b) (4) (iii).

14.2 The lab utilizes a 30 mL final volume, all solid weights and reagent ratios are conducted based on the 0.3 g versus the 0.5 g initial weight accordingly.

14.3 Mercury calibration standards are prepared and digested weekly for SW-846 analysis of soils and waters. The stability and performance of standards prepared weekly has been evaluated and documented.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – QC Summary

Appendix B – Working Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7470A, 1994.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471A, 1994.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7000a, Revision 1, July 1992.

Test Methods for Evaluating Water and Solid Waste, Physical/Chemical Methods, SW-846, Method 7471B, Revision 2, Feb 2011.

Methods for Chemical Analysis of Water and Wastes, Method 245.1. Rev.3.0, 1994.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

Minnesota Pollution Control Agency, Laboratory Quality Control and Data Policies, July 2011.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
Appendix A	Updated MB Acceptance Criteria and Corrective Action.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0054	Mercury in Liquid and Solid/Semi-Solid Waste by 7470A, 7471, 7471B, and 245.1	03

Appendix A: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
ICAL	Daily	r ≥ 0.995 RSE < 20%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	± 10% for SW-846 7000 series methods and ± 5% for 245.1	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR and West Virginia require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV[4]	At the beginning of each run. Depending on data quality objectives it may be required that a CRDL bracket samples.	± 30% (or specified by the client)	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve). If the CRDL is biased low, no data can be reported for the target elements failing criteria.	Qualify outages and explain in case narrative.
CCV[5]	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R):	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.

CCB	Daily, before sample analysis, after every 10, and at end of analytical window	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL. WIDNR and West Virginia require samples to be reported to the MDL.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Method Blank	One per 20 samples	Method 7470/7471: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs. Method 245.1: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1.	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD[1]	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	80-120% for 7470/7470A and 7471/7471B. 85-115% for 245.1 % RPD ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD[2,3]	One per 20 samples for 7470/7470A and 7471/7471B. One per 10 samples for 200.8	80-120% for 7470/7470A[3] and 7471/74/1B. 245.1: 70-130% %RPD: 20%	If the percent recovery for the MS and MSD fall outside the control limits, the results are flagged that they are outside acceptance criteria along with the parent sample. If the RPD exceeds the acceptance criteria,	Qualify analytes with MS out of criteria.

			the MSD sample and associated parent sample need to be flagged. If MS or MSD fails and spike amount is less than 4 times the native concentration in the sample, remove M1 flag and replace with P6 flag. If the RPD is outside the limit, report the data and footnote the samples with precision outliers. The footnote only applies to samples within the same batch containing the sample used for the MS and MSD analyses.	
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Post Digestion Spike	Per client request	Refer to project specific technical specifications.	Qualify outages	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Result must be less than the absolute value of the Reporting Limit (LOQ). NC requires blanks to be clean to ½ RL.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x FB detections, report the data.	Qualify outages and explain in case narrative.

[1]WIDNR requires the use of a lab created matrix solution from unused samples.

[2]In the event that only samples identified as Equipment Blanks and/or Field Blanks are available, and LCS/LCSD will be prepared in place of MS/MSD.

[3]In the absence of method specified recovery limits, results will be evaluated based on specifications outlined by the MPCA guidelines for Inorganic Analysis.

[4]A reporting limit verification is performed by analyzing a CRDL at ± 30% while the method has no low end criteria.

[5] ICV/CCV criteria is ± 10% while the 7000 series indicates ± 20%, the tighter criteria is applied to allow for instrumentation to be utilized for any mercury method throughout an analytical shift.

Appendix B: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Solvent	Solvent Volume (mL)	Final Total Volume (mL)	Final Concentration (µg/L)
Mercury Calibration Intermediate.	Mercury Stock (10 ug/mL)	5	Reagent water	985	1000	50
	Concentrated nitric acid	10				
Standard 0	Intermediate Standard (50 µg/L)	0	Reagent water	30	30	0
Standard 1		0.12		29.88		0.2
Standard 2		0.6		29.4		1.0
Standard 3		1.8		28.2		3.0
Standard 4		3.0		27		5.0
Standard 5		6.0		24		10
CRDL		0.12		29.88		0.2
ICV/CCV	Mercury Stock 1000 mg/mL	0.15	Reagent water	29.85	30	5.0
ICB/CCB	N/A	N/A	Reagent water	30	30	0
Low Level Mercury Calibration Intermediate Standard; Prepare every 6 months.	Calibration Mercury Stock (10 mg/L)	0.100	Reagent water	984.9	1000	1.0
	Concentrated nitric acid	5.0				
	Concentrated hydrochloric acid	10				
Standard 0	Intermediate Standard (1.0 µg/L)	0	Reagent Water	30	30	0
Standard 1		0.30		29.7		0.010
Standard 2		0.75		29.25		0.025
Standard 3		1.5		28.5		0.050
Standard 4		3.0		27		0.100
Standard 5		6.0		24		0.200
CRDL		0.30		29.7		0.01
Low Level Mercury ICV/CCV **Intermediate** Standard. Prepare every 6 months	ICV/CCV Mercury Stock (10 mg/L)	0.4	Reagent water	184.6	200	20
	Concentrated nitric acid	5.0				
	Concentrated hydrochloric acid	10				
Low Level Mercury ICV/CCV	Low Level Mercury ICV/CCV Intermediate (75 µg/L)	0.15	Reagent water	29.85	30	0.10
Lower Level Mercury ICB/CCB	N/A	N/A	Reagent water	30	30	0

ATTACHMENT B-4

RMAP XRF SOPs

Attachment B-4
XRF SOPs
Index

	SOP Number	SOP Title	# Pages
1	RMAP-SOP-XRF-001, Rev. 0	Analysis of Lead by Field Based Portable XRF Spectroscopy by Niton XLp 300 Series XRF in Paint Samples	11
2	RMAP-SOP-XRF-002, Rev. 0	Analysis of Lead, Arsenic, and Mercury by Field Based Portable XRF Spectroscopy by Niton XL2 Series XRF	11

Attachment B-4

Analysis of Lead by Field Based Portable X-Ray Fluorescence Spectroscopy by Niton XLp 300 Series XRF in Paint Samples

Signatures and Approvals

Approved: Date:

Eric Hassler, Director

Department of Reclamation and Environmental Services

Butte-Silver Bow County

Approved: Date:

Mike Mc Anulty, Liability Manager

Atlantic Richfield Company

The RMAP SOP XRF-001, Rev. 0 is effective on the date of approval.

1.0 OBJECTIVES AND APPLICATION

This standard operating procedure (SOP) describes the field procedure for the determination of total lead in paint by Portable X-Ray Fluorescence (XRF) Spectroscopy using a Niton XLp 300 Series XRF. This SOP is applicable to field analysis of paint samples performed under The Butte-Silver Bow (BSB) County *Revised Final Multi-Pathway Residential Metals Abatement Program Plan* (RMAP) (BSB & Atlantic Richfield Company, 2022) (hereafter referred to as the Program) designed to mitigate exposure to lead paint for residents of the Butte Priority Soils Operable Unit (BPSOU), the larger Butte community as a whole, and rural residential development within the Silver Bow Creek/Butte Area Superfund Site.

1.1 Target Analyte List and Limits of Quantitation

The target analyte for this SOP is lead in paint with a limit of quantitation (LOQ) set at the HUD Action Level of 1 mg/cm^2. The LOQ is the value to which analytes are reported as detected or not detected in the final report. When the sample result is greater than the LOQ the results are considered quantitative based on the K&L Mode specified in this SOP. The LOQ is verified daily by running an XRF Calibration Check at 1.04 mg/cm2 initially, after every ten samples, and at the end of the analysis and evaluating against an acceptance criterium of ± 20%.

1.2 Applicable Matrices

This SOP is applicable to paint samples collected as part of the Program. LBP sample analysis should include performing XRF analyses of paint at multiple locations to ensure each painted surface is represented. The specific number and location of XRF measurements should be performed in accordance with the requirements specified in Chapter 7 of the U.S. Department of Housing and Urban Development (HUD) Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing (HUD, 2012). Paint assessment will begin with a visual inspection of the building following HUD Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing (HUD, 2012) to determine if there are potential LBP hazards (see Table 1). Details of LBP sampling locations are detailed in individual work plans and are beyond the scope of this SOP.

2.0 SUMMARY OF METHOD

The field sampler(s) must follow the manufacturer's procedures for operating the Niton XLp 300 Series XRF instrument. Paint assessments will begin with a visual inspection of the building following the United States (US) Housing and Urban Development (HUD) guidelines to determine if there are potential lead-based paint (LBP) hazards. Interior and Exterior components of the building, including outbuildings, will be tested with the portable XRF to determine the presence of LBP.

3.0 INTERFERENCES

The Niton XLp 300 does not require any substrate correction factors. Care must be taken to not contaminate or tear the window material on the XRF. Calibration checks must be performed in a manner that does not falsely influence the calibration material.

4.0 ACRONYMS AND DEFINITIONS

DQO: Data Quality Objective

FSP: Field Sampling Plan

HUD: Housing and Urban Development; Federal agency

K&L Mode: A quantitative method for the analysis of paint which allows for determination of results with a 95% Confidence Level.

LBP: Lead-based Paint

LOQ: Limit of Quantitation

PCS: Performance Characteristic Sheet; HUD related terminology describing acceptable operating specifications for a specific XRF make/model. Such documentation is required when using an XRF for LBP analysis in a residential or child occupied facility setting. A copy of the Niton XLp 300 Series XRF PCS is included in Exhibit C.

PPE: Personal Protective Equipment

RMAP: The Butte-Silver Bow (BSB) County Revised Final Multi-Pathway Residential Metals Abatement Program Plan (RMAP) (BSB & Atlantic Richfield Company, 2021)

SOP: Standard Operating Procedure

SRM: Standard Reference Material

XRF: X-Ray Fluorescence; as used in this SOP, XRF indicates units that utilize an electrically powered x-ray tube that is only capable of emitting ionizing radiation when the instrument is powered on.

5. KEY RESPONSIBILITIES

The key project responsibilities shall be clearly defined in the project-specific work plan and QAPP for all RMAP activities by a certified (licensed) lead-based paint inspector.

6.0 HEALTH AND SAFETY

6.1 Personal Protective Gear

The field sampler will wear Level D PPE (hard, hat, high visibility vest, long sleeve shirts, hard toe boots, safety glasses and nitrile gloves (changed between sampling locations). Additional safety measures will be detailed in individual FSPs as required**. Does this PPE match interior XRF sampling by BSB and AR.**

6.1 Radiation Source Type

The Niton XLp 300 contains a ^{109}Cd radioactive isotope source. This source is secured in a solid tungsten alloy source holder. The ^{109}Cd source constantly emits ionizing radiation that is blocked by the source holder, except when the instrument shutter is opened for analysis

5.2 Safety

Radiation must be treated with respect. All users will take care while using the XRF so that no one, including the operator, is exposed to radiation. To minimize your exposure, never put your hand or any

other body part on or near the sample window of the XRF, especially when the shutter is open for analysis. Never point the XRF at yourself or anyone else.

The XRF must be used in accordance with the manufacturer's instructions as detailed in Chapter 1 of the user's manual (Exhibit A). All reasonable measures, including the concepts of time, distance, and shielding should be implemented to limit radiation exposure to as low a level as reasonably achievable. This includes minimizing time around the instrument when it is energized, maximizing the distance from the instrument window, and shooting into high density materials whenever possible. As a precautionary measure, only the XRF operator should be near the XRF while it is actively collecting data; all other people should maintain a 10-foot distance during operation. Open the shutter only to analyze a sample. The shutter can only be opened after the user has logged on to the analyzer using a password. The three warning lights on the XRF will go on when one of the shutters is open and will stay on as long as one of the shutters remains open.

Operators will visually inspect the XRF for damage prior to use. If there is damage, the instrument will not be used. If there are any problems with how the XRF is working, do not attempt to repair the XRF yourself. Opening the instrument may expose the user to the radiation and will void the warranty. Any damage or operational issues must be reported to the Project Manager (PM) who will coordinate repair with the manufacturer.

The XRF is not waterproof and should not be used in heavy rain. If necessary, the instrument may be used in light rain, inside a large zipper locking bag to limit exposure for exterior sample analysis.

The XRF should be stored and transported without the batteries installed. If the instrument is left in a vehicle unattended for any period of time, the vehicle must be locked. The instrument must not be left in a vehicle overnight. When not in use, the instrument must be stored in the locked equipment room at the consultant or agency location. The instrument must be signed out before use and signed in after use.

Additional Details on Radiation Safety are contained in Chapter 1 of the user's manual (Exhibit A).

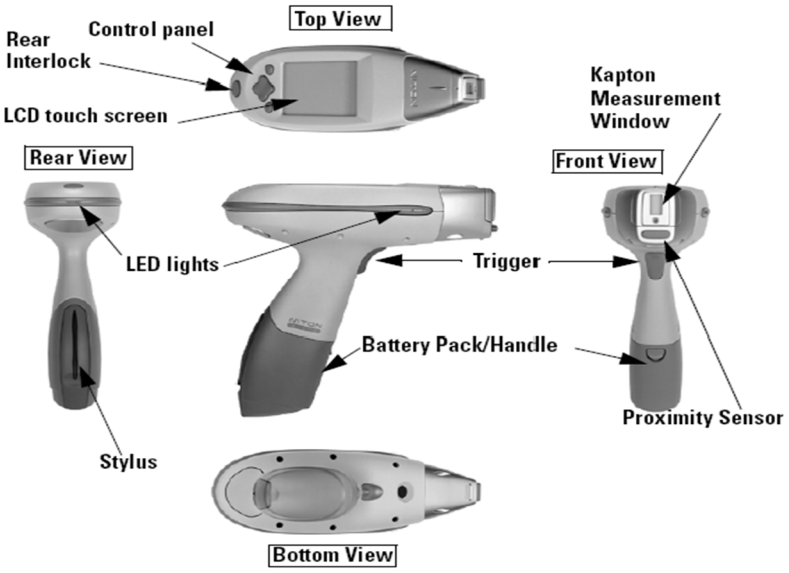
Leak Tests (According to the Niton Manual leak tests are required every six months) is this performed by BSB or other consultants?

5.3 Radiation Monitoring

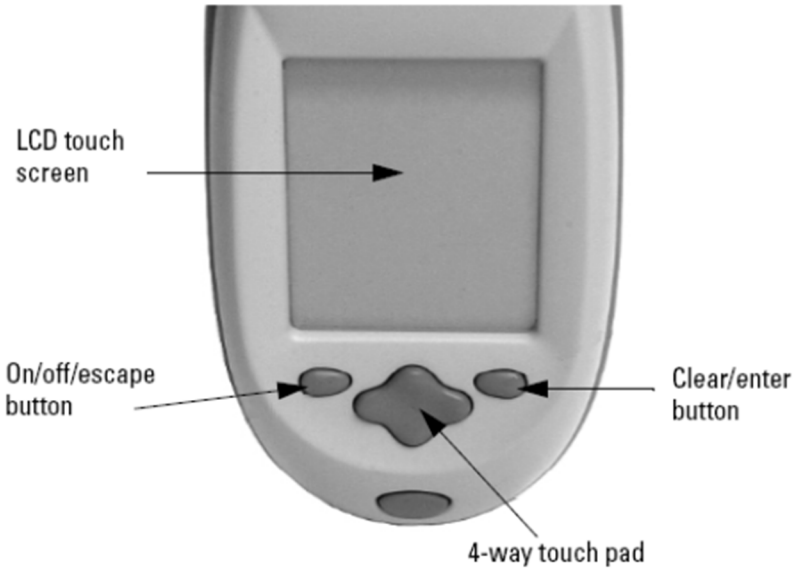
Inspectors should wear radiation dosimeters to measure their exposure, although excessive exposures are highly unlikely if the instruments are used in accordance with the manufacturer's instructions. If feasible, persons should not be near the other side of a wall, floor, ceiling, or other building component surface being tested. Does BSB or AR require individual users to have radiation badges?

6.0 GENERAL PROCEDURE FOR OPERATING THE XRF

Refer to the Niton XLp 300 Series XRF User's Manual (Exhibit A) for additional instructions as necessary. As an overview the XRF has the following instrument layout.



The control panel is located on the top of the XRF, below the LCD touch screen, to select menus for analysis.



6.1 Starting the XRF

To turn on the XRF, depress the on/off/escape button on the control panel for approximately 3 seconds until you hear a beep. The XRF will then show a restart screen and will automatically count down from 9 to 0 in seconds. When the restart is complete the Restart Screen will be replaced by a Login Screen. Tap anywhere on the screen to continue.

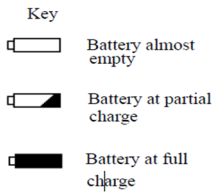
A Warning Screen will appear that requires the user to acknowledge a radiation hazard when flashing. Press “Yes” to continue.

A virtual numeric keypad will appear, enter the temporary passcode (1234E) or the user defined password. The main menu will then appear. Check the date and time. The date and time must be accurate for data traceability for XRF measurements. If the date and time are incorrect, see Set Time and Date on pages 2-9 of the user's manual (Exhibit A).

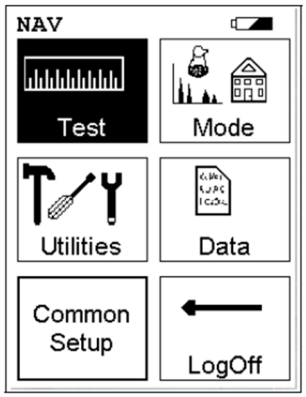
6.2 Setting the Instrument for use and Calibration

Touch the screen and the NAV Menu will appear. Select the word **NAV** and a drop-down list of options will appear. Select the **Language** option then select **English** and **Close**.

The Battery Life Indicator is visible at the top right of each screen to monitor battery life during instrument operation. Keep one battery in the charger while using the other to avoid work slowdown or stoppages.



All XRF functions are accessible from the Main Menu shown below showing multiple icons and are accessed by pushing the icon on the screen.



Select the Mode icon to access the different analysis modes. Select Pb Paint Mode to detect and quantify the amount lead present in painted surfaces. Select the K&L Mode on the Pb Paint Mode Menu. The action level should be set to 1.0 at the bottom of the screen. If the action level is not set correctly, see page 5 of the user's manual (Exhibit A) to enter or reset the level. Push Return to go to the Main Menu.

6.3 Calibration

Select the Utilities icon on the Main menu. The Utilities Menu enables the user to choose multiple analysis and instrument settings on the XRF.

Allow the XRF to warm up for 10 minutes prior to instrument calibration. The XRF should be auto calibrated each time the unit is turned on, daily prior to use or if the unit is turned off during the day or the

battery replaced. If the instrument is to be turned off during the course of an inspection, calibration checks should always be done before the instrument is turned off (calibration checks do not need to be done each time an instrument enters an automatic “sleep” state while still powered on).

Select the Calibrate icon to access the Calibrate Menu. Select the Calibrate Detector Icon to begin a standard calibration of the XRF detector. The instrument will calibrate using an internal program and no external standard calibration is performed. Avoid vibration, loud noise, strong electronic fields, or other possible interferences during calibration. The XRF calibration screen will be displayed until the calibration is complete. Record the calibration results for traceability. No substrate correction factors are applied for brick, concrete, drywall, metal, plaster or wood substrates. The NomSec, CPS, and Perc values should be recorded. Press the Return icon or escape button to return to the main menu.

7.0 LEAD PAINT ANALYSIS AND PROCEDURE

Select the Test icon from the Main Menu. The XRF will operate in the testing mode currently selected (K&L mode). Hold the XRF window against the surface to be analyzed, making sure the sample window is as flat as possible against the surface, and depress the XRF trigger to take a reading. A 20 second measurement time is recommended.

7.1 Calibration Check and Quality Control Samples

Before analyzing primary samples, quality control (QC) samples must be analyzed to confirm the XRF is functioning within specifications.

Analyze the XRF Calibration Check Unit Lead Paint Standard Reference Material (SRM 2573) for a 20 second measurement time and record the data. Take and record three measurements for the initial calibration check and calculate the average rounding to the same number of significant figures reported by the XRF display If the average is within the acceptance limits, proceed with the analysis of the blank.

If the results are outside acceptance limits, perform a new initial calibration, reanalyze SRM 2573 and reanalyze the samples analyzed after the last passing calibration, reanalyze the SRM 2573 three times. If the recalibration is within the acceptance limits, record the original and reanalysis data and proceed with the blank analysis. If outside of the acceptance limits, contact the PM.

Calibration checks should be taken through the SRM paint film with the film positioned at least 1 foot (0.3 meters) away from any potential source of lead. The NIST SRM film should not be placed on a toolbox, suitcase, or surface coated with paint, shellac, or any other coating to take calibration check readings. Rather, the NIST SRM film should be attached to a solid (not plywood) wooden board or another non-metal rigid substrate such as drywall or attached directly to the XRF probe. The SRM should be positioned so that readings of it are taken when it is more than 1 foot (0.3 meters) away from a potential source of error. For example, the NIST SRM film can be placed on top of a 1 foot (0.3 meter) thick piece of Styrofoam or other lead-free material, as recommended by the manufacturer before taking readings. Similarly, readings between a successful calibration check and a subsequent unsuccessful calibration check must be discarded and the samples retested

Warning! During sample measurement three blinking lights will appear and continue until the trigger is released and the measurement is completed. If the LED lights continue to blink at any other time, discontinue use, remove the battery pack, store in the shielded holster, and contact the project PM for follow-up by the Niton Service Center.

Analyze the Blank Standard Reference Material (SRM 2570) for a 20 second measurement time and record the data. If the result is within the acceptance limit, proceed with the sample analysis. If the results are outside acceptance limits, wipe the XRF window clean and reanalyze the SRM 2570 for a 20 second measurement time. If within the acceptance limits, record data and proceed with the sample analysis. If outside of the acceptance limits, contact the PM.

Analyze a replicate sample once for every twenty samples. Select a location with positive lead results (if available) and reanalyze the same spot for a 20 second measurement time. The relative percent difference (RPD) between the averaged results for the sample and the replicate should be < 35%

The following table provides the acceptance limits for the QC samples.

Additional SRM samples are provided in Exhibit B of the SOP for use if necessary to check the accuracy of the instrument at different concentrations.

QC Samples for Lead	Frequency	Standard Value	Acceptance Limits
SRM 2573 (Calibration check)	Initially, prior to sample analysis, after every 10 samples, and at the end of the sequence. A total of three readings are averaged and compared to the standard value	1.040 ± 0.064 mg/cm^2	0.8 to 1.2 mg/cm^2
SRM 2570 (Blank)	Initially, prior to sample analysis, after every 10 samples, and at the end of the sequence	< 0.001	< ½ LOQ. Not detected is the goal.
Replicate Analysis	5% of samples, 1 per every 20 samples	-	35% RPD.

Additional SRM samples are provided in Exhibit B for use, if necessary, to check the accuracy of the instrument at different concentrations.

7.2 Sample Analysis

If the calibration check and blank analysis are acceptable the instrument is now ready for sample analysis. Each sample location will be analyzed a single time for 20 seconds and the result recorded.

Repeat the analysis of a single calibration check and blank analysis after every ten samples.

After each sample location is tested, gently wipe any dust or debris from the XRF Kapton window using a cotton swab. If the Kapton window becomes frayed, ripped, punctured, or contaminated with paint, replace it with a new window in accordance with Chapter 9 of the user's manual (Appendix A). Recalibrate the instrument and perform a calibration check and blank prior to sample analysis

After the final sample analysis at the residence, finish the sequence with a calibration check and blank analysis.

XRF Results are classified as positive if they are greater than or equal to the threshold of 1.0 mg/cm^2 and negative if they are less than the threshold. Record the XRF reading to the same number of significant figures displayed by the XRF.

8.0 Evaluating the Quality of XRF Testing (PCS specification)

1. Randomly select ten testing combinations for retesting from each house or from two randomly selected units in multifamily housing. Use the K+L variable time mode readings.
2. Conduct XRF retesting at the ten testing combinations selected for retesting.
3. Determine if the XRF testing in the units or house passed or failed the test by applying the steps below.
4. Compute the Retest Tolerance Limit by the following steps:
5. Determine XRF results for the original and retest XRF readings. Do not correct the original or retest results for substrate bias. In single-family housing a result is defined as the average of three readings. In multifamily housing, a result is a single reading. Therefore, there will be ten original and ten retest XRF results for each house or for the two selected units.
6. Calculate the average of the original XRF result and retest XRF result for each testing combination.
    - Square the average for each testing combination.
    - Add the ten squared averages together. Call this quantity C.
    - Multiply the number C by 0.0072. Call this quantity D.
    - Add the number 0.032 to D. Call this quantity E.
    - Take the square root of E. Call this quantity F.
    - Multiply F by 1.645. The result is the Retest Tolerance Limit.
    - Compute the average of all ten original XRF results.
    - Compute the average of all ten re-test XRF results.
    - Find the absolute difference of the two averages.
    - If the difference is less than the Retest Tolerance Limit, the inspection has passed the retest. If the difference of the overall averages equals or exceeds the Retest Tolerance Limit, this procedure should be repeated with ten new testing combinations. If the difference of the overall averages is equal to or greater than the Retest Tolerance Limit a second time, then the inspection should be considered deficient.

Use of this procedure is estimated to produce a spurious result approximately 1% of the time. That is, results of this procedure will call for further examination when no examination is warranted in approximately 1 out of 100 dwelling units tested.

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9.0 DATA COLLECTION

Sample identifications, data storage, and archiving may be different for each data user. Data Storage procedures are contained in Chapter 4 and 8 of the user's manual (Exhibit A).

For quality assurance, and to protect against data loss or sample ID confusion, Field Samplers must maintain hand-written or computer-based sampling forms or notes of all relevant results from each sample, even if the XRF is equipped with a data logging function. This step is important in the event data collection software malfunctions or files are corrupted or deleted.

10.0 ROUTINE MAINTENANCE GUIDELINES

Detailed maintenance procedures are provided in Chapter 9 of the user's manual (Exhibit A).

11.0 Waste Disposal

No waste from sample analysis is generated. PPE will be properly disposed by the sampling team in accordance with company guidelines.

11.0 EXHIBITS

Exhibit A – Niton XLp 300 Series Analyzer User's Guide, Version 5.2.1 P/N 500-926

Exhibit B – ThermoFisher Scientific Lead Paint Standard Summary, Surface Lead mg/cm^2

Exhibit C – Niton XLp 300 Series XRF Performance Characteristic Sheet

12.0 REFERENCES

The Butte-Silver Bow (BSB) County Revised Final Multi-Pathway Residential Metals Abatement Program Plan (RMAP) (BSB & Atlantic Richfield Company, 2021)

Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP), (Residential Parcels) October 2022.

U.S. Department of Housing and Urban Development, Guidelines for the Evaluation and Control of Lead-Based Paint Hazards in Housing, Chapter 7, Lead-Based Paint Inspection, 1997-2012 Revision

13.0 REVISION HISTORY

Section Number	Description of Change
Revision 0	**Initial Draft SOP**

TABLE 1: RMAP LEAD BASED PAINT DEFINITIONS[1]

	Total area of deteriorated paint on each component		
Type of building component	Intact*	Fair*	Poor*
Exterior components with large surface areas	Entire surface is intact	Less than or equal to 10 square feet	More than 10 square feet
Interior components with large surface areas (walls, ceilings, floors, doors)	Entire surface is intact	Less than or equal to 2 square feet	More than 2 square feet
Interior and exterior components with small surface areas (windowsills, baseboards, soffits, trim, etc.)	Entire surface is intact	Less than or equal to 10 percent of the total surface area of the component	More than 10 percent of the total surface area of the component

[1] – Definitions are taken from 2012 HUD guidelines.

*Intact surfaces require only monitoring by the property owner and are not considered lead-based paint hazards.

*Surfaces in fair condition may be repaired and/or monitored but are not considered to be lead-based paint hazards.

*Surfaces in poor condition are considered to be lead-based paint hazards and should be addressed through remediation or interim controls.

Attachment B-4

Analysis of Lead, Arsenic and Mercury in Dust Samples by Field Based Portable X-Ray Fluorescence Spectroscopy by Niton XL2 Series XRF

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the field procedure for the determination of total lead, arsenic and mercury by Portable X-Ray Fluorescence (XRF) Spectroscopy using a Niton XL2 Series XRF. This SOP is applicable to field analysis of dust samples performed under The Butte-Silver Bow (BSB) County *Revised Final Multi-Pathway Residential Metals Abatement Program Plan* (RMAP) (BSB & Atlantic Richfield Company, 2021) (hereafter referred to as the Program) designed to mitigate exposure to sources of arsenic, lead, and mercury contamination for residents of the Butte Priority Soils Operable Unit (BPSOU), the larger Butte community as a whole, and rural residential development within the Silver Bow Creek/Butte Area Superfund Site.

1.1 Target Analyte List and Limits of Quantitation

The target analytes for this SOP are lead, arsenic and mercury in dust with a limit of detection (LOD) set at 10mg/Kg based on the analysis of the SiO2 blanks.

1.2 Applicable Matrices

This SOP is applicable to dust samples collected as part of the Program.

The LOD is the value to which analytes are reported as detected or not detected in the final report. The stability and calibration of the XRF is verified daily by running an XRF calibration checks prior to sample analysis, after every ten samples, and at the end of the analysis and evaluating against an acceptance criterium of ±20%.

2.0 SUMMARY OF METHOD

The field sampler(s) must follow the manufacturer's procedures for operating the Niton XL2 Series XRF instrument.

3.0 INTERFERENCES

See Exhibit B, US EPA SOP 6200, Section 4.0, for a detailed discussion of interferences that may affect XRF readings. These include matrix interference, moisture content, sample positioning, chemical matrix effects, and spectrum overlap associated with certain elements. A small sample would be any sample that is smaller than the measurement window. Small samples present a unique risk because they don't block the entire beam path. The difficulty with placing small samples down on a work surface to analyze them is that you may get readings from the work surface that interfere with analytical results. A test stand is an effective way of analyzing small samples accurately and safely.

Never hold samples during analysis or look into the path of the primary beam. X-rays are attenuated more by denser and higher atomic mass materials, and less through lighter materials such as dust. This causes higher dose rates in the scattered radiation. If you are frequently handling low density samples, you should consider the use of test stands, backscatter shields, or the equivalent.

4.0 ACRONYMS AND DEFINITIONS

DQO: Data Quality Objective

K&L Mode: A quantitative method for the analysis of dust which allows for determination of results with a 95% Confidence Level.

LOQ: Limit of Quantitation

RMAP: The Butte-Silver Bow (BSB) County Revised Final Multi-Pathway Residential Metals Abatement Program Plan (RMAP) (BSB & Atlantic Richfield Company, 2021)

SOP: Standard Operating Procedure

SRM: Standard Reference Material

XRF: X-Ray Fluorescence; as used in this SOP, XRF indicates units that utilize an electrically powered x-ray tube that is only capable of emitting ionizing radiation when the instrument is powered on.

5.0 HEALTH AND SAFETY

5.1 Radiation Source Type

The Niton Model XL2 analyzer contains an x-ray tube which emits radiation only when the user turns the x-ray tube on. When the x-ray tube is on and the shutter is open, as during a measurement, the analyzer emits a directed radiation beam. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding. Additional details regarding radiation are presented in Chapter 2 of the User's Manual

5.2 Safety

Radiation must be treated with respect. All users will take care while using the XRF so that no one, including the operator, is exposed to radiation. To minimize your exposure, never put your hand or any other body part in front of the XRF analyzer, especially when the shutter is open for analysis. Never point the XRF at yourself or anyone else.

The XRF must be used in accordance with the manufacturer's instructions as detailed in Chapter 1 of the user's manual (Exhibit A). All reasonable measures, including the concepts of time, distance, and shielding should be implemented to limit radiation exposure to as low a level as reasonably achievable. This includes minimizing time around the instrument when it is energized, maximizing the distance from the instrument window, and shooting into high density materials whenever possible. As a precautionary measure, only the XRF operator should be near the XRF while it is actively collecting data; all other people should maintain a 10-foot distance during operation. Open the shutter only to analyze a sample. The shutter can only be opened after the user has logged on to the analyzer using a password. The shutter warning lights on the XRF will go on when one of the shutters is open and will stay on as long as the shutter remains open.

Operators will visually inspect the XRF for damage prior to use. If there is damage, the instrument will not be used. If there are any problems with how the XRF is working, do not attempt to repair the XRF yourself. Opening the instrument may expose the user to the radiation and will void the warranty. Any damage or operational issues must be reported to the Project Manager (PM) who will coordinate repair with the manufacturer. There is always a safe way to handle samples whether they are small, irregularly shaped, or of low density. Never look into the path of the primary beam

The XRF is not waterproof and should not be used in heavy rain. If necessary, the instrument may be used in light rain, inside a large zipper locking bag to limit exposure.

The XRF should be stored and transported without the batteries installed. If the instrument is left in a vehicle unattended for any period of time, the vehicle must be locked. The instrument must not be left in a vehicle overnight. When not in use, the instrument must be stored in the locked equipment room at the consultant or agency location. The instrument must be signed out before use and signed in after use.

Additional Details on Radiation Safety are contained in Chapter 2 of the user's manual (Exhibit A).

Also, consider the use of protective accessories such as a shielded test stand or backscatter shield (or equivalent - analyzer case) when performing routine and/or frequent analysis of any of the following:

- light materials (such as plastic, wood, dust or similarly low density/low atomic mass samples)
- samples that are smaller than the analysis window.

5.3 Radiation Monitoring

The Niton XL2 analyzer is designed to be safe to operate provided that it is used in accordance with manufacturer's instructions. Under conditions of normal use, monitored operators seldom receive a measurable dose and have not been known to receive in excess of 10% of the annual occupational dose limits (a criteria that would require monitoring under regulation in the U.S.)

6.0 GENERAL PROCEDURE FOR OPERATING THE XRF

Refer to the Niton XL2 Series XRF User's Manual (Exhibit A) for additional instructions as necessary. As an overview the XRF has the following instrument layout.

The primary beam is a directed beam out of the front of the analyzer that can have high dose rates. The secondary beam, or scattered beam, has much lower dose rates.

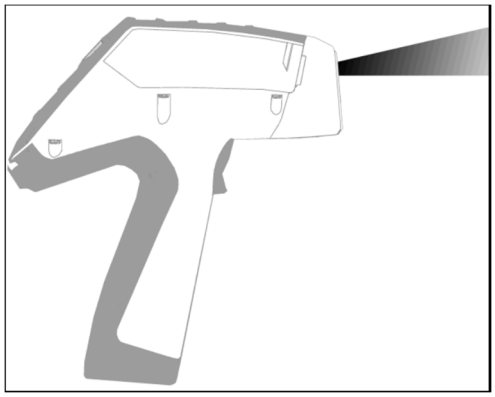


Figure 1. Primary Beam

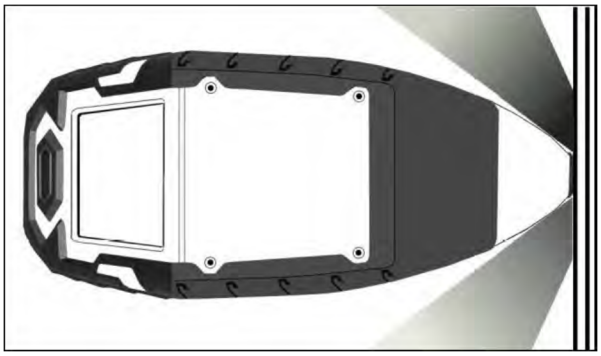


Figure 2. Secondary (Scattered) Beam

When the lights are flashing, the primary beam is on, and radiation is being emitted from the front of the analyzer.

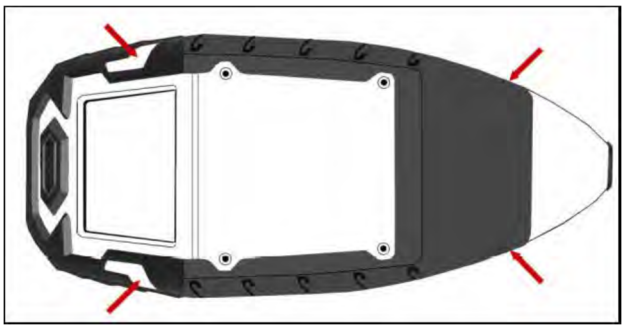


Figure 3. The X-ray Beam Indicator Lights

As mentioned, many times in this chapter, never place any part of your body in the path of the x-ray beam.

Small Samples

A small sample would be any sample that is smaller than the measurement window. Small samples present a unique risk because they don't block the entire beam path. The difficulty with placing small samples down on a work surface to analyze them is that you may get readings from the work surface that interfere with analytical results. A test stand is an effective way of analyzing small samples accurately and safely. Never hold samples during analysis or look into the path of the primary beam.

6.1 Starting the XRF

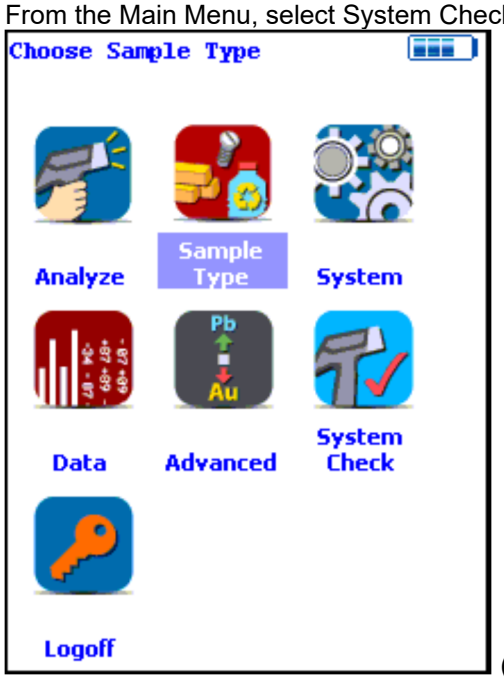
Attach a charge battery to the analyzer and turn it on. Allow the XRF to warm up for 5 minutes prior to instrument calibration to allow the instrument electronics to stabilize. The XRF should be auto calibrated each time the unit is turned on, daily prior to use or if the unit is turned off during the day.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon. Detailed information on this procedure is available in Setting the Date and Time of the User's Manual.

In a fixed-base environmental (office, trailer etc.), connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. (Consult “Using Your Analyzer With Your PC” on page 109 of the User’s Manual for details, if necessary.)

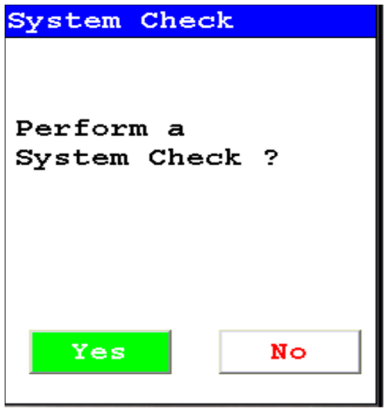
During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.

From the Main Menu, select System Check icon then the Yes button.



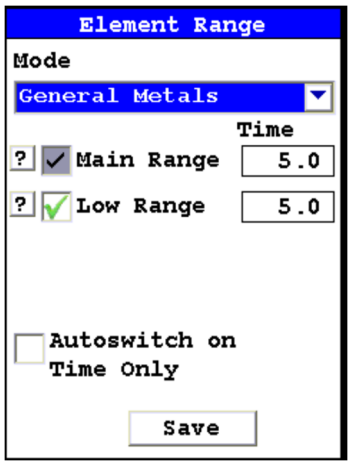
(Figure 1.)

System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either “System OK” or one of the failure errors.



If the unit shows a failure error, then perform a second System Check by clicking Recheck. If the unit still does not show a “System OK,” discontinue XRF analysis and contact the Project Manager for further instructions.

Thermo Scientific Niton XL2 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. Select the "Main Range" filter provides optimum sensitivity for lead.



Select the Sample Type icon to access the different analysis modes.

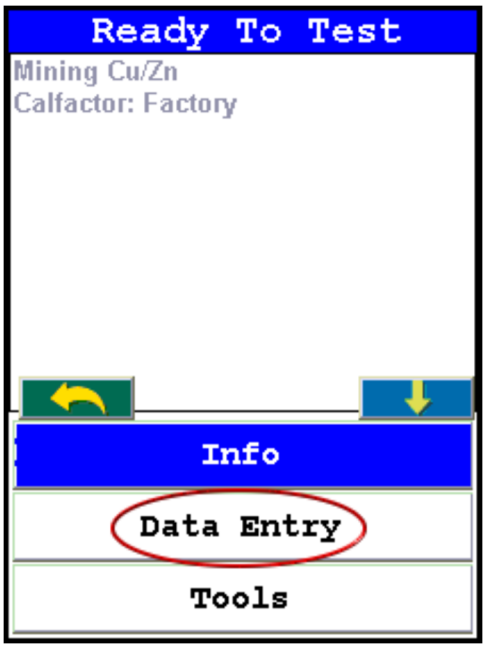
Sample Analysis

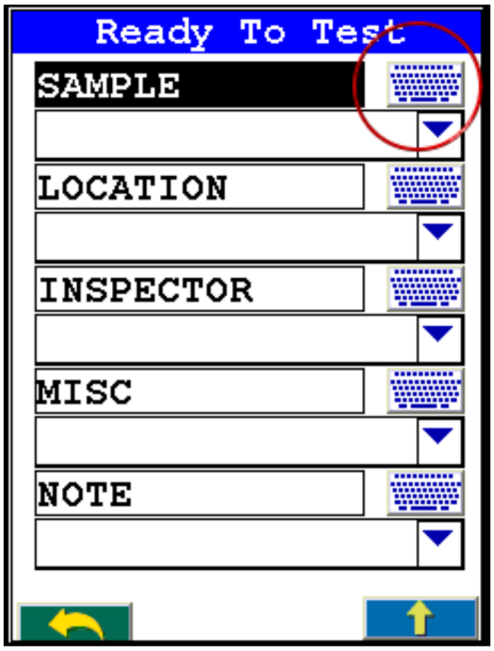
Select the **General Metals Mode** Icon to detect and quantify the amount lead present in dust.

Select the Analyze Mode Icon


Analyze
Sample Type
System
Data
Pb
Au
Advanced
System Check
Logoff

Select the Data Entry Icon to enter sample collection data.





Enter the data on the sample using the Virtual Keyboard.

ANALYSIS OF BAGGED DUST SAMPLES

1. Select the Mode icon. Select General Metals from the Mode Menu.

2. Select the Analyze icon.

3. Select the Data Button if you wish to do any data entry. Enter the data on the sample using the Virtual Keyboard.

4. Analyze the following QC samples prior to the analysis of site samples.

We need to check if these are still within expiration date and if BSB has these. Can mercury be detected at this level? Does BSB have set LOQs?

Table 1: Quality Control Limits

QC Sample for Lead	**Standard Value**	**Acceptance Limits (± 20%)**
SRM 2709 (LOQ SRM)	18.9 mg/Kg (ppm)	15.1 – 22.7 mg/kg (ppm)
SRM 2711 (Calibration check near Regulatory Limit)	1162 mg/Kg (ppm)	929.6 to 1394.4 mg/kg (ppm)
QC Sample for Arsenic	**Standard Value**	**Acceptance Limits (± 20%)**
SRM 2710 (Calibration check)	626 mg/Kg (ppm)	500.8 – 751.2 mg/kg (ppm)
QC Sample for Mercury	**Standard Value**	**Acceptance Limits (± 20%)**
SRM 2710 (Calibration check)	32.6 mg/Kg (ppm)	26.1 - 39.1 mg/kg (ppm)
QC Blank Sample	Standard Value	Acceptance Limits
Silicon dioxide blank	< Non-detected	< 10 mg/Kg. Not detected is the goal.

5. If the calibration checks and blank analysis are acceptable the instrument is now ready for sample analysis.

6. Place the dust sample collected in a clean whirl pack or zipper locking style bag and clean the sample bag to be analyzed so it is free of all surface contamination. Remove any large stones or debris. A lint-free wipe of isopropyl alcohol (not rubbing alcohol) should be sufficient to clean the bag. Mix the sample thoroughly by kneading the bag. The finer and more homogeneous material will yield more accurate results.

7. The thickness of the plastic in the bag used limits the accuracy of the measurements. Using a 1 mil-thick polyethylene bag offers a reasonable compromise between accurate readings and bag durability. The box of clean bags should be tested by adding silicon dioxide to the bag prior to use.

8. Analyze the plastic storage case surface to determine if the surface is free of lead. This step is important to determine if lead is present on the case prior to sample analysis. If the case readings are positive for lead, clean the case prior to sample analysis and reanalyze. Since the dust samples are thin in mass, background concentrations of the case may be measured in addition to the sample concentrations.

9. Place the bag of dust on the case surface or other substrate that does not contain lead and flatten

to form a continuous uniform layer of at least 1 cm (0.4 inch) thickness (if possible). If limited dust is available shake the bag to consolidate the dust for analysis to better cover the analyzer window.

10. Place the sample window flat against the bag. Do not hold bagged samples in your hand during testing. Do not analyze the bagged samples on top of potentially contaminated surfaces.

11. Place the analyzer so that the sample covers the analysis window. Samples will be analyzed in Trigger-Only method. With the Trigger-Only method, you only need to place the measurement window flush with the sample to be analyzed and pull the trigger for sample analysis to be initiated.

12. Initiate the analysis by depressing the trigger of the analyzer.

13. When the sample has been sufficiently analyzed. The default Minimum Test Time is set to 60 seconds. Stop the analysis.

14. View the composition returned.

15. Remove the sample.

16. Repeat the analysis of the calibration checks and blank analysis after every ten samples.

17. After the final sample analysis at the residence, finish the sequence with calibration checks and blank analysis.

Alternatively, sample may be place in an XRF Sample cup and follow the instructions on pages 61-63 of the IL2 Users Manual and analyzed using the XRF Cup stand holder.

9.0 DATA COLLECTION

Sample identifications, data storage, and archiving may be different for each data user. Data Storage procedures are contained in Chapter 6 and 7starting on page 83 of the user's manual (Exhibit A).

For quality assurance, and to protect against data loss or sample ID confusion, Field Samplers should maintain hand-written notes of all relevant results from each sample, even if the XRF is equipped with a data logging function.

10.0 ROUTINE MAINTENANCE GUIDELINES

Detailed maintenance procedures are provided in Chapter 8 of the user's manual (Exhibit A).

11.0 EXHIBITS

Exhibit A – Niton XL2 Series Analyzer User's Guide, Version 8.0.1 March 2012

12.0 REFERENCES

The Butte-Silver Bow (BSB) County Revised Final Multi-Pathway Residential Metals Abatement Program Plan (RMAP) (BSB & Atlantic Richfield Company, 2021)

Residential Metals Abatement Program (RMAP) Quality Assurance Project Plan (QAPP), (Residential Parcels) October 2022.

US EPA Method 6200, Field Portable X-ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Revision 0, February 2007

13.0 REVISION HISTORY

Section Number	Description of Change
Revision 0	**Initial Draft SOP**

ATTACHMENT B-5

WATER LABORATORY SOPs

Attachment B-5
Water Laboratory SOPs
Index

Laboratory	SOP Number	Revision #	Effective Date	SOP Title	# Pages
Pace	ENV-SOP-MIN4-0044	6	02/23/21	Preparation of Aqueous Samples for ICPMS Analysis by 200.8 and 3020A	13
Pace	ENV-SOP-MIN4-0043	4	02/22/21	Metals Analysis by ICP/MS - Method 6020 and 200.8	24

Pace Analytical®
ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0044 **Revision:** 06

Document Title: Preparation of Aqueous Samples for ICPMS Analysis by 200.8 and 3020A

Department(s): Metals

Date Information

Effective Date: 23 Feb 2021

Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

Document Number: ENV-SOP-MIN4-0044 **Revision:** 06

Title: Preparation of Aqueous Samples for ICPMS Analysis by 200.8 and 3020A

All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0044

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	22 Feb 2021, 03:26:12 PM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	General Manager 2	22 Feb 2021, 03:41:40 PM	Approved
Andrew Mickelson (009792)	Manager	22 Feb 2021, 04:03:19 PM	Approved
Krista Carlson (004514)	Project Manager 1	22 Feb 2021, 05:37:58 PM	Approved

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TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Preparation of Aqueous Samples for ICP-MS Analysis
TEST METHOD EPA 200.8 and 3020A
ISSUER: Pace ENV – Minneapolis– MIN4

1.0 Scope And Application

This standard operating procedure (SOP) describes the laboratory procedure for the preparation of aqueous samples using hot block digestion as described in EPA Method 3020A and EPA 200.8.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in the associated analytical ENV-SOP-MIN4-0043 *Metals Analysis by ICP/MS – Method 6020 and 200.8* (or equivalent replacement).

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, and domestic waste waters.

Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

2.0 Summary of Method

A 25mL aliquot sample is digested in concentrated nitric and hydrochloric acids. After digestion, samples are brought to a final volume of 25mL. Determinative analyses include using Inductively Coupled Plasma (ICP-MS) technologies for trace metals in solution.

Samples requiring dissolved metals analysis must be filtered through a 0.45 micron (µm) filter prior to preservation.

3.0 Interferences

Refer to laboratory SOP ENV-SOP-MIN4-0043 for discussion of potential interferences.

4.0 Definitions

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 Health and Safety

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Preparation of Aqueous Samples for ICP-MS Analysis
TEST METHOD EPA 200.8 and 3020A
ISSUER: Pace ENV – Minneapolis– MIN4

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection.

[1] *Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours following acidification. Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are stored either at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE
TITLE: Preparation of Aqueous Samples for ICP-MS Analysis
TEST METHOD EPA 200.8 and 3020A
ISSUER: Pace ENV – Minneapolis– MIN4

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description	Vendor/Item #/Description
Mechanical pipettes	Various sizes	Fisher Scientific or equivalent
Hot Block ™	54 Place Hot Block	Environmental Express
Analytical Balance	Ability to weigh to the nearest 0.01g	Fisher Scientific or equivalent

7.2 Supplies

Supply	Description	Vendor/Item #/Description
Digestion Cups	50 mL verified to class A specification	Environmental Express or equivalent
Vapor Recovery Device	Reflux cap or Watch glass	Environmental Express or equivalent
Filters	0.45 um	Celltreat or equivalent
Filters	filter mates	Environmental Express, # SC0401

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
De-ionized (DI) water	ASTM Type II	Verify that background levels of volatile compounds are acceptable by analysis
Concentrated nitric acid (HNO_3)	Trace Metal grade	Fisher brand
Concentrated hydrochloric acid (HCl)	Trace Metal grade	Fisher brand

8.2 Standards

Reagent/Standard	Concentration/Description	Requirements/Vendor/Item #
Metals Spike - Stock solution standards for LCS and MS/MSD	The solution identifications are ZPACEMN-105and ZPACEMN-106. See Appendix A for composition	Purchased from Spex (or equivalent). Store at room temperature. Expires as specified by manufacturer.

9.0 PROCEDURE

9.1 Equipment Preparation

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9.1.1 Support Equipment

Calibrate variable and fixed volume pipettes as specified in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

Calibrate the thermometer as specified in in SOP ENV-SOP-MIN4-0161 *Support Equipment* (or equivalent replacement). Calibration records are kept in the QA Office.

Calibrate the turbidimeter as specified in SOP ENV-SOP-MIN4-0110 *Turbidity.* Calibration is performed every 30 days or as needed.

9.1.2 Equipment

The hot block digestors are set to maintain a digestion temperature of 95 +/- 2°C. Use a NIST-traceable thermometer inserted into a digestion cup filled with 50mL of DI to measure the temperature of the hot block. The temperature should be checked in different wells of the hot blocks such that all wells are evaluated over a period of time. Record the temperature of each hot block daily in the temperature logbook.

9.1.3 Turbidity Screen

Samples submitted under SDWA may be analyzed directly without digestion if the turbidity is <1 NTU with the exception of samples requiring the determination of silver. All other samples will be digested following procedures outlined in section 9.2.

9.1.3.1 Verify the expiration date for the current calibration.

9.1.3.2 Using the barcode scanner and barcode sheet (Appendix B) scan the CRDL barcode to enter the sample ID into the instrument. Place the CRDL vial into the vial compartment and close the lid. Repeat with the CCV and CCB.

9.1.3.3 All quality control check samples must meet acceptance criteria prior to analyzing samples. If criteria are not met, the instrument may need to be recalibrated.

QC Sample	True Value	Acceptance	Frequency
CRDL	0.5 NTU	60-140%	Daily, prior to each analytical batch
CCV	10 NTU	90-110%	Daily, before sample analysis, and after every 10 samples.
CCB	N/A	< 1 NTU	Daily, before sample analysis, and after every 10 samples.

9.1.3.4 Allow the samples to come to room temperature before analysis.

9.1.3.5 Mix the samples gently but thoroughly to disperse the solids throughout the container, allowing for air bubbles to disappear prior to taking an aliquot of sample. Carefully dab off any water or moisture on the outside of the sample cell and remove any smudges using a Kimwipe.

9.1.3.6 Scan the sample IDs into the instrument from the barcodes on the batch worklist.

9.1.3.7 Ensure the sample has remained homogenous and place vial containing sample into the vial compartment and close the lid. Record results using preplog template F-VM-M-023 *ICP/ICPMS DW Turbidity* (or equivalent replacement). Detections

exceeding 1 NTU will be scheduled for digestion and analysis, results less than 1 NTU will be scheduled for direct analysis.

9.2 Sample Preparation

9.2.1 Obtain and label digestion tubes in the order for which samples will be poured out.

9.2.2 Transfer a well-mixed 25 mL acid preserved aliquot of the sample to a labeled digest cup. Document the initial volume used.

9.2.2.1 Create a method blank (MB) and laboratory control sample (LCS) using DI water.

9.2.2.2 If the samples are filtered in the lab for dissolved metals, an associated filter blank must be performed and be digested with the batch of samples filtered. The filter blank is not in substitution of the MB, but in addition to.

9.2.3 Spike the LCS (if applicable, LCSD) and matrix spike/matrix spike duplicate (MS/MSD) samples with 0.025 mL of the appropriate spiking standards.

9.2.4 Add 0.5 mL concentrated HNO_3 and 0.25 mL concentrated HCl to each sample.

9.2.5 Cover each digest cup with a ribbed plastic watch glass.

9.2.6 Place samples in a hot block at 95°C +/- 2°C in the hot block. Document temperature of the hot block.

9.2.7 Gently reflux samples down to approximately 5 mL volume. Do not allow the samples to boil or to go to dryness.

9.2.8 Remove from hot block. Document the temperature of the hot block.

9.2.9 Allow the digest to cool. Bring up to a final volume of 25 mL with DI water, cap and mix.

Note: Filter the samples if needed – filtration is to be done only if there is concern that insoluble materials may clog the nebulizer. If any sample is filtered, the MB and LCS must also be filtered. Use the filter mates to plunge-filter the sample in the existing cup.

9.3 Documentation

9.3.1 Digestion Records

Record the necessary information in the electronic prep log using template version F-MN-I-328 (or equivalent replacement). Information includes batch and sample ID, initial and final volumes, initial and final time, prep date, prep analyst, supporting equipment, and lot numbers of solutions used. Also include any additional comments if needed.

10.0 Data Analysis And Calculations

10.1 Calculations

Refer to associated analytical SOP for equations and common calculations.

11.0 Quality Control and Method Performance

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to associated analytical SOP for acceptance criteria and required corrective action.

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QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)[1,2]	As needed when insufficient native sample volume exists
Matrix Spike (MS)	Prepared with each batch of samples. Client specific requirements may result in a greater number of MS or MS/MSD sets in a batch
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Filter Blank (FB)	If applicable

[1]WIDNR requires the use of a lab created matrix solution from unused samples.
[2]In the event that only samples identified as Equipment Blanks and/or Field Blanks are available, and LCS/LCSD will be prepared in place of MS/MSD.

11.2 Method Performance

11.2.1 Method Validation

11.2.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* for these procedures.

11.3 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative

measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to the associated analytical SOP for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV

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corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1. The scope of the method for 3020A has been expanded to include additional metals that require HCl for best solubility and stability at the levels of analysis required by ICPMS. Due to this requirement method 3020A has been modified to include the addition of HCl to the digestion.

14.2. Our procedure uses a final concentration of HNO_3 at 2% and a final HCl concentration of 1%. This is consistent with the digestion prescribed in EPA Method 200.8, however the final HNO_3 concentration differs from that prescribed in method 3020A.

14.3. Method 3020A has been modified to follow EPA 200.8 given the scope of metals in 200.8 are similar to the scope of metals in 6020A.

14.4. Our procedure uses 25 mL initial and final volumes using the hot block digestion system rather than glassware and 100 mL sample volume.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Stock Standard Summary

Appendix B – Turbidity Barcodes

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A, 1992.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3005A.

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U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

40 CFR Appendix B to Part 136, *Definition and Procedure for the Determination of the Method Detection Limit - Rev 2*, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
7.2/8.2	Updated tables with new standards, spike information and remove resin pellets (N/A)
9.1.3.3	Updated CCRDL to CRDL.
App A	New standards, table updated accordingly.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0044	Preparation of Aqueous Samples for ICP-MS by 200.8 and 3020A	05

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Appendix A: Metals Standard Reference

Stock standards used for aqueous sample preparation

ZPACEMN-105		ZPACEMN-106	
Element	(ug/mL)	Element	(ug/mL)
Ca	2000	Si	500
Fe	2000	Sb	100
Mg	2000	Mo	100
K	2000	Sn	100
Na	2000	Ti	100
Al	2000	S	2000
Ba	100	As	100
Be	100	Se	100
Bi	100	Pd	20
B	100	Pt	20
Cd	100		
Th	100		
Cr	100		
Co	100		
Cu	100		
Li	100		
P	100		
Mn	100		
Pb	100		
Ni	100		
Ag	50		
Sr	100		
Tl	100		
V	100		
Zn	100		
U	100		

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Appendix B: Turbidity Barcodes

Turbidity

CRDL	
CCVA	CCVB
CCV	CCB
CAL1	CAL2
ICV	ICB

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ENVIRONMENTAL SCIENCES

Document Information

Document Number: ENV-SOP-MIN4-0043 **Revision:** 04

Document Title: Metals Analysis by ICP/MS - Method 6020 and 200.8

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All dates and times are in Central Time Zone.

ENV-SOP-MIN4-0043

QM Approval

Name/Signature	Title	Date	Meaning/Reason
Janielle Ward (007319)	Manager - Quality	22 Feb 2021, 11:06:56 AM	Approved

Management Approval

Name/Signature	Title	Date	Meaning/Reason
Adam Haugerud (005828)	General Manager 2	17 Feb 2021, 04:18:39 PM	Approved
Andrew Mickelson (009792)	Manager	18 Feb 2021, 08:49:25 AM	Approved
Krista Carlson (004514)	Project Manager 1	18 Feb 2021, 10:54:23 AM	Approved

1.0 SCOPE AND APPLICATION

This standard operating procedure (SOP) describes the laboratory procedure for the determination of dissolved and total recoverable metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS).

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The target analytes and the normal LOQ that can be achieved with this procedure are provided in Table 1, Appendix A.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed. The current LOQ for each target analyte that can be determined by this SOP as of the effective date of this SOP is provided in Table 1, Appendix A.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

1.2 Applicable Matrices

This SOP is applicable to ground, surface, drinking, and storm runoff water samples; industrial, domestic waste waters and solids.

Dissolved elements are determined after suitable filtration and acid preservation. In order to reduce potential interferences, dissolved solids should not exceed 0.2 % (w/v).

For the determination of total recoverable analytes in aqueous samples containing particulate and suspended solids a digestion step is required prior to analysis.

2.0 SUMMARY OF METHOD

Prior to analysis, samples must be solubilized or digested using appropriate sample preparation methods. For the total recoverable determination of analytes in drinking water by 200.8 where sample turbidity is < 1 NTU, the sample is made ready for analysis by the appropriate addition of nitric acid, mixed, and allowed to equilibrate for the required time prior to analysis.

Sample solutions are introduced by pneumatic nebulization into a plasma, in which desolvation, atomization and ionization occurs. Ions are extracted from the plasma through a differentially pumped vacuum interface and sorted on the basis of their mass-to-charge ratio. The ions transmitted through the quadrupole are detected by an electron multiplier. Ion intensities at each mass are recorded and compared to those obtained from external calibration standards to generate concentration values for the samples. Results are corrected for instrument drift and matrix effects using internal standards.

3.0 INTERFERENCES

Isobaric Elemental Interferences – Isobaric elemental interferences result when isotopes of different elements have the same nominal mass-to-charge ratio and cannot be resolved with the instruments spectrometer. One way to solve this problem is to measure a different isotope for which there is no

interference. Alternatively, one can monitor another isotope of the element and subtract an appropriate amount from the element being analyzed, using known isotope ratio information. Corrections for most of the common elemental interferences are programmed into the software.

Isobaric Polyatomic Interferences – Isobaric polyatomic interferences result when ions containing more than one atom have the same nominal mass-to-charge ratio as an analyte of interest and cannot be resolved by the instrument's spectrometer. An example includes ClO+ (mass 51), which interferes with V, and must be corrected by measuring ClO+ at mass 53. When possible an interference free isotope should be chosen for measurement.

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes.

Memory interferences can occur when there are large concentration differences between samples or standards, which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affects the extent of the memory interferences, which are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of

solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

Samples should be collected in accordance with a sampling plan and procedures appropriate to achieve the regulatory, scientific, and data quality objectives for the project.

The laboratory does not perform sample collection or field measurements for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Aqueous	250 mL Plastic	25 mL	Acidified[2] with nitric acid to pH<2, stored ambient	Must be analyzed within 180 days of collection. If mercury is requested, analysis must occur within 28 days of sample collection.
Solid	8 oz glass jar	1 gram	<6°C, but above freezing	

[1]*Minimum amount needed for each discrete analysis.*
[2] *Samples must equilibrate for a minimum of 24 hours following acidification. Lead and Copper Rule Monitoring and Reporting Guidance for Public Water Systems, EPA 816-R-10-004, March 2010, Exhibit II-9, Samples must stand in the original container used for sampling for at least 28 hours after acidification.*

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory ENV-SOP-MIN4-0008 *Sample Management*, or equivalent replacement. Chemical preservation is checked and recorded at time of receipt or prior to sample preparation.

After receipt, samples are either stored at ambient or 6°C until sample preparation. Prepared samples digestates are stored at ambient temperatures until sample analysis.

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 21 days from date of final report and then disposed of in accordance with Federal, State, and Local regulations.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment

Equipment	Description
ICPMS (Inductively Coupled Plasma Mass Spectrometer)	Agilent 7700, 7800 7900 ICPMS instrumentation equipped with interference reduction technology. Each instrument has an associated auto-sampler, rough pump and recirculating chiller.

Centrifuge	Thermo Sorvall Legend XT
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Mechanical pipettors	Eppendorf, Fisher brand or equivalent replacement, various sizes
Glassware	Class A volumetric flasks and graduated cylinders of various sizes

7.2 Supplies

Supply	Description
Argon gas	Praxair or equivalent, High purity grade, 99.99%
Collision Gas	Praxair or equivalent, Ultra high purity He, Ultra high purity H_2,
Analytical Balance	Sartoriius or equivalent, capable of weighing to 0.01g
Auto-sampler tubes	Moldpro or equivalent, 15 mL metals free auto-sampler tubes
Digestion cups	Moldpro or equivalent, 50 mL disposable digestion cups
Data-Uploading Software	Pace internal software used to transfer data from the instrument to the LIMS

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Description
Reagent water	ASTM Type II
Nitric Acid (HNO_3)	Fisher Scientific, A-509-P212 or equivalent replacement
Hydrochloric acid (HCl)	Fisher Scientific, A-508-P212 or equivalent replacement
2% (v/v) Nitric Acid/1% (v/v) Hydrochloric Acid Solution	Used for instrument blanks, standards and dilutions. Prepared in 1 L increments utilizing a volumetric flask and transferring into a C&G narrow mouth storage bottle. This is measured by mixing 20 mL of HNO_3 trace metals grade acid and 10 mL of HCl trace metals grade acid and DI H2O, and bringing to volume of 1 L.
Rinse Blank	2-5% (v/v) Nitric Acid solution for rinsing between runs. Combine76 mL of HNO_3 trace metals grade acid and 38 mL of HCl trace metals grade and DI H2O, and bringing to volume of 1 G.

8.2 Standards

Reagent	Description
Calibration Stock Standards	Custom blend of elements. See Appendix D for the standard information
Agilent Tune Solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
EPA Tune solution	Purchased multi-element standard from a qualified vendor, 10ug/mL.
Internal Standard Stock Solution	Various suppliers; single element standards to be mixed prior to use with concentrations of 1,000 and 10,000 ug/mL
Working Standards	See Appendix C

9.0 PROCEDURE

9.1 Equipment Preparation

Pre-Start Checks: Turn on the computer and load the software. Initiate appropriate operating configuration of the instrument's computer according to the instrument manufacturer's instructions. Check the following:

9.1.1 Support Equipment

- Vacuum pump oil - Examine the sight glasses of the vacuum pump. Oil should be no darker than a light brown color. If it is, change the oil in the pump according to the directions in the manufacturer's guide.
- Chiller temperature, pressure and water level - The temperature should be regulated at 17 ± 1ºC. Check the current temperature on the chiller to ensure it is within this range. Check the inlet cooling water pressure that must be between 55 and 60psi. Check to ensure that chiller water level is full. If it is not, fill with Polyclear 30.
- Verify the level of nebulizer waste and rinse waste, if more than half full, empty it into the acid waste stream.
- Ar/O pressure - The argon supply pressure should be set at about 80psi. If the supply argon pressure falls below about 45psi, a safety interlock automatically shuts off the torch.
- Helium / Hydrogen pressure - The helium and hydrogen supply pressure should be set at about 15 and 9 psi respectively.
- Wash solution level - The wash solution supply is maintained in a 4-liter carboy. Ensure that there is sufficient volume present for the analytical sequence.
- Peristaltic pump tubing - Change the sample and internal standard tubing, spray chamber drain tubing and the rinse station tubing as needed. Signs of degradation include flattened sections and hazy appearance. Allow at least 30 minutes for break-in period.
- Interface cones - Remove and inspect the outside of the sampling and skimmer cones around the orifice. Install a new set of cones if needed or clean the existing cones using the following procedure: Carefully polish each cone with silver polish and cotton swabs dampened with deionized water. Rinse cones with deionized water and blow-dry with house air supply, being careful not to damage the cones. After the cones are fully dry, replace them in the instrument. Allow for conditioning of the cones with a solution containing sufficient concentrations of major cations. The orifice should be circular and about 1mm in diameter. Examine the orifice periodically with a magnifier to determine if there are irregularities that may impair instrument performance. DO NOT use a cone with a significantly degraded tip.

9.1.2 Instrument

Lighting Torch and Warm-Up: After all pre-start checks pass inspection, perform the following steps:

- Torch Ignition - Click on the Plasma icon to open the Instrument window, and then click on the plasma on button to light the plasma. This takes a little over a minute to complete. (See instrument software guide.)
- Warm-up- Instrument is allowed to warm-up 30 minutes. Instrument has a timer to let you know when it is ready to move on to the next step.
- Check peristaltic pump flow by monitoring bubble movement in the pump tubing. Adjust tension as needed to achieve a smooth flow.

- Start-up Configuration - Once the analysis tubing is placed in the Agilent tune solution and stable signal is achieved, the start-up configuration can be initiated. See section 9.1.2.1 for Agilent tune performance monitoring and criteria.
- Create New Experiment File – Open template from the drive. Apply the proper run name for the day (MMDDYYICPMS#). Introduce EPA tune solution and allow signal to stabilize. Initiate performance verification for each mode of analysis. Save each performance report to the network drive. See section 9.1.2.1 for EPA tune acceptance criteria.

9.1.2.1 Routine Instrument Operating Conditions

The instrument is configured to go through the manufacturer recommended startup tune procedure which includes; Torch Alignment, Axis/Resolution, EM settings, Plasma Correction, Standard Lenses tune, and standard mode performance verification. The measured ratios of oxides 156/140 and doubly charged 70/140 should be <3%. The measured masses of ^{7}Li, ^{89}Y, ^{205}Tl are monitored for initial resolution/axis tuning. EPA Performance verification is later performed for each cell condition used for sample analysis.

EPA Tune Verification - The EPA tuning standard must be analyzed in each mode of analysis to verify resolution and mass calibration are within the required specifications. The tuning standard is analyzed in each mode of analysis at least five times and the relative standard deviation (RSD) must be <5% for all analytes contained in the tuning standard. Conduct mass calibration and resolution checks in the mass regions of interest. If the mass calibration differs more than 0.1 amu from the true value, then the mass calibration must be adjusted to the correct value. The resolution must also be verified to be <0.9 amu full width at 5% peak height.

Pace Minneapolis maintains approval for the analysis of up to 35 elements by the EPA Methods 200.8, 6020, 6020A, 6020B for water and soil matrices. All target analytes are analyzed either in a Helium mode (Collision Cell), hydrogen (Collision Cell), or No gas mode on the Agilent instruments depending on the sample matrix type. The use of interference reduction technologies (Collision Cell) is not allowed for drinking water analysis. Separate calibrations are performed for samples reporting by regulation of the SDWA.

9.2 Initial Calibration

9.2.1 Calibration Design

The calibration curve must consist of a minimum of a calibration blank and five non-zero standards for each mode of analysis. Use the average of at least three integrations for both calibration and sample analyses. Using the instrumentation software, prepare a standard curve for each element by plotting absorbance versus concentration. The working range varies with each analyte, see appendix C for summary. The calibration is a linear regression using equation; $y = mx + b$ The analyst may employ a regression equation that does not pass through the origin, however forcing through zero is not allowed. Additional calibration specifications may be referenced in ENV-POL-CORQ-0005 *Acceptable Calibration Practices for Instrument Testing*, or equivalent replacement.

9.2.2 Calibration Sequence

Calibration Blank (CAL0)

CAL1
CAL2
CAL3
CAL4
CAL5
CAL6 (optional)
CAL7 (optional)
ICV
ICB
CRDL
ICSA
ICSAB
CCV
CCB
Client samples
CCV
CCB
CRDL (Optional)

9.2.3 ICAL Evaluation

9.2.3.1 Curve Fit

With a multi-point calibration, the regression calculation will generate a correlation coefficient (r) that is the measure of the "goodness of fit" of the regression line to the data. In order to be used for quantitative purposes, the correlation coefficient must be > 0.998.

9.2.3.2 Relative Standard Error (RSE)

%RE is measured at the lowest calibration level and at a point near the mid-level of the calibration (the continuing calibration verification level is recommended). In order for a standard curve to be acceptable, the correlation coefficient/coefficient of determination criterion specified in the method must be met **and** both the low-level and mid-level %RE measures must meet the acceptance criteria. The low-level %RE acceptance criteria is 60%-140% and the mid-level is 90-110%.

9.2.3.3 Initial Calibration Verification

In addition to meeting the linearity requirement, any new calibration curve must be assessed for accuracy in the values generated. To assess the accuracy, a single standard from a secondary source must be analyzed and the results obtained must be compared to the known value of the standard. This step is referred to as Initial Calibration Verification. The ICV is analyzed immediately following an initial calibration curve.

9.2.4 Continuing Calibration Verification

A CCV followed immediately by a CCB must be analyzed after every 10 samples and at the end of the analytical batch to verify the system is still calibrated.

9.3 Digestate Preparation

9.3.1 Homogenization and Subsampling

All solid matrices are subject to centrifuge at a rate of 1000 rpm for 15 minutes or allowed to settle overnight prior to analysis. Once samples have been centrifuged or allowed to settle, an initial dilution of 20 fold is performed on each sample. This is completed by taking 4.75mL of 2% HNO3 / 1% HCL diluent and mixing with a 0.25mL aliquot of sample by means of vortex.

Aqueous samples are inverted multiple times and poured without initial dilution unless historical data demonstrates otherwise.

9.4 Analysis

The instrument performs sample analysis by executing 100 mass sweeps per replicate. Three replicates are utilized for an average result which must fall within a 20% RSD for the replicate values. If any sample or QC is found to have a concentration of >5x the RL and >20% RSD it must be evaluated for interference. If a matrix interferent is determined to be the cause, dilute the sample by 5x and re-analyze. Perform further dilutions if necessary.

The instrument(s) have been setup and configured in conjunction with manufacturer specifications. Masses were carefully selected to avoid and/or minimize interferences. Internal standard selection was based on performance for the appropriate mass range. Internal standard association must remain within 50 amu of targeted analyte.

The total recoverable sample digestion procedure is suitable for the determination of silver in aqueous samples containing concentrations up to 0.1 mg/L. For the analysis of wastewater samples containing higher concentrations of silver, succeeding smaller volumes of well mixed sample aliquots must be prepared until the analysis solution contains < 0.1 mg/L silver.

10.0 Data Analysis And Calculations

See the laboratory SOP ENV-SOP-MIN4-0171 *Laboratory Calculations*, or equivalent replacement, for equations for common calculations.

10.1 Hardness as CaCO3 in mg/L = 2.497 * [Ca in mg/L] + 4.118 * [Mg in mg/L]

10.2 Concentration of lead = summation of signals at 206, 207, and 208 m/z.

10.3 Silica (SiO2) (µg/L) = Silicon (Si) (µg/L) * DF * 60.09 amu (SiO2 molecular weight) / 28.09 amu (Si atomic weight)

Where: DF is the sample Dilution Factor

10.4 The corrected dry weight concentration can be calculated using the following:

$$corrected\ dry\ wt\ conc = \frac{\left(c \times \frac{v_f}{wt_i}\right)}{\%\ dry\ wt}$$

Where, c = concentration on instrument, µg/L

v_f = final volume, L
wt_i = initial weight, g

$$\%Dry\ weight = \frac{Sample\ Dry\ Weight}{Sample\ Wet\ Weight} \text{x}100$$

10.5 Calculate the Relative Percent Difference (RPD) between the matrix spike and matrix spike duplicate using Equation 1:

Equation 1

$$\%RPD = \frac{|S - D|}{(S + D)/2} x100$$

Where, S = Sample result, mg/L or mg/kg

D = Duplicate sample result, mg/L or mg/kg

11.0 QUALITY CONTROL AND METHOD PERFORMANCE

11.1 Quality Control

The following QC samples are prepared and analyzed with each batch of samples. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Method Blank (MB)	1 per batch of 20 or fewer samples.
Laboratory Control Sample (LCS)	1 per batch of 20 or fewer samples.
Laboratory Control Sample Duplicate (LCSD)	As needed
Matrix Spike (MS)	1 per batch of 20 or fewer samples for 6020 (A)(B). 1 per batch of 10 or fewer samples for 200.8
Matrix Spike Duplicate (MSD)	1 per batch of 20 or fewer samples.
Sample Duplicate	Performed at client request.
Serial Dilution	1 per batch of 20 or fewer samples.
Post Digestion Spike	1 per batch of 20 or fewer samples for method 6020(A)(B).
Internal Standard	An appropriate internal standard is required for each analyte and sample determined by ICP-MS.

Internal Standard	Associated element
Scandium 45	Li, Be, B, Na, Mg, Al, Si, K, Ca, Ti, V, Cr, Mn, Fe, Se
Germanium 72	Co, Ni, Cu, Zn, As, Sr
Indium 115	Mo, Pd, Ag, Cd, Sn, Sb
Terbium 159	Ba, Pt, Hg, Tl, Pb, Bi
Iridium 193	U Th

11.2 Instrument QC

The following Instrument QC checks are performed. Refer to Appendix B for acceptance criteria and required corrective action.

QC Item	Frequency
Tune	Daily prior to any calibration
Initial Calibration	Daily
Initial Calibration Verification	Immediately after each initial calibration
Initial Calibration Blank	Immediately after each initial calibration
Continuing Calibration Verification	Prior to the analysis of any samples and after every 10 injections thereafter. Samples must be bracketed with a closing CCV standard.
Continuing Calibration Blank	Following every CCV injection
CRDL / LLCCV verification	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.
ICSA verification	At the beginning of each sample run sequence after the CRDL. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.
ICSAB verification	At the beginning of each sample run sequence after the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.

11.3 Method Performance

11.3.1 Method Validation

11.3.1.1 Detection Limits

Detection limits (DL) and limits of quantitation (LOQ) are established at initial method setup and verified on an on-going basis thereafter. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* and to the laboratory's SOP ENV-SOP-MIN4-0163 *Determination of LOD and LOQ* (or equivalent replacement) for these procedures.

11.4 Analyst Qualifications and Training

Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP ENV-SOP-MIN4-0165 *Orientation and Training Procedures* (or equivalent replacement) for more information.

12.0 Data Review And Corrective Action

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP ENV-SOP-MIN4-0092 *Data Review Process* (or equivalent replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be near the midpoint of the calibration range. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

Refer to Appendix B for a complete summary of QC, acceptance criteria, and recommended corrective actions for QC associated with this test method.

13.0 Pollution Prevention And Waste Management

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to: reduced solvent extraction, solvent capture, use of reusable containers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's Chemical Hygiene Plan / Safety Manual.

14.0 Modifications

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or

extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.1 Tuning criteria observed is more stringent than required by the SW846 methods so that the same criteria can be used for both methods 6020 and 200.8.

14.2 The following elements are not listed in the method 6020A recommended analyte list; bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium, thorium, and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.3 The following elements are not listed in the method 200.8 recommended analyte list: bismuth, boron, calcium, iron, lithium, magnesium, palladium, platinum, potassium, silica, silicon, sodium, strontium, tin, and titanium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

14.4 The following elements are not listed in the method 6020B recommended analyte list: bismuth, boron, lithium, molybdenum, palladium, platinum, silica, silicon, strontium, tin, titanium and uranium. The accuracy and precision for the analysis of these analytes have been demonstrated in the matrices of interest, at the concentration of interest, and in the same manner as the elements recommended in the method.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

Appendix A – Target Analyte List and Routine LOQ

Appendix B – QC Summary

Appendix C – Working Standard Summary

Appendix D – Stock Standard Summary

17.0 REFERENCES

Pace Quality Assurance Manual- most current version.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-V1-2009.

TNI Standard, *Management and Technical Requirements for Laboratories Performing Environmental Analyses*, EL-VI-2016-Rev.2.1.

U.S. Environmental Protection Agency. Method 200.8, Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometer, Revision 5.4, EMMC Version, May 1994.

U.S. Environmental Protection Agency. SW846 Method 6020, Inductively Coupled Plasma – Mass Spectrometry, Revision 0, 9/94.

U.S. Environmental Protection Agency. SW846 Method 6020A, Inductively Coupled Plasma – Mass Spectrometry, Revision 1, 02/2007.

U.S. Environmental Protection Agency. SW846 Method 6020B, Inductively Coupled Plasma – Mass Spectrometry, Revision 2, 7/2014.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3020A.

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846, Third Edition. Method 3050B.

40 CFR Appendix B to Part 136, Definition and Procedure for the Determination of the Method Detection Limit - Rev 2, August 28, 2017.

18.0 REVISION HISTORY

This Version:

Section	Description of Change
6.0	Updated sample retention from 45 to 21 days.
8.2	Internal Standard Stock Solution – added “1,000 and”
9.2.1	Updated 3 to 5 non-zero standards. Added “The working range…C for summary.”
9.2.2	Added “(optional)” to CAL6. Added “CAL7 (optional)”.
10.0	Added sections 10.4 and 10.5.
11.1	Updated Thoridium 232 to Iridium 193.
14.0	14.2 & 14.4: removed “-238” from uranium. 14.2: added thorium.
17.0	Removed references for Fisions and Region 9 Laboratory SOP.
Appendix A	Added Thorium. Updated Silica and Silicon entries. Removed Mercury NPW and potable water entries.
Appendix B	Updated ICAL Acceptance Criteria. Updated methods referenced in MB Acceptance Criteria. Added LDR acronym to QC Item.
Appendix C & D	Re-formatted tables.

This document supersedes the following document(s):

Document Number	Title	Version
ENV-SOP-MIN4-0043	Metals Analysis by ICP/MS – Method 6020 and 200.8	03

Appendix A: Target Analyte List and Routine LOQ[1]

Analyte	Non-Potable Water (ug/L)	Potable Water (ug/L)	Soil (mg/kg)
Aluminum	20.00	20.0	20.00
Antimony	0.50	0.50	0.50
Arsenic	0.50	0.50	0.50
Barium	0.30	0.30	0.30
Beryllium	0.20	0.20	0.20
Bismuth	0.50	-	0.50
Boron	10.00	-	10.00
Cadmium	0.08	0.08	0.08
Calcium	40.00	-	40.00
Chromium	0.50	0.50	0.50
Cobalt	0.50	-	0.50
Copper	1.00	1.00	1.00
Iron	50.00	-	50.00
Lead	0.10	0.10	0.20
Lithium	0.50	-	0.50
Magnesium	10.00	-	10.00
Manganese	0.50	0.50	0.50
Mercury	-	-	0.20
Molybdenum	0.50	-	0.50
Nickel	0.50	0.50	0.50
Palladium	0.50	-	-
Platinum	0.50	-	-
Potassium	100.00	-	100.00
Selenium	0.50	0.50	0.50
Silica	214.00	-	214.0
Silicon	100.00	-	100.00
Silver	0.50	0.50	0.50
Sodium	50.00	-	50.00
Strontium	0.50	-	0.50
Thallium	0.10	0.10	0.10
Thorium	0.50	-	0.50
Tin	0.50	-	2.000
Titanium	1.00	-	1.00
Vanadium	1.00	1.00	1.00
Uranium-238	0.50	0.50	0.50
Zinc	5.00	5.00	5.00

[1] *Values in place as of effective date of this SOP. LOQ are subject to change. For the most up to date LOQ, refer to the LIMS or contact the laboratory.*

Appendix B: QC Summary

QC Item	Frequency	Acceptance Criteria	Corrective Action	Qualification
Tune	Daily prior to any calibration	Adjust spectrometer resolution to produce a peak width of approximately 0.75 amu at 5% peak height. This must be completed using 5 replicates with a resulting RSD of <5%.	Adjust mass calibration if it has shifted by more than 0.1 amu from unit mass. Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis.
ICAL	Daily	r ≥ 0.998 a Midlevel (recommended near ICV/CCV concentrations) %RE 90-110% Low-Level (Cal1) %RE 60-140%	Identify and correct source of problem, repeat.	None. Do not proceed with analysis.
ICV	After Each ICAL	All analytes must be within ± 10% of the true value. (%R)	Identify source of problem, re-analyze. If repeat failure, repeat ICAL. Analysis may proceed if it can be demonstrated that the ICV exceedance has no impact on analytical measurements. For example, the ICV %R is high, CCV is within criteria, and the analyte is not detected in sample(s).	Qualify analytes with ICV out of criteria.
ICB	Immediately after the initial calibration verification	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A)(B) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the ICB exceedance has no impact on analytical measurements. For example, the ICB has detections and the analyte is not detected in sample(s).	Qualify analytes with ICB out of criteria.
CRDL / LLCCV	At the beginning of each run for 6020/6020B/200.8 and must be analyzed at the beginning of each run, and once at the end of each analytical batch for 6020A.	For 6020/200.8: The acceptance criteria are ± 40% (or specified by the client). For 6020A: The acceptance criteria are ± 30% (or specified by the client). 6020B: The acceptance criteria is ± 20% (or specified by the client).	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CRDL exceedance has no impact on analytical measurements. For example, the CRDL %R is high and the analyte is not detected in sample(s). For example, the CRDL %R is high and the analyte detections exceed the continuing calibrations verification level (midpoint of the curve).	Qualify outages and explain in case narrative.

			If the CRDL is biased low, no data can be reported for the target elements failing criteria.	
CCV	Daily, before sample analysis, after every 10, and at end of analytical window.	All analytes must be within ± 10% of the true value. (%R): %RSD between multiple integrations must be ≤ 5%	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCV exceedance has no impact on analytical measurements. For example, the CCV %R is high, and the analyte is not detected in sample(s).	Qualify analytes with CCV out of criteria.
CCB	Daily, before sample analysis, after every 10, and at end of analytical window	All elements of interest must be evaluated to a criterion of +/- ½ of the RL for method 6020 (A) and samples originating from NC. All elements of interest must be evaluated to +/- the RL for method 200.8, and 6020 (B). WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. Analysis may proceed if it can be demonstrated that the CCB exceedance has no impact on analytical measurements. For example, the CCB has detections and the analyte is not detected in sample(s).	Qualify analytes with CCB out of criteria.
Internal Standards	Every field sample, standard and QC sample	For method 6020, the intensity of internal standard in the ICB/CCB and ICS (ICSA/AB) standards must not deviate more than 80-120% from its original intensity in the associated calibration blank. The intensity of internal standard in the samples and remaining QC must not deviate more than 30-120%. For method 6020A/B, the intensity of the internal standard must not fall below 70% and not exceed 130% from its original intensity in the associated calibration blank. For Method 200.8 the intensity of internal standard in the samples and QC must not deviate more than 60-125% from its original intensity in the associated calibration blank.	Troubleshoot instrument performance. Reanalyze samples and dilute if needed.	Qualify outages and explain in case narrative.
Interference check solutions	ICSA containing high concentrations of C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti is analyzed at the beginning of each sample run sequence after the CRDL. ICSAB containing high concentrations of	ICSA all spiked elements are to be within 20% of the expected true value. The non-spiked elements are to be below the RL. ICSAB all spiked elements are to be within 20% of the expected true value.	Identify and correct source of problem, repeat performance verification(s).	None. Do not proceed with analysis for elements that cannot be verified.

	C, Cl, Al, Ca, Fe, K, Mg, Mo, Na, P, S and Ti and mid-range concentrations of the remaining elements is analyzed at the beginning of each sample run sequence following the ICSA. 6020A and 6020B requires the ICSA/AB be analyzed every 12 hours thereafter.			
Method Blank (MB)	One per 20 samples	Method 200.8: The method blank is considered to be acceptable if it does not contain the target analytes that exceed 1/2 LLOQ or project-specific DQOs. Method 6020, 6020A and 6020B: The method blank is considered to be acceptable if it does not contain the target analytes that exceed the LLOQ or project-specific DQOs.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If the method blank exceeds the criteria, but the associated samples are either below the reporting level or other DQOs, or detections in the sample are >10x MB detections then the sample data may be reported. J-flag qualification will be applied for blank detections between the LOQ and LOD when DQOs require evaluation to the MDL.	Qualify outages and explain in case narrative.
LCS	One per 20 samples	6020/6020A/6020B: 80-120% 200.8: 85-115%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
LCSD	An LCSD must be substituted in the event of insufficient sample volume for a matrix spike duplicate sample.	6020/6020A/6020B: 80-120% 200.8: 85-115% %Diff ≤ 20%	Identify source of problem, re-analyze. If reanalysis of the LCS fails, all samples affected by the failing LCS elements need to be re-digested and re-analyzed. If LCS recovery is > QC limits and these compounds are non-detect in the associated samples	Qualify analytes with LCS out of criteria.
MS/MSD	One per 20 samples for 6020 / 6020A / 6020B One per 10 samples for 200.8	6020/6020A/6020B: 75-125% 200.8: 70-130%	Perform a SD and PDS on any elements that fail to meet criteria for method 6020(A)(B).	Qualify analytes with MS out of criteria.
Sample Duplicate	Per client request	%Diff ≤ 20%	Qualify outages	Qualify outages.
Serial Dilution[1]	One per batch of 20 samples or less		If criteria is not met, original sample and dilution shall be	Qualify outages.

		6020/6020A fivefold dilution must agree within ± 10% of the original determination if analyte concentration is >50x MDL. 6020B 1:5 dilution of sample 25x > LLOQ or 1:5 dilution of MS since reasonable concentrations are present, results to agree to ± 20%.	reanalyzed. If reanalysis fails, it is determined to be matrix interference.	
Post Digestion Spike[2]	One per batch if there is a MS failure.	6020/ 6020A 80-120% 6020B applicable to elements failing MS, results to agree to +/- 25%. Recommended if high concentration sample not available for dilution test.	If the element fails to meet the recovery criteria, reanalyze. If reanalysis fails, it is determined to be matrix interference.	Qualify outages.
Laboratory Filter Blank (FB)	Analyzed only with batches of lab filtered dissolved metals, one per batch of 20 or less.	Target analytes must be less than reporting limit. NC samples are required to be < ½ RL for target analytes. WIDNR and West Virginia require samples to be reported to the MDL. The blanks must be clean to the data quality objectives.	Identify source of problem, re-analyze. If reanalysis of the MB fails, all samples affected by the failing MB elements need to be re-digested and re-analyzed. If sample(s) non-detect, report the data. If sample result >10x MB detections, report the data.	Qualify outages and explain in case narrative.
Linear Dynamic Range (LDR)	For method 6020B: Following calibration, the laboratory may choose to analyze a standard at a higher concentration than the high standard in the calibration. If a linear range standard is not analyzed for any specific element, the highest standard in the calibration becomes the linear range.	The standard must recover within 10% of the true value, and if successful, establishes the linear range. In each scenario, the linear range is established using 90% of the highest calibration level or LDR sample.	The linear range of the instrument must be adjusted until 90% recovery of the reference standard can be achieved as well as maintaining the minimum number of calibration standard requirements.	N/A

[1]To prepare a 5-fold dilution: take a 1 mL aliquot from the sample and add to 4 mL of diluent. Note: this is a typical process for 200.8 and 6020W. It can be replicated for the preparation of highly concentrated samples by starting with a diluted "parent" sample and then performing the stepwise dilution process.
[2]To Prepare a Post Digestion Spike: An aliquot of the parent sample used for the MS, prepared at the same dilution as the parent sample. The spike addition should produce a minimum level of 10 times the lower limit of quantitation; routine spike volume is 0.020 mL of 20/250 mg/L and 1mg/L mercury stock concentration(s).

Appendix C: Working Standard Summary

Standard	Standard(s) Used	Standard(s) Amount (mL)	Diluent	Diluent Volume (mL)	Final Total Volume[1] (mL)	Final Concentration (ug/L)
Internal Standard	6020-Ge	1	See table 8.1	495	500	2000
	6020-Sc	1				
	6020-Tb	1				
	6020-In	1				
	6020-Ir	1				
Bi/Th primary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Bi/Th secondary (Intermediate)	6020-Th	0.5		49.5	50	1,000
	6020-Bi	0.5				
Hg 10ppb (intermediate)	HG-LL Stock	0.05		49.95	50	10
6020 Hg-SPK	MERC-STK1	0.05		49.95	50	1000
Hg (Intermediate) C	MERC-STK2	0.25		249.75	250	1000
6020-SPK (intermediate)	Bi-STK	0.2		4.6	10	20,000 / 250,000 / 500,000
	Th-STK	0.2				
	HP7375	5				
6020-SPK2 (intermediate)	HP7376	1		9	10	20,000
6020-SPK3 (intermediate)	HP7379	1		9	10	20,000 / 10,000
CAL-SPK1 (intermediate)	HP7375	0.25		9.5	10	25000/12500/1000/500/10
	HP7379	0.05				
	HP7376	0.05				
	6020Hg-SPK	0.1				
	Bi/Th Intermediate	0.05				
Cal 0	N/A	N/A		50	50	0
Cal 1	ZPACEMN103	0.1		9.7	10	Varied
	ZPACEMN104	0.1				
	Hg 10ppb (intermediate)	0.1				0.1
Cal 2	CAL-SPK1	0.1		9.9	10	250/125/10/5/0.1
Cal 3	CA:L-SPK1	0.5		9.5	10	1250/625/50/25/0.5
Cal 4	CAL-SPK1	1		9	10	2500/1250/100/50/1

Cal 5	CAL-SPK1	2.5		7.5	10	6250/3120/250/125/2.5
Cal 6	CAL-SPK1 (intermediate)	5		-	5	25000/12500/1000/500/10
CRDL	ZPACEMN-103	0.1		9.6	10	varied
	ZPACEMN-104	0.1				
	6020 Hg-SPK	0.2				0.2
ICS-A	ICS-ICPMS	0.25		9.75	10	25000/500
ICS-AB	ICS-ICPMS	0.25		9.56	10	27500/26200/1250/600/100/50/4
	6020-SPK	0.05				
	6020-SPK2	0.05				
	6020-SPK3	0.05				
	6020Hg-SPK	0.04				
ICV / CCV add Hg	XPACEMN-75	0.05		49.31	50	4/80/1000
	XPACEMN-76	0.02				
	Bi/Th Intermediate	0.4				
	XPACEMN-77	0.02				
	Hg Intermediate C	0.2				

[1]Alternate final volumes may be prepared at the discretion of the scientist, so long as the concentrations specified above are maintained.

Appendix D: Stock Standard Summary

Stock Standard Concentrations

	HP7379	HP7376	HP7375	XPACEMN 77	XPACEMN 76	XPACEMN 75	ZPACEMN 103	ZPACEMN 104	ICS-ICPMS	Agilent Tune	EPA Tune
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Aluminum	-		1000			1000	2		1,000		
Antimony		200		200				0.005			
Arsenic	200				200			0.05			
Barium	200				200		0.03				10
Beryllium	200				200		0.02				10
Bismuth							0.05				
Boron		200		200			1				
Cadmium	200				200		0.008				
Calcium			1000			1000	4		1,000		
Chromium	200				200		0.05				
Cobalt	200				200		0.05			10	10
Copper	200				200		0.1				
Iron			500			500	5		1,000		
Lead	200				200		0.01				
Lithium	200				200		0.05			10	10
Magnesium			1000			1000	1		1,000		10
Manganese	200				200		0.05				
Molybdenum		200		200				0.05	20		
Nickel	200				200		0.05				
Palladium		200		200				0.05			
Platinum		200		200				0.05			
Potassium			1000			1000	10		1,000		
Selenium	200				200			0.05			
Silicon			500			500		10			
Silver	100				100		0.05				
Sodium			1000			1000	5		1,000		
Strontium	200				200		0.05				
Thallium					100		0.01			10	10

Tin		200		200		20		0.05			
Titanium		200		200		20		0.1	20		
Vanadium	200				200		0.1				
Zinc	200				200		0.5				
Uranium	200						0.05				10
Indium											10
Cesium					200						10
Cerium										10	
Yttrium										10	10
Rhodium											10
Thorium							0.05				

Single Element Stock Standard Concentrations

	Bi-STK (Spex)	Bi-STK (Agilent)	6020-Th (Spex)	6020-Th (Agilent)	MERC-STK1	MERC-STK2	HG-LL Stock	6020-Ge	6020-Sc	6020-Tb	6020-In	6020-Ir
Analyte	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Bismuth	1000											
Bismuth		1000										
Thorium			1000									
thorium				10000								
Mercury					1000							
Mercury						1000						
Mercury							10					
Germanium								1000				
Scandium									10000			
Terbium										1000		
Indium											1000	
Iridium												1000

ATTACHMENT C

RESIDENTIAL DRINKING WATER

SAMPLE COLLECTION PROCEDURES

Attachment C

The City-County of Butte-Silver Bow
Department of Reclamation & Environmental Services
Residential Metals Abatement Program (RMAP) Butte Silver Bow

Residential Drinking Water Sample Collection Procedures

Background: On June 7, 1991, the US EPA published a regulation in the *Federal Register* to control lead in drinking water. The purpose of the lead regulation is to protect public health by minimizing lead levels in drinking water. The presence of lead in drinking water is primarily due to the corrosion and distribution of household plumbing materials including lead containing solder, and therefore, tap water samples are collected at kitchen or bathroom taps of residences.

Process: Drinking water will be sampled as a component of an elevated blood level (EBL) investigation under the RMAP for the presence of lead on a location-specific basis. Once BSB has determined that residential drinking water should be sampled at a particular residence, BSB will contact the homeowner to initiate and streamline the sampling and analysis process as much as possible for homeowners. Upon request by the homeowner, BSB will provide a certified clean sample bottle and sampling instructions. Once the sample has been collected in accordance with the procedures below, BSB will pick up the sample and ship the sample to the laboratory for analysis. The results will be provided electronically to BSB for review. After BSB review the drinking water results will be reported directly to the homeowner. No analysis or shipping costs will be incurred by the homeowner.

SAMPLE INFORMATION	CONTACT INFORMATION
BSB Assigned Sample Identifier	**Name:**
Name of Well Owner:*	**Address:**
City/State/Zip:*	**City/State/Zip:**
Source of Water (if known) City or Private Well	**Email:**
Collection Date:	**Phone #:**
Collection Time:	
Sampler Name:*	Signature of Sampler
Sampler Phone #*:	Received by (Laboratory Use Only

*If different than contact information provided.

Laboratory Use Only
Received By:
Date:
Time:

Please perform sampling as detailed below.

Pre-Sampling Preparation

1. A kitchen cold water faucet is the preferred tap for obtaining a drinking water sample.

2. Warning: Do not collect samples from water faucets connected to a water softener or those having a filtration unit. Do not remove faucet aerators prior to flushing the lines or prior to sampling for lead. In the event these conditions cannot be met substitute a water faucet in a bathroom or other tap used for obtaining drinking water,

3. In the event a tap contains a mixed hot and cold source, turn off the hot source before flushing. Pre-flush the tap for several minutes with cold water, the rate of flushing should be similar to filling a glass for drinking.

4. Turn off the tap and allow the water to stand in the pipes for a minimum of 6 hours to approximately 10 hours during which there is no water used from the tap. BSB recommends pre-flushing the system at night by running cold water through the faucet then collecting the sample first thing in the morning after the water has been undisturbed in your pipes overnight.

Collection of Sample

1. Using a separate sink, wash hands thoroughly prior to sampling to ensure potential contamination is removed prior to sampling.

2. Following the 6 to10-hour stagnation period, the water sample collected for lead testing must be "first draw" water sample. Open the empty 1-L plastic bottle provided by the laboratory being careful not to touch the inside of the sample lid or vial or the threads of the bottle. Place the cap on a stable surface with the inside of the cap facing up. Do not put the cap in a jacket pocket or elsewhere where contamination may occur.

3. Place the sample bottle under the faucet, slowly open the cold water tap, and immediately fill the sample container at about the same rate that you would normally fill a drinking glass. Completely fill (to the top of the bottle shoulder) the bottle and close the bottle tightly being careful to not touch the inside of the cap or water sample. <u>DO NOT CHANGE THE FLOW RATE</u>; otherwise, particles can be dislodged from piping. Do not overflow the sample bottle.

4. Using an indelible blue or black ball-point pen, complete the pre-printed sample label (date and time of sample collection) on the sample bottle.

The sample does not require refrigeration or other handing procedures

5. In the event errors occur during sampling, call the BSB contact information on this form to request another sampling kit.

6. Sample results will be submitted to BSB for evaluation and storage in the BSB RMAP database.

7. After evaluation of the data BSB will contact you with an interpretation of results.

If you have any questions please contact the following:

<u>Butte-Silver Bow Residential Metals Abatement Program</u>
Phone: 406-497-5040

ATTACHMENT D

LEVEL A/B ASSESSMENT CHECKLIST

LEVEL A/B FIELD DOCUMENTATION SCREENING REVIEW

SILVER BOW CREEK/BUTTE AREA NATIONAL PRIORITIES LIST SITE, BUTTE PRIORITY SOILS OPERABLE UNIT, RESIDENTIAL METALS ABATEMENT PROGRAM PROJECT

SOIL SAMPLES COLLECTED ON

MONTH DAY, XXXX

RESIDENT IDENTIFICATION: XXXXX

SAMPLE DELIVERY GROUP(S): XXXXXXXX

DATE

Prepared for:

ATLANTIC RICHFIELD COMPANY
317 Anaconda Road
Butte, MT 59701

Prepared by:

ENVIRONMENTAL STANDARDS, INC.
1140 Valley Forge Road
P.O. Box 810
Valley Forge, PA 19482-0810

INTRODUCTION

This quality assurance (QA) review of field documents is based upon an examination of the data generated during the collection of the field samples on DATE, as part of the Silver Bow Creek/Butte Area National Priorities List (NPL) Site, Butte Priority Soils Operable Unit, Residential Metals Abatement Program (RMAP) sampling event. This review was performed using guidance from the RMAP Quality Assurance Project Plan (QAPP; XXXXXX), Section 6.1.2.1 Field Data Verification.

The Level A/B review is documented on the checklist below as described in the CFRSSI Data Management/Data Validation (DV/DM) Plan (ARCO, 1992a) and the CFRSSI DM/DV Plan Addendum (AERL, 2000), and will be used in the verification process for field documentation related to samples collected for laboratory analyses.

Data that meet the Level A and Level B criteria and are not qualified as estimated or rejected during the analytical data validation process are assessed as enforcement quality data and can be used for all Superfund purposes and activities. Data that meet only the Level A criteria and are not rejected during the data validation process can be assessed as screening quality data. Screening quality data can be used only for certain activities, which include engineering studies and design. Data that do not meet the Level A and/or B criteria and/or are rejected during the data validation process are designated as unusable. The determination of enforcement quality data and screening quality data will be made in conjunction with the data validation report and qualified based on the requirements of Section 6.3 of the QAPP. Identification of enforcement, screening or unusable data will be added to the electronic data deliverables.

SECTION 1 LEVEL A/B FIELD DOCUMENTATION SCREENING REVIEW

1. General Information

Site:
Project:
Client:
Sample Matrix:

2. Screening Result

Data are:

- Unusable ☐
- Level A ☐
- Level B ☐

3. Level A Criteria: The following must be fully documented

Criteria		Comments
Sampling date	Yes ☐ No ☐	Recorded in Logbook ☐ COC ☐ Bottle Labels ☐
Sampling team or leader name	Yes ☐ No ☐	Recorded in Logbook ☐ COC ☐
Physical description of sampling location	Yes ☐ No ☐	Recorded in Logbook ☐ Field Forms ☐ Photo Log ☐
Sample collection depth (soils)	Yes ☐ No ☐	Recorded in Logbook ☐ Field Forms ☐
Sample collection technique	Yes ☐ No ☐	Collected in accordance with the SOPs in attachment C-1 of QAPP Yes ☐ No ☐
Field preparation technique	Yes ☐ No ☐	Collected in accordance with the SOPs in attachment C-1 of QAPP Yes ☐ No ☐
Sample preservation technique	Yes ☐ No ☐	Soils for mercury analysis submitted on ice? Yes ☐ No ☐ Soils for lead and arsenic submitted at ambient temperature? Yes ☐ No ☐
Sample shipping records	Yes ☐ No ☐	Did sample arrive at < 6°C but not frozen (mercury analysis)? Yes ☐ No ☐ _____ Reported (corrected) temperature

4. Level B Criteria – The following must be fully documented.

Criteria		Comments
Field instrumentation methods and standardization complete.	Yes ☐ No ☐	Field equipment calibrated if used? Yes ☐ No ☐
Sample container preparation	Yes ☐ No ☐	Unpreserved bottles provided by laboratory and lot number tracked? Yes ☐ No ☐
Collection of field duplicates (1/20 minimum)	Yes ☐ No ☐	
Sampling equipment decontamination	Yes ☐ No ☐	Dedicated sampling equipment decontaminated per QAPP Yes ☐ No ☐
Field custody documentation	Yes ☐ No ☐	COC complete and signed (performed during SCUR review) Yes ☐ No ☐
Shipping custody documentation	Yes ☐ No ☐	Custody Seals applied to sample shipment cooler (performed during SCUR review) Yes ☐ No ☐ Custody Seals intact (performed during SCUR review) Yes ☐ No ☐
Traceable sample designation number	Yes ☐ No ☐	Sample IDs in Logbook match COC? Yes ☐ No ☐
Field logbook(s), custody records in secure repository	Yes ☐ No ☐	All notes are complete in a PDF Yes ☐ No ☐ Secure repository under RMAP protocols
Completed field forms	Yes ☐ No ☐	Are field forms, complete, legible, and signed? Yes ☐ No ☐

5. Authorization of Field Documentation Screening Review

Report prepared by: NAME, Staff Geoscientist
Report reviewed by: NAME, Senior Geoscientist
Report approved by: NAME, Senior Quality Assurance Chemist
Report approved by: NAME, Technical Director of Chemistry/Principal
Date: DATE

SECTION 2 ENFORCEMENT/SCREENING DEFINITIONS

E Enforcement quality. No qualifiers, U qualifier or J qualifier (see note below) and meets Level A and B criteria.

S Screening quality. J or UJ qualifier and/or meets only Level A criteria.

R Unusable. R qualifier and/or does not meet Level A or B requirements.

Enforcement/Screening Designation

	Meets Level A and B	Meets Level A	Does not meet Level A or B
No qualifier, A, U, or laboratory results reported between the MDL and RL with a J qualifier	E	S	R
J, J+, J-, or UJ	S	S	R
R	R	R	R

Note: It is appropriate to note that sample results qualified as estimated “J” by the laboratory because the reported result is between the MDL and RL, values are considered enforcement data if no other qualifiers were required during validation.

SECTION 3

CONSULTANT FIELD DATA SUPPORT DOCUMENTATION

ATTACHMENT E

MANUFACTURER PROCEDURES

ATTACHMENT E-1

HIGH VOLUME SMALL SURFACE SAMPLER

(HVS3) OPERATION MANUAL

CS_3, INC.

HIGH VOLUME SMALL SURFACE SAMPLER
HVS3

OPERATION MANUAL

SERIAL NUMBER
2004-39
DEC. 27, 2004

****

WARNING

Before use of this sampling equipment read the instruction manual and note the hazard warnings in the manual and on the equipment. CS_3, Inc. hereby disclaims all liability for use of the equipment unless prior written approval of the use or installation design is given. CS_3, Inc. does not warrant the accuracy or operation of the equipment and hereby disclaims all express warranties and implied warranties of merchantability or fitness for purpose.
****

TABLE OF CONTENTS

1.0 INTRODUCTION AND APPLICATIONS

1.1 Background

The purpose of the High Volume Small Surface Sampler (HVS3) is to collect representative samples of surface dust which can then be analyzed for lead, pesticides, and other toxic contaminants.

Why do we need to study surface dust? Dust can act as a concentrator of pollutants, collecting them from the environment on the dust surfaces. Even when pollutants are at insignificant levels in the air they may be at much higher levels on dust. This has been especially demonstrated with house dust. House dust has been shown to be a significant source of toxic contaminants, particularly for very young children. The quantity of lead in each square meter of carpet appears to be the best single predictor of a toddler's blood lead level. Young children ingest the toxic contaminants in floor dust through frequent hand-to-mouth contact.

Where do the lead, pesticides, and toxic other contaminants in house dust come from? Lead in house dust can come from leaded house paint, which was used in interior paints in many parts of the U.S.A. and other countries as late as the early 1970s. Peeling paint inside a house adds to the lead in house dust. It wears off the outside of the house and is then carried in on the soles of shoes. Lead from motor vehicle exhaust is found in dust and can be tracked into the house. Lead from the work place can be brought home on shoes or work clothes. Similarly, pesticides and other chemicals may be brought in from the yard or garden on shoes and clothing. It may also be applied inside the home. Older pesticides such as DDT have been found in dust even when they can't be detected in the air.

1.2 Applications

The HVS3 can be used to collect surface dust for the study of pollutant source and migration paths for total exposure assessment. It can be used on roadways to determine the silt loading data required to calculate fugitive dust emission factors. The HVS3 can be used on exposed soils to determine the possible sources of pollutants in ambient samples for source apportionment studies.

1.3 Safety

The HVS3 can be used to collect samples where hazardous materials may be present in the soil. However, this manual does not address the safety problems associated with such use. It is the responsibility of each user to determine appropriate health and safety practices prior to use.

1.4 References

Development of a High Volume Surface Sampler for Pesticides in Floor Dust. U.S. EPA (EPA 600/SA-88/036, PB 89-124630/-AS).

Development of a High Volume Small Surface Sampler for Pesticides and Toxics in House Dust, draft final report, RTI Proj. No. 171-01, EPA Work Assignment N. 11-71.

ASTM Standards:

D 422-63 Particle-size Analysis cf Soils F 6-8-79 Carpet-embedded Dirt Removal Effectiveness of Household Vacuum Cleaners.

D 5438-94 Standard Practice for Collection of Floor Dust for Chemical Analysis.

2.0 DESCRIPTION OF THE HVS3

2.1 General Description

An illustration of the HVS3 is provided in Figure 2-1. A schematic diagram of the air flow through the HVS3 is provided in Figure 2-2.

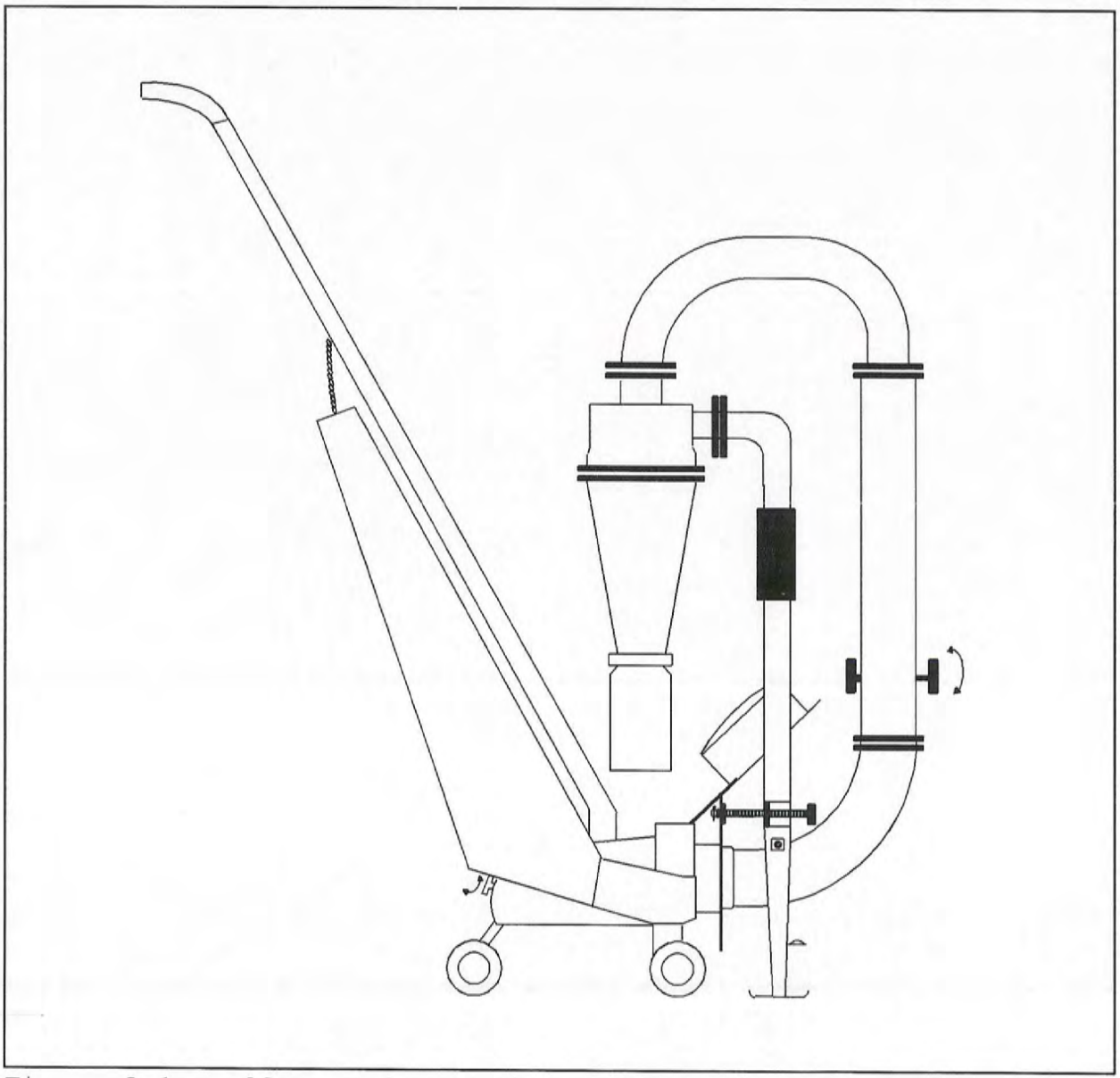

Figure 2-1. Illustration of the High Volume Small Surface Sampler (HVS3).

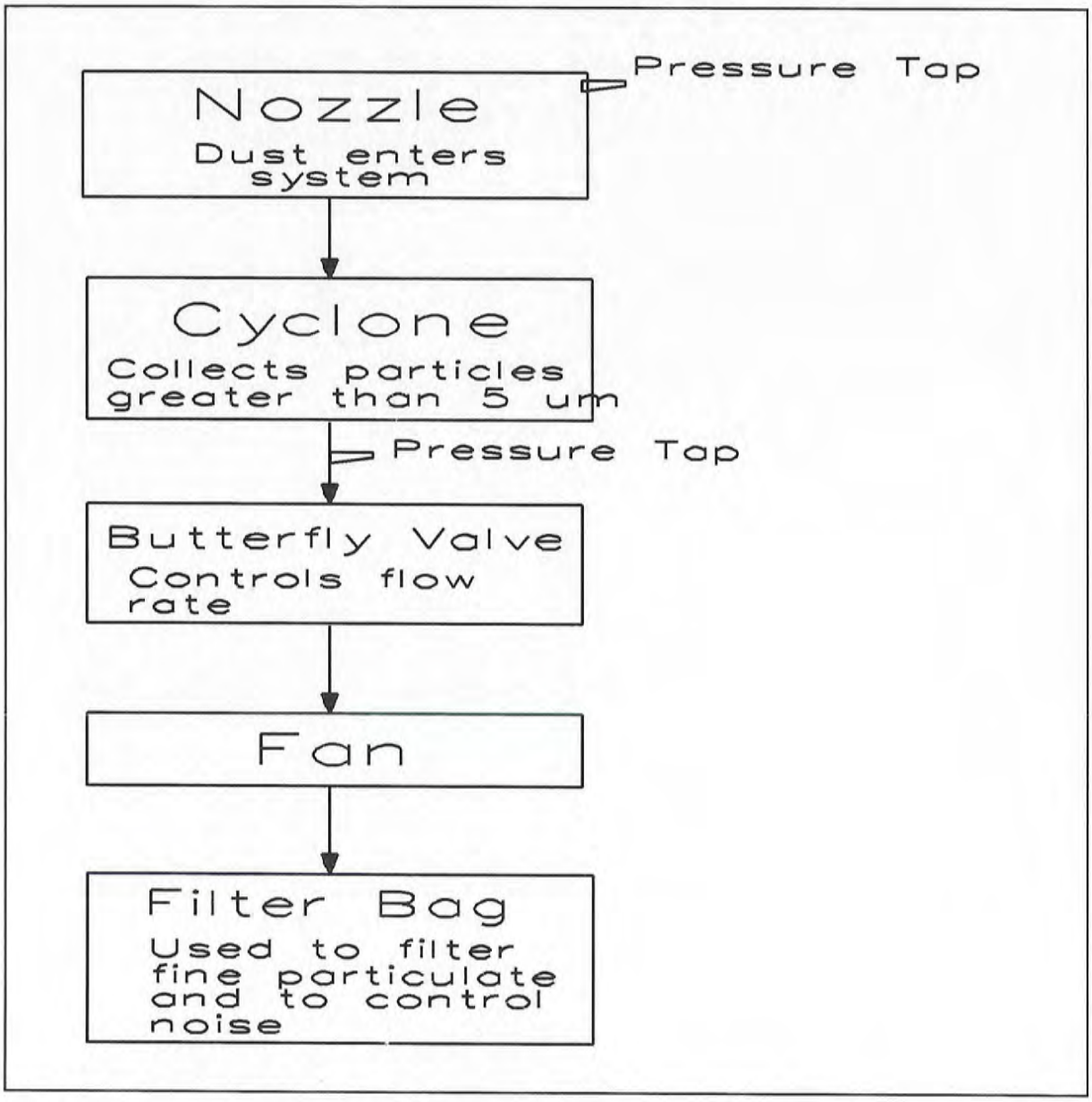

Figure 2-2. Schematic diagram of air flow through HVS3.

The surface dust enters the system through the nozzle. The nozzle is specially designed to move across a floor with little resistance, while still maintaining a sufficient seal to collect a sample. The dust then travels up to the cyclone, which collects the majority of the particles greater than 5μm in diameter. The collected particles in the cyclone catch bottle are retrieved by removing the catch bottle. The bottle can be capped for storage of the sample without transferring it to another container. Generally, the sample will be sieved to remove the larger particles and only the particles that are less than 150μm will be sent in for analysis.

2.2 Principles of Operation

The dust in the carpet is swept into the nozzle by the high velocity air being drawn through the carpet. The recommended pressure drop and flow rate will be sufficient to generate the air velocity required to lift the dust particles in the air stream.

The cyclone relies on centrifugal force to collect the particles. The larger particles move to the outside wall while the smallest particles follow the air stream to the center and out the exhaust tube. The larger particles slide down the walls and into the catch bottle at the bottom of the cyclone. On average, the cyclone collects 99% of the house dust picked up by the nozzle. Any dust that is not collected moves through the fan and is collected by the vacuum cleaner bag.

2.3 Equipment Specifications

The vacuum fan of the HVS3 is model 1028Z. Its motor has the following capabilities:

Max. Amps	10.0
Max. Watts:	950
RPM:	12100

The vacuum fan can maintain a flow rate of greater than 20 cfm (.566 cubic meters/min.).

The cut-point of the cyclone is calculated to be less than 5μm at 50% efficiency at 20 cfm (.566 cubic meters/min.).

The HVS3 sampling train is made from aluminum, and has some Silicon or Teflon tubing and gaskets. The catch bottle is PE (Polyethylene) or FEP (Teflon).

Pesticide grade solvents should be used to collect and analyze samples when pesticides are measured. Use of materials other than aluminum, stainless steel, Teflon or glass for handling chemicals or sampling has been associated with poor results in collecting pesticide samples.

3.0 ASSEMBLY

3.1 Parts List

A parts list for the HVS3 is given in the table on the following page. A figure showing the location of each part on an assembled HVS3 is provided in Figure 3-1.

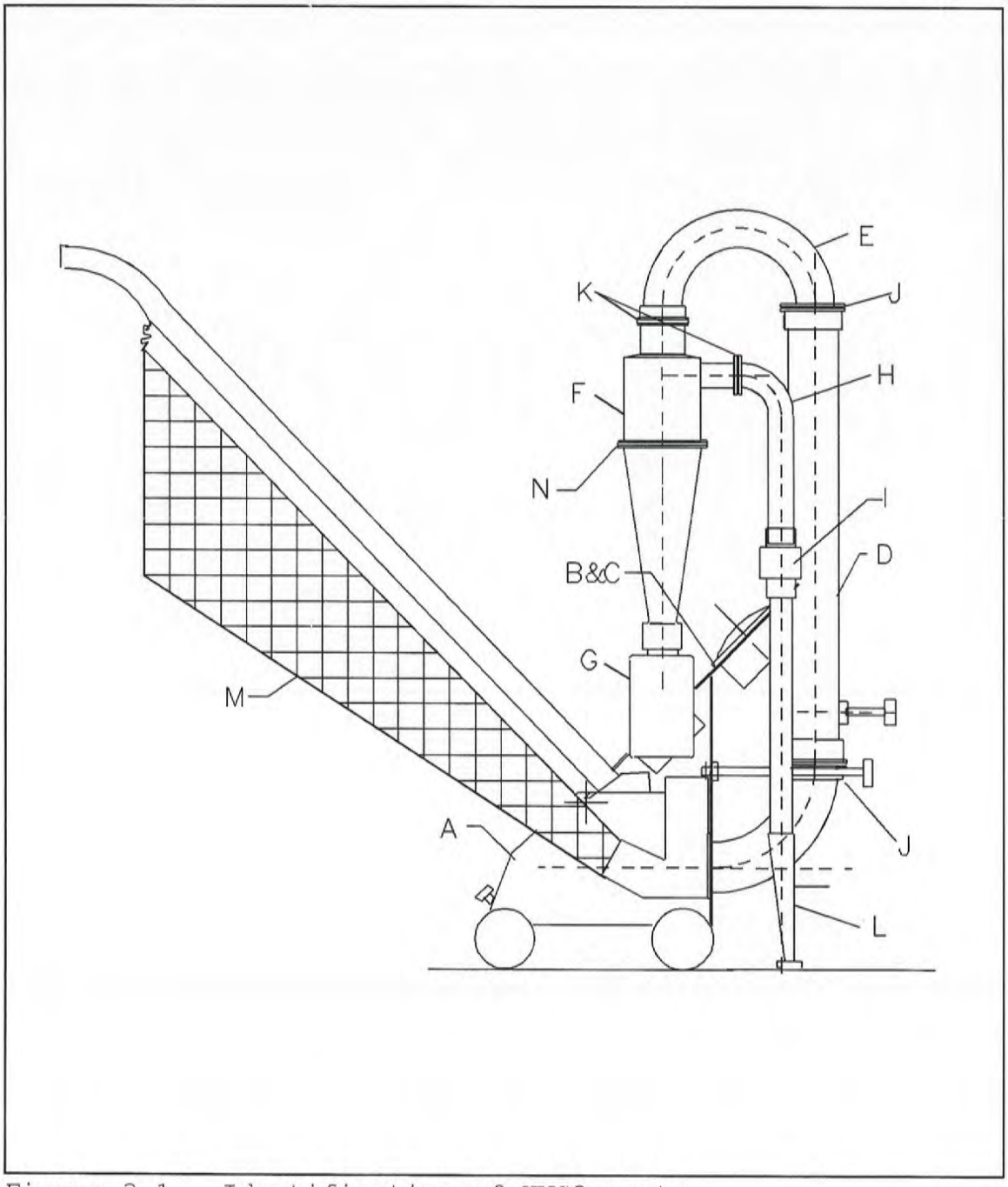

Figure 3-1. Identification of HVS3 parts.

HVS3 Parts Description Table

Part #	Qty.	Description
A	1	Model 1020D Vacuum Platform
B	1	Mounting Plate with Magnehelic mount
C	2	[1]Magnehelic gages, 0-15" & 0-10"
D	1	Control valve tube
E	1	U-Tube
F	1	3"diameter Aluminum Cyclone
G	1	P.E. or (F.E.P.) Catch Bottle
H	1	Cyclone Inlet Elbow
I	1	Tygon or (F.E.P) Flex Joint
J	2	2" clamps with gaskets
K	2	1½" clamps with gaskets
L	1	Suction Nozzle with level
M	1	Vacuum Filter Bag
N	1	3" clamp with gasket

3.2 Pre-Assembly Inspection and Cleaning

When the HVS3 is initially received, the unit will be partially assembled. Read this entire manual before proceeding with pre-assembly. It is necessary for the parts from the cyclone to the nozzle to be disassembled and cleaned with a mild detergent, rinsed with distilled water and allowed to air dry.

3.3 Calibration

A full flow calibration of the cyclone should be done a minimum of once a year. It should be calibrated with a laminar flow element, spirometer with a minimum capacity of 10 cubic feet (0.3 cubic meters), or a Roots meter. The pressure drop through the cyclone will determine the gas flow volume. The unit has been tested at our laboratory and the settings in this manual represent the proper gas volume for testing. These can change over time with wear on the components.

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[1] Magnehelic is a registered trademark of Dwyer Instruments, Inc.

3.4 Assembly (A section at the back of this manual has pictures of the assembly process).

To assemble the HVS3, follow these steps:

1. The vacuum platform and the mounting plate have been sealed with silicone rubber. Do not remove the plate from the vacuum housing.

2. Position the 2"ϕ control tube onto the 2"ϕ elbow and clamp it with a 2" clamp and gasket.

3. Position the 1½"ϕ U-tube and clamp it loosely to the vertical control tube.

4. Position the nozzle on the mounting plate. A small clip pin and washer are positioned behind the mounting plate with the plastic washer and wingnut on the front side. (Be sure that you have noted the arrangement prior to disassembling the unit.)

5. Line up the nozzle 1"ϕ elbow with the inlet of the cyclone. If the position of the U-tube and nozzle looks good, clamp the cyclone in place and tighten the adjusting screws on the tri-clamps to seal and hold the cyclone in position.

6. Check the position and adjustability of the nozzle. If the arrangement is satisfactory, connect the small gage tubing to the proper fittings.

7. Attach the catch bottle to the bottom of the cyclone.

4.0 EQUIPMENT PREPARATION

4.1 Pre-sampling Checklist

This is a useful checklist of things that need to be taken care of prior to going into the field for sampling:

Weighing scale for samples, 0.1 mg - 1000 g.
Stopwatch or wrist chronometer
Two measuring tapes
Masking tape for outlining sections for sampling
Marking pen
Extra cyclone catch bottles and caps
Manila envelope for leak check
Thermometer
Brush for cleaning and cleaning agent
Relative humidity meter
Screwdriver
HVS3 sampling train

If sampling in a residence contact the homeowner or appropriate person to explain the purpose of the test, obtain informed consent, and set a date for the test. Request home occupants to refrain from vacuuming so there will be a 7-day interval between the test and the most recent vacuuming of the area to be sampled.

The amount of sample that will need to be collected depends on the type of laboratory analysis to be performed. Consult with the laboratory which will do the analysis to determine the minimum amount of sample required for the anticipated concentrations of the pollutant of concern.

Place a label on the cleaned catch bottle and record a tare weight (with lid on).

It is advisable to assemble the HVS3 prior to any field sampling in order to carry out a leak check and find any damaged pieces. Prior to any sampling the zero should be adjusted on the Magnehelic gages.

4.2 Leak Check

Place a thick manila envelope or file folder underneath the nozzle to seal off the nozzle. Turn on the HVS3 with the switch located at the top of the handle. The flow Magnehelic gage should read between 0-0.02 inches of water. Use a 0-1.0 inch Magnehelic gage if a good reading can not be achieved with the flow Magnehelic gage.

If the gage reads more than 0.02 inches of water, check that all connections of gage tubing are correct.

If all tubing is connected properly and the flow through the system still exceeds 0.02 inches of water it is necessary to check all gaskets and tightness of clamps, catch bottle and material covering the nozzle.

5.0 SAMPLING

5.1 Pre-test Survey

Just prior to testing complete a data form recording all the information requested and sketch the area to be sampled. See Fig 5-1.

Select a sampling area according to the established protocol for your sampling campaign. This should be determined prior to testing.

Place two measuring tapes on the rug to be sampled, parallel to each other and on either side of the main traffic path through the sample area. The tapes should have heavy black marks every 75 mm (3"), be between 0.5 (20") to 1.5 meters (4'-11") apart, and be extended as far as space will permit. They should be taped to the rug every 30 cm (12") with masking tape. It is recommended that the sampling area be at least one meter from any outside door to increase the representative nature of the sample. When sampling bare floors use masking tape to lay out as large a sampling area as possible. See Figure 5-2 for example of sampling layout.

5.2 Setting the Nozzle Pressure Drop

Clean the plastic wheels and nozzle lip with a [2]Kim-wipe before placing the sampler on the test area. Place the sampler in the corner of the sampling area at the lower left position. Adjust the flow rate and pressure drop according to the type of surface to be tested. The two factors that affect the efficiency of the sampling system are the flow rate and pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and the distance between the surface and the nozzle. The nozzle position is regulated by the height control knob on the back of the HVS3 and the nozzle level adjustment knob on the front side of the nozzle. The flow rate is regulated by the use of a butterfly valve located on the down stream side of the cyclone on the control tube. The flow is measured by the pressure drop across the cyclone. The higher the flow the higher the pressure drop. The nozzle position must be adjustable in height to regulate the complete system. See Figure 5-3 for illustration of the nozzle height, level control and butterfly valve.

To set the HVS3 on level loop carpet adjust the height of the nozzle until the bubble level is centered. If you get to a point where the nozzle level bubble is not centered but the HVS3 is close to the position required use the leveling knob on the nozzle. Once it is level, set the flow rate with the butterfly valve. Check the flow rate on the flow Magnehelic gage. Tip the HVS3 forward for a few seconds so the carpet will seal to the nozzle. The flow should be set so the Magnehelic gage reads 5 inches. Next read the pressure drop across the nozzle. The reading should be approximately 10 inches. If it does not, recheck the flow and/or check that the nozzle is still level.

To set the HVS3 for use on plush carpet, first read the pressure drop across the nozzle. Set the pressure drop at approximately 9 inches on the nozzle gage. This is done by using the height adjustment knob and the level knob to keep the nozzle level. Then check the flow rate. Using the butterfly valve, set the flow rate for approximately 8 inches. Then check the pressure drop across the nozzle again. You will notice that it has changed from 9 inches. This is due to the increased flow rate which increased the pressure drop across the nozzle and vice versa. Set the nozzle pressure drop to 9 inches again using the height adjustment. Then check the flow rate again. You will probably need to make a few small adjustments three or four times until it is set right. It need not be exact. The flow rate should be between 7 to 8 inches and the nozzle pressure drop can range from 9 to 9.5 inches.

If the correct pressure drop cannot be reached or the nozzle leveled on either type of carpet, you will need to change the position of the nozzle by loosening one of the clamps on the 1"ϕ tube and adjusting the nozzle to the desired position.

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[2] Kim-wipe is a registered trademark of Kimberly-Clark

HIGH VOLUME SMALL SURFACE SAMPLER DATA SHEET

Operator_____ Date_____ Sample Ident. #___

Sampling site_____

Type of Surface: Rug___ Hardwood floor___ Other_____

Type of Rug: Plush____ Level loop____ Flat____ Multilevel_____

Shag____

Type of Vacuum: Upright___ Canister___ Other_____

Last Vacuumed_____ Temp._____ Humidity_____%

Comments_____

Location of Area Sampled:

Leak Check: Yes___ No___ 10 sec. cleaning at end: Yes___ No___

Total Sample Time: Min._____ sec._____

Flow Rate_____" Nozzle Press. Drop_____"

Bottle final Wt._____grams Tare Wt._____grams Net Wt._____grams

Pan & Sample: ____grams Pan Tar Wt. ____grams Net Wt.____grams

Total Dust: _____grams/m^2
Fine Dust: _____grams/m^2
Cyclone Sample #:_____
Lab Sample #:_____

Figure 5-1. Recommended data recording form

The HVS3 will operate best when the handle is at an angle that allows for smooth movement . The lever at the bottom of the handle should be placed in the notch that sets the handle to a 45° angle with the floor. With handle at this angle and a firm pressure, the HVS3 will be much less likely to nosedive.

Begin sampling by moving the nozzle between the ends of the two tapes. The sampler is moved back and forth four (4) times on the 75 mm (3"inch) wide first strip, moving the sampler at approximately .5 meters (two feet) per second. Move in a straight line between the numbers on the tape. After four double passes gradually angle over to the second strip on the next pass and repeat four (4) double passes. Repeat procedure until all strips have been sampled or you have enough sample.

After sampling approximately a 0.5 square meter, check the amount of collected material in the bottom of the catch bottle. 6 mm (¼") of dust is about 6 to 8 grams of material. If there is less than 6 mm (¼") of dust, sample an additional 0.5 square meter area next to the area already sampled. Hair and fluff such as carpet fibers should be excluded from the sample when determining if you have enough sample catch. Keep sampling in the area laid out until you have enough sample catch or you have sampled all this area. If you do not have enough sample layout another area and sample it. Switch off the HVS3 when you have enough sample. The catch bottle can be removed, labeled, and capped for storage and analysis. Record the dimensions of the sampled area on the data sheet.

If the area to be sampled is very dirty or has not been cleaned frequently, care must be taken to avoid filling up the cyclone catch bottle on the first sample area. If you suspect this will be the case start with a 0.25 square meter sampling area. Then take a second and a third area as before until the catch bottle is 25% full to get a representative sample.

Adjust the flow rate and nozzle pressure drop according to the following chart:

Carpet	Flowrate cfm (in. H2O)	Nozzle Press. Drop, in. H_2O
Plush	20 cfm, 8 inches H_2O	9 inches H_0O
Level Loop	16 cfm, 5 inches H_2O	10 inches H_2O

Use the same flow rate and pressure drop on multilevel and shag rugs as is used for plush rugs.

5.4 Sampler Cleaning

After the sample bottle is removed, open the butterfly to maximum flow, tip the sampling train back so that the nozzle is 2 inches off the surface, and switch on the HVS3. Place a hand covered by a rubber glove over the bottom of the cyclone and alternate closing and opening the cyclone for 10 seconds to free any loose material adhering to the walls of cyclone and tubing. It is not necessary to catch this small amount of material.

Remove the sampler to a well-ventilated cleaning area that is free from dust. See appendix for cleaning tips. Remove the cyclone cone, bellows connector, and elbow at the top of the nozzle tubing from the sampler. Clean the nozzle, bellows connector, elbow and cyclone with pesticide grade methanol. Use rubber gloves. Clean the parts separately and rotate each item so that all internal surfaces are washed and brushed three times. Alternate between applying methanol with a 500 ml squeeze bottle and brushing. There should be no visible trace of dust when you have finished.

A 12" by 18" baking pan can be used to catch the methanol. This wash can be analyzed at the discretion of the operator. The total amount of dust removed in the air and wet cleaning is usually less than one percent of the collected dust. The air and wet cleaning is done to prevent contamination passing from on sample to another. The small amount of methanol in the pan may be disposed of by setting it outside and letting it evaporate.

The pieces of the sampler can be allowed to air dry for 30 minutes at room temperature or reassembled immediately and dried by drawing air through the sampler for 5 minutes.

5.5 Air Cleaning for Lead Samples

If lead is the only pollutant to be measured follow the procedure as noted above through the first paragraph of 5.4. Before the sampler is taken apart brush the nozzle and bellows four times while a gloved hand is held over the bottom of the cyclone and the HVS3 is run at maximum flow. Place an envelope over the nozzle to seal it and brush the inside of the cyclone while the sampler is being run with maximum air flow. Take the nozzle, bellows connector, and upper nozzle elbow off the machine and brush until there is no trace of dust showing.

On every fifth sample do a methanol cleaning as described in 5.4.

The TLV for methanol is 200 ppm for an 8 hour exposure (and 250 ppm exposure for a 15 minute exposure). There is very little odor. Its low vapor pressure makes it less flammable and a better cleaning agent for dust than other solvents. Since skin is a route of exposure rubber gloves should be worn during cleaning. Methanol can be shipped by Federal Express air in less than quart amounts and by surface transportation in gallon amounts. If adequate ventilation is not available in the cleaning area a face mask with organic vapor cartridges should be worn.

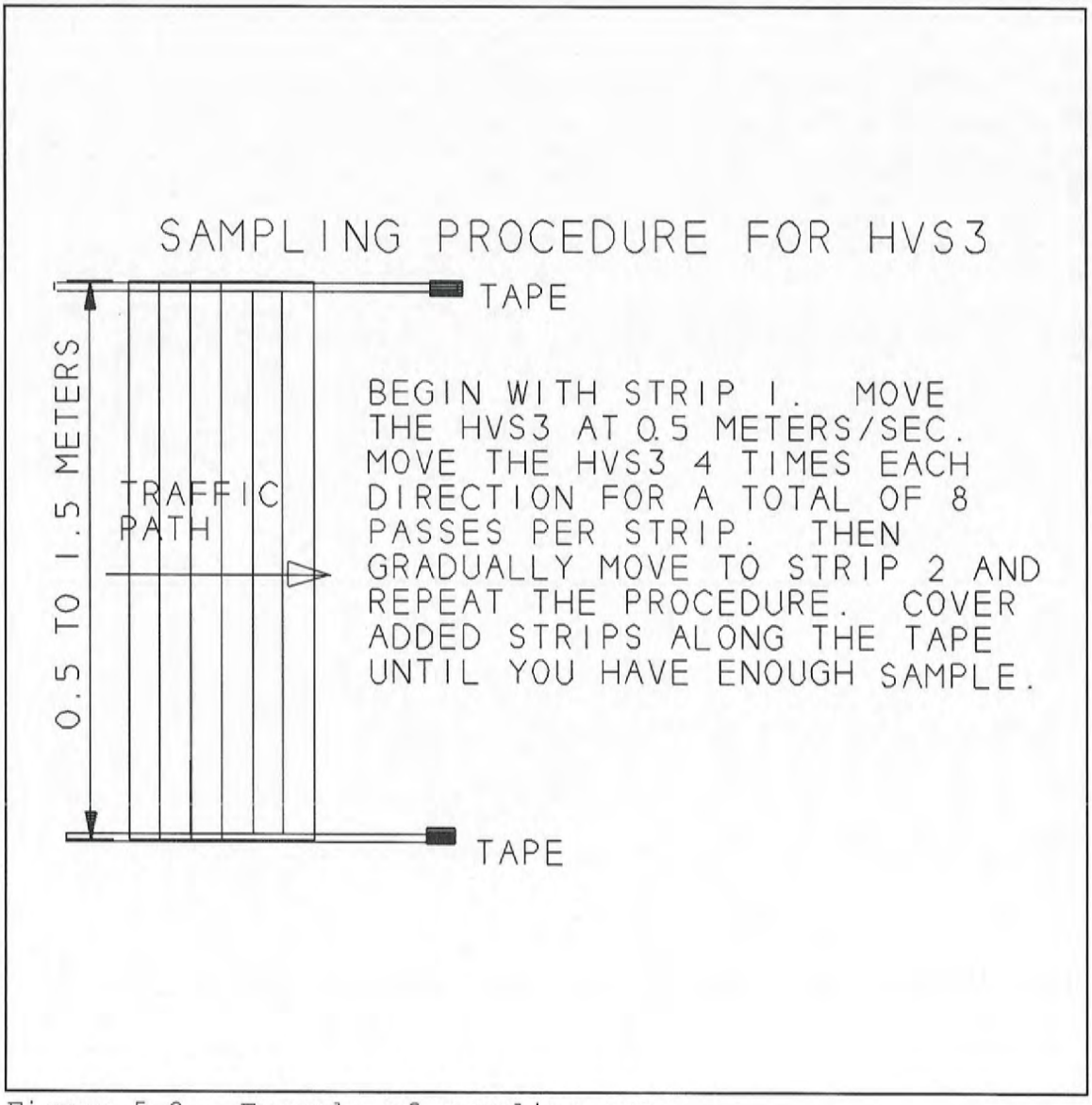


Figure 5-2. Example of sampling area.

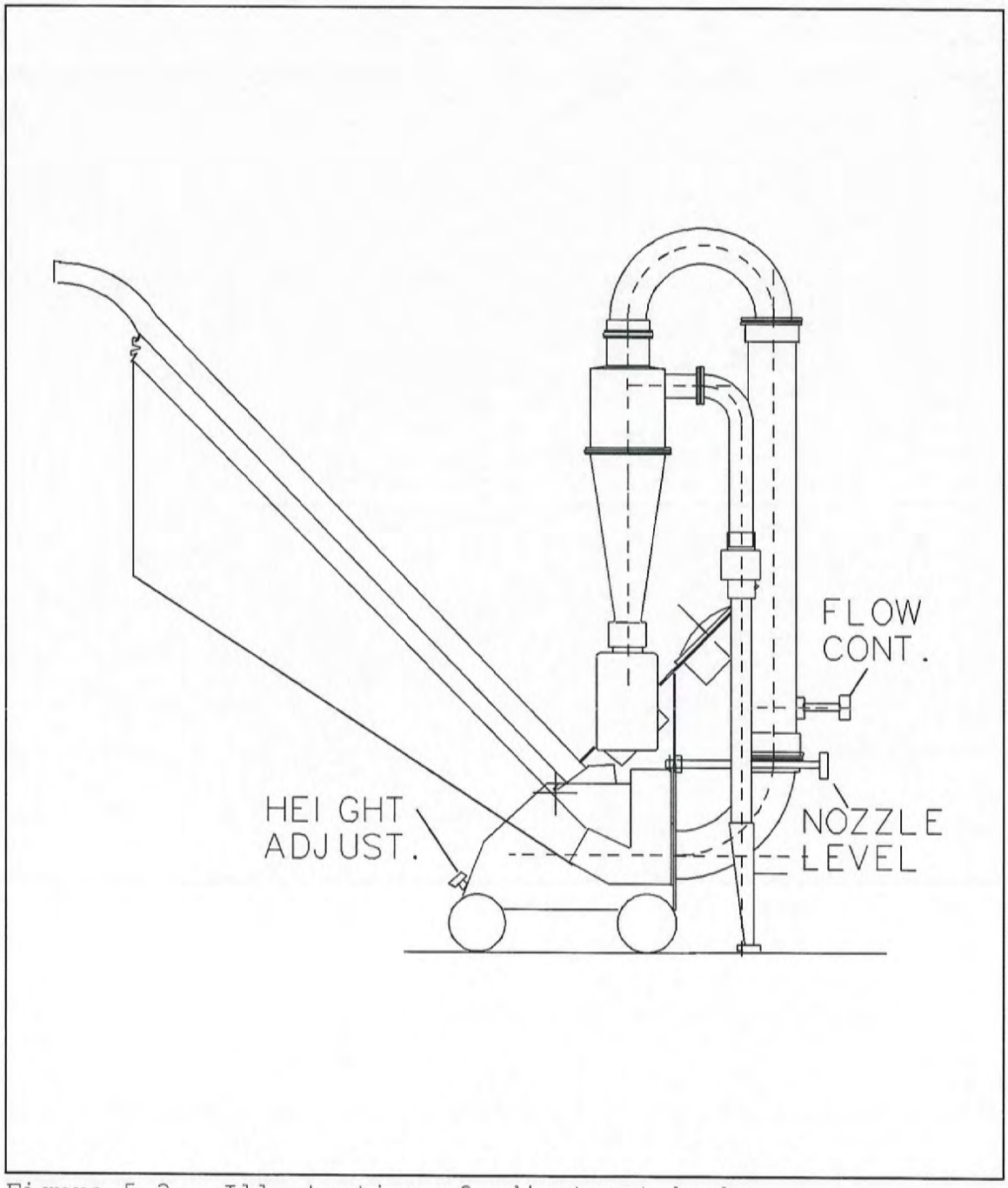


Figure 5-3. Illustration of adjustment knobs.

6.0 SAMPLE ANALYSIS

6.1 Sieving the Sample

The samples should be sieved for five minutes in a shaker using the ASTM Method D 422-63, with a 100 mesh screen above the pan to determine the weight of fine dust below 150 microns. Weigh to the nearest 0.1 gram.

6.2 Saving the Sample

To prepare your samples for analysis, clean a piece of aluminum foil with a pesticide grade methanol solution. When it has dried, pour the fine dust from the cyclone catch bottle onto the middle of the foil. Carefully fold the aluminum foil into a small package, keeping the dust in the middle. Clean a glass jar with the same cleaning solution and dry it. Place the foil pouch in the jar.

Cover the jar opening with another piece of clean foil and put the lid on the jar. Label the jar for reference. Seal the seam of the lid to the jar with Teflon tape. If the samples are being analyzed for pesticides or polycyclic aromatic hydrocarbons (PAHs), place the sieved sample in an ice chest with dry ice to keep it at approximately 0 degrees Celsius. It is now ready to be shipped off to the laboratory for analysis. Alternative cleaning solvents and methods for storage of samples, shipping and preparation for analysis may be required for some contaminants and should be prescribed in specific sample campaign protocols. The catch bottle may be used for storage and shipping.

7.0 DATA ANALYSIS

7.1 Calculations

Calculate the amount collected in the cyclone by subtracting the cyclone catch bottle tare weight from the final weight.

Calculate the fine dust by subtracting the tare weight of the pan from the final weight.

Calculate the loading for fine dust per square meter (g/m^2) for the household by dividing the final dust value by the area measured (expressed in square meters).

When the analysis results are received from the lab, it is possible to calculate the loading of lead, pesticides, or other toxics per square meter ($micrograms/m^2$) in the same way.

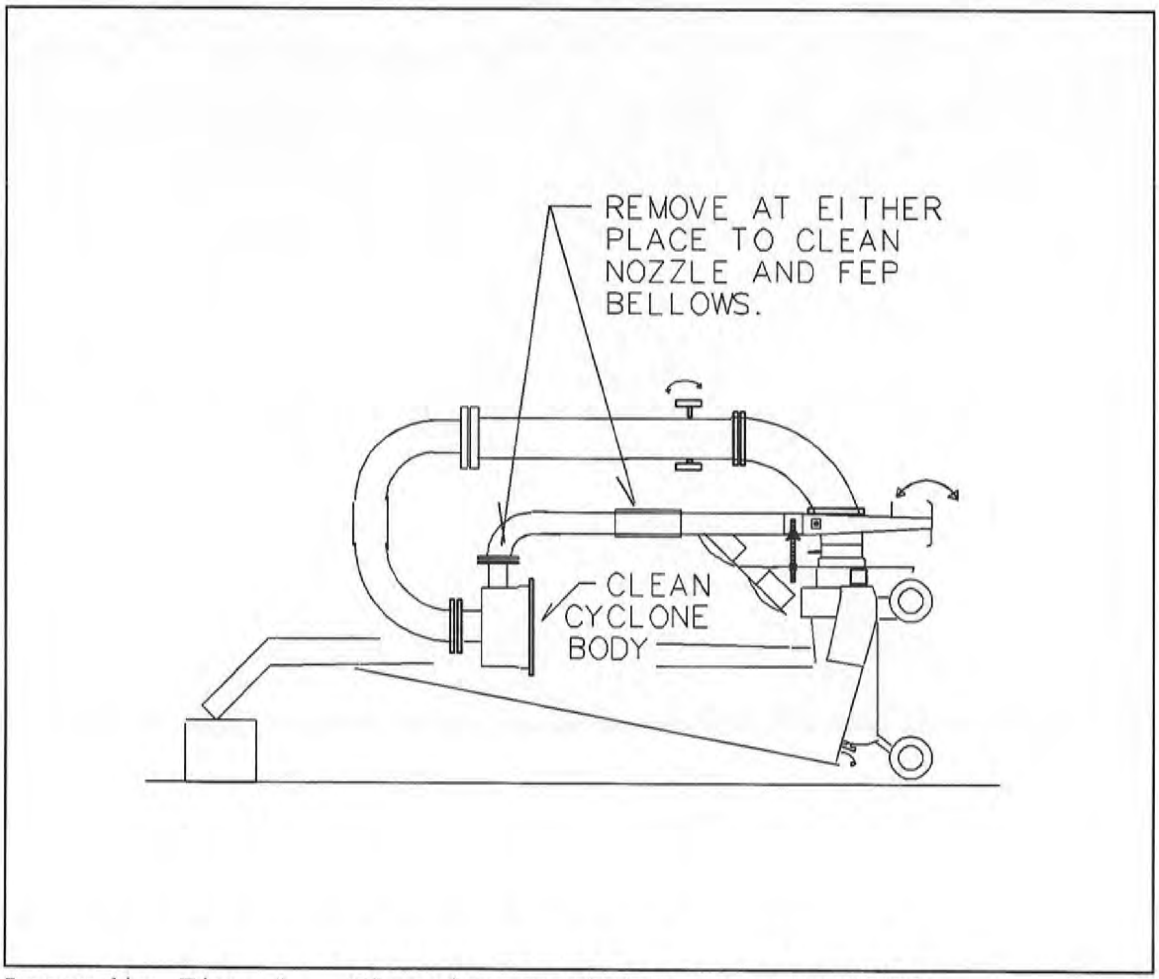
APPENDIX

CLEANING TIPS FOR THE HVS3

1. To allow for easier cleaning of the HVS3 cyclone body and cone place the sampler on a level surface such as a table or counter. Remove the 3" quick clamp holding the cone to the body and set the cone to the side for cleaning. Lay the sampler on its back so the control tube is horizontal which will allow for easy access to the cyclone body. You can disconnect the bag from the fan housing to avoid soiling it.

2. To clean the nozzle remove either the flex joint connector or remove the 1" quick connect clamp holding the cyclone inlet elbow to the cyclone body. Remove a small spring clip located on the back side of the vacuum platform mounting plate. This will allow for the complete nozzle assembly to be cleaned separated from the HVS3.

3. Reassemble the sampler after following the cleaning instructions and the system is ready to use again.



Appendix Fig. 1. Cleaning position for the HVS3.

ITEMS OF CONSIDERATION

1. When using the HVS3 the filter bag will become blinded due to the material passing the cyclone. All of the fine particulate will fill the pores of the bag and cause the suction of the vacuum to diminish. When the suction reading at the nozzle, flow or both can not be maintained then the bag should be replaced with a fresh one.

2. Over time the brushes in the motor of the vacuum system will wear. This will affect the performance of the system. If you have replaced the bag as described above and you can not attain the proper suction the motor will need servicing.

3. Other catch bottles can be used in place of the catch bottle provided with the sampler. A 250 ml wide mouth Nalgene bottle will fit the cyclone discharge, and can be purchased through many scientific supply houses.

SAMPLE CONTAINER PART NUMBERS

The standard sample container that attaches to the HVS3 & 4 cyclone has the following part numbers.

For LDPE (polyethylene) the Nalgene part number is: 2103-0008
This is a 250 ml. (8oz.) wide mouth bottle.

For FEP (fluorinated ethylene propylene) for use with
pesticide testing the Nalgene part number is. 2100-0008
This is a 250 ml. (8oz.) wide mouth bottle.

An adapter to use [3]I-Chem glass sample bottles (341-0250) is available. Contact CS_3, Inc. (1 800 910 9398) for information and pricing.

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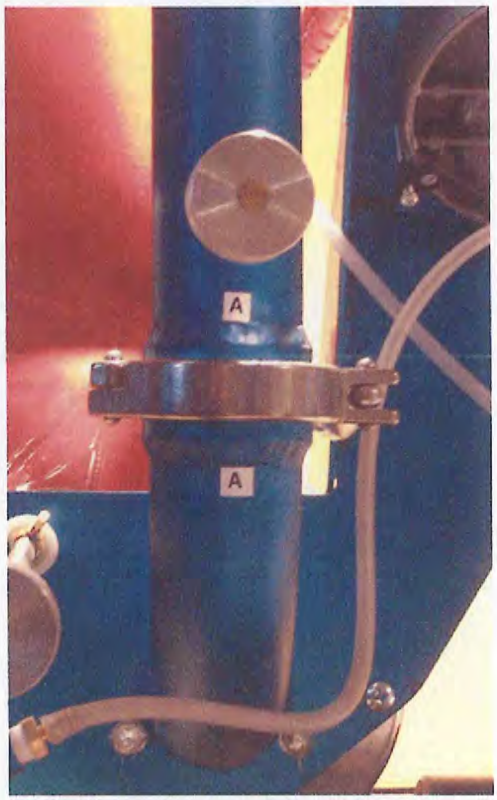
[3] I-Chem is a registered trademark of Nalgene

HVS3 ASSEMBLY

The following pictures and descriptions will allow anyone not familiar with the HVS3 sampling system to assemble the unit.

With the vacuum platform and gage plate on a table and the gage plate elbow facing you attach the control tube. It is 16" long, made of 2" diameter tubing. The picture below show the match marks "**A**" are aligned. You do not need to exact as we will later make final adjustments.

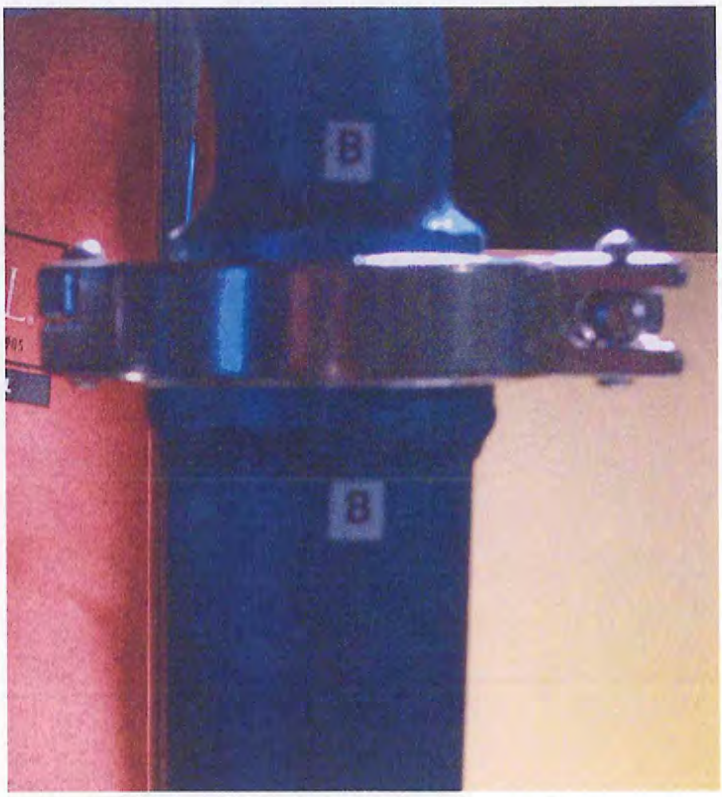


Control tube to gage plate elbow assembly

To assemble this connection place a 2.5" diameter red silicone gasket onto the end of the elbow on the gage plate. Set the control tube onto the elbow with the control knob facing away from the gage plate and at the bottom of the control tube. After the tube is in position apply the appropriate clamp to secure the joint. Tighten the wing nut on the clamp just enough to secure the clamp.

Step 2

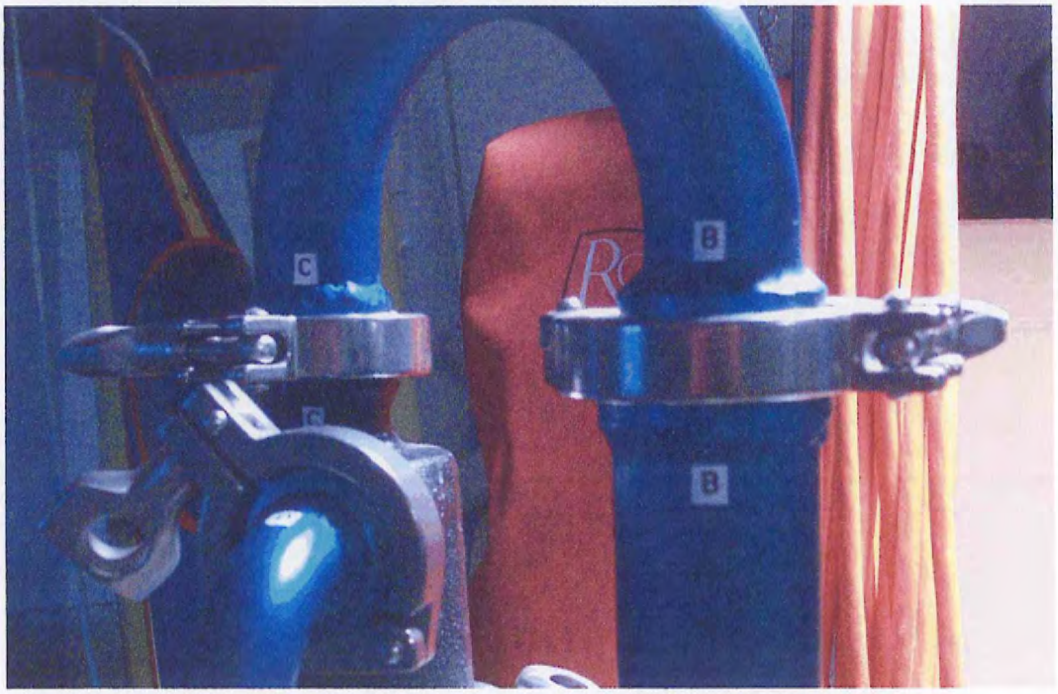
Set a 2.5" diameter red silicone gasket on top of the control tube. Pick up the 180 degree elbow (1.5" diameter) and align the match marks "**B**" as shown below.



U-tube to control tube connection.

This connection is just like the previous one. Once you have the match marks close attach the clamp and tighten the wing nut until the joint is secure.

Now proceed to attach the cyclone to the U-tube. The cyclone goes on to the other end of the 1.5" elbow just attached to the control tube. The match marks "**C**" should be aligned as shown. At this connection a 2" diameter red silicone gasket is used. This will point the inlet of the cyclone away from the sampler vacuum. Clamp the cyclone so it is just secure. The proper orientation is shown in the next picture.



U-tube to control tube and cyclone to U-tube connection.

One you have the cyclone oriented correctly place use a clamp (smaller than previously used clamps), to secure the cyclone in place.

Step 4

The nozzle, adjusting screw assembly and flex joint with cyclone inlet elbow should be assembled prior to attaching it to the cyclone inlet.

Nozzle Assembly

To attache the nozzle assembly to the cyclone inlet place a 2" diameter by 1" inside diameter gasket (red silicone for dust sampler and white Teflon for pesticides), between the inlet elbow flange and the inlet flange on the cyclone. Align the match marks "**D**" and secure with a clamp.

After the nozzle is attached to the cyclone you must insert the end of the adjusting screw into the hole on the vacuum mounting plate.

The orientation of the adjusting screw attached properly are depicted in the following pictures.

Adjusting screw mounted properly to vacuum mounting plate.

The attachment of the adjusting screw requires that a washer and spring clip be place behind the vacuum mounting plate. This secures the adjusting screw and allow for nozzle adjustment.

Adjusting screw with washer and clip mount to vacuum plate.

The HVS3 is now assembled. To adjust the position of any part just loosen the clamp at the joint that needs to be changed and rotate the part to the new position. You may have to change the next joint and so on to get the orientation correct.

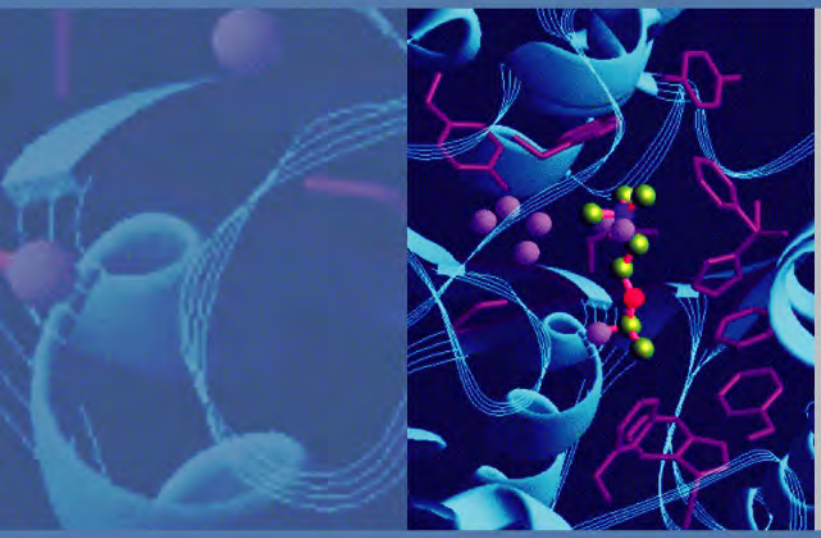
To adjust the height of the nozzle to the surface to be sampled it may be necessary to change the insertion length of the cyclone inlet elbow or nozzle into the flex joint. For most situations the flex joint assembly is correct when the vacuum height adjusting screw just allows the sampling flange on the nozzle to just touch a hard surface with the screw adjusted for maximum forward position.

Should you have questions please contact CS_3, Inc. at 208 265 8115.

ATTACHMENT E-2

THERMO FISHER SCIENTIFIC NITON XL2

ANALYZER USER'S GUIDE



Thermo Fisher Scientific Niton Analyzers

XL2 Analyzer

Version 8.0.1

User's Guide (Abridged)

Refer to NITON XL2 Resource Guide for complete information

Revision A **March 2012**

Part of Thermo Fisher Scientific

Thermo SCIENTIFIC

Release history:

For Research Use Only. Not for use in diagnostic procedures.

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Manual Overview

Warnings, Cautions, and Notes

Warnings

Warnings are extremely important recommendations, violating which may result in either injury to yourself or others, or damage to your analyzer and/or data. Warnings will always be identified as Warnings in the text, and will always be visually presented as follows:
WARNING This is a Warning.

Example Warning:

WARNING Tampering with the 5,500 ppm (Lead high) lead-in-soil standard may cause exposure to lead dust. Keep all standards out of reach of children.

Cautions

Cautions are important recommendations. Cautions will always be identified as Cautions in the text, and will always be visually presented as follows:
CAUTION This is a Caution.

Example Caution:

CAUTION Never tamper with Test Standards. They should not be used unless they are completely intact

Notes

Notes are informational asides which may help you with your analyses. Notes will always be identified as Notes in the text, and will always be visually presented as follows:
Note This is a Note.

Example Note:

Note For defensible Quality Control, keep a record of the time and precision of every calibration

Figures

Figures are illustrations used to show what something looks like. Figures will always be labelled and identified as Figures directly below the Figure itself, and will always be visually presented as follows:

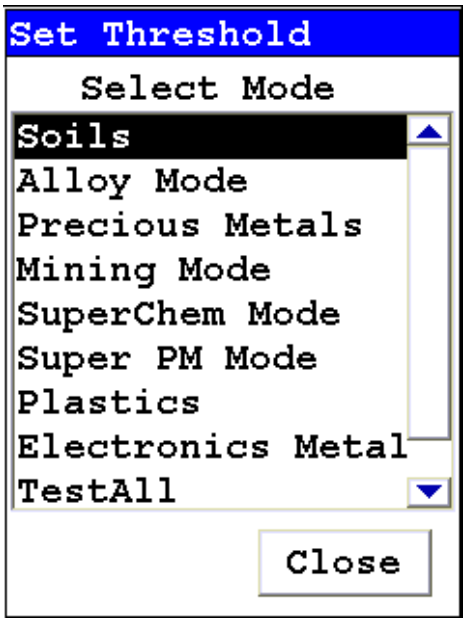


Figure 1. This is a Figure

Physical Buttons

Physical Buttons are actual buttons on the analyzer which must be pushed to activate their function. Physical Buttons will always be identified as Buttons in the text, and will always be visually presented as follows:

This is a Physical Button.

Example Physical Buttons:

On/Off/Escape Button, Clear/Enter Button, Interlock Button, and Trigger Button.

Other Hardware

Other Hardware refers to any physical part of the analyzer which performs a necessary function. Other Hardware will always be visually presented as follows:

This is an example of Other Hardware.

Example Other Hardware:

Battery, Touch Screen Display, Measurement Window, and USB Cable

Using Your Analyzer

This section discusses the basics of using your analyzer, no matter the specific type of analysis you wish to perform. First we go over analyzer safety, particularly radiation safety. Using an X-ray based analyzer safely is very important, and not difficult, provided you read, understand, and follow these guidelines. Secondly, we outline the startup procedure we recommend for daily use to ensure that your analyzer is performing properly and at its most efficient level.

Safely and Effectively Using Your Analyzer

CAUTION Niton analyzers are not intrinsically safe analyzers. All pertinent Hot Work procedures should be followed in areas of concern.

WARNING Always treat radiation with respect. Do not hold your analyzer near the measurement window during testing. Never point your analyzer at yourself or anyone else when the shutter is open.

Radiation and General Safety

This section covers topics related to radiation safety and general safety when using a Thermo Scientific Niton XL2 analyzer. At a minimum all operators of the analyzer should be familiar with the instructions provided in this chapter in order to handle the analyzer in a safe manner. In addition to reading the information presented on the following pages, Thermo Fisher Scientific recommends that instrument users participate in a radiation safety and operational training class.

Radiation Protection Basics

The Niton Model XL2 analyzer contains an x-ray tube which emits radiation only when the user turns the x-ray tube on. When the x-ray tube is on and the shutter is open, as during a measurement, the analyzer emits a directed radiation beam - see Figures 1-1 and 1-2. Reasonable effort should be made to maintain exposures to radiation as far below dose limits as is practical. This is known as the ALARA (As Low as Reasonably Achievable) principle. For any given source of radiation, three factors will help minimize your radiation exposure: Time, Distance, and Shielding.

Time

The longer you are exposed to a source of radiation the longer the radiation is able to interact in your body and the greater the dose you receive. Dose increases in direct proportion to length of exposure.

Distance

The closer you are to a source of radiation, the more radiation strikes you. Based on geometry alone, dose increases and decreases with an inverse-squared relation to your distance from the source of radiation (additional dose rate reduction comes from air attenuation). For example, the radiation dose one foot from a source is nine times greater than the dose three feet from the source. Remember to keep your hands and all body parts away from the front end of the analyzer when the shutter is open to minimize your exposure.

Shielding

Shielding is any material that is placed between you and the radiation source. The more material between you and the source, or the denser the material, the less you will be exposed to that radiation. Supplied or optional test stands are an additional source of shielding for analysis. A backscatter shield accessory is also available and may be appropriate in some applications.

Exposure to Radiation

Human dose to radiation is typically measured in rem, or in one-thousandths of a rem, called millirem (mrem), 1 rem = 1000 mrem. Another unit of dose is the Sievert (Sv), 1 Sv = 100 rem. The allowable limit for occupational exposure in the U.S (and many other countries) is 5,000 mrem/year (50 mSv/year) for deep (penetrating) dose and 50,000 mrem/year (500 mSv/year) for shallow (i.e., skin) dose or dose to extremities. Deep, shallow, and extremity exposure from a properly used Niton XL2 analyzer should be less than 200 mrem per year, (2.0 mSv per year) even if the analyzer is used as much as 2,000 hours per year, with the shutter open continuously. The only anticipated exceptions to the 200 mrem maximum annual dose are: 1) routine and frequent analysis of plastic samples without use of a test stand, backscatter shield, or similar additional protective measures, or 2) improper use where a part of the body is in the primary beam path.

Note NEVER OPERATE THE DEVICE WITH A PART OF YOUR BODY IN THE PRIMARY BEAM PATH OR WITH THE PRIMARY BEAM PATH DIRECTED AT ANYONE ELSE.

Also, consider the use of protective accessories such as a shielded test stand or backscatter shield (or equivalent) when performing routine and/or frequent analysis of any of the following:

- light materials (such as plastic, wood, or similarly low density/low atomic mass samples)
- thin samples (such as foils, circuit boards, and wires)
- samples that are smaller than the analysis window.

Shown in Table 1 are the typical background radiation doses received by the average member of the public. The radiation dose limits for radiation workers in the US are also shown in Table 2.

Table 1. Typical Radiation Doses Received (Source: NCRP 1987)

Category	Dose in mrem	Dose in mSv
Average total dose in US (annual)	360	3.6
Average worker exposure (annual)	210	2.1
Average exposure for an underground miner	400	4.0
Exposure for airline crew (1,000 hours at 35,000 ft)	500	5.0
Additional from living in Denver at 5300' (annual)	25	.25
Additional from 4 pCi/l radon in home	1,000	10.0
Typical Chest X-Ray	6	0.06
Typical Head or Neck X-Ray	20	0.2
Typical pelvis/hip x-ray	65	0.65
Typical lumbar spine x-ray	30	0.3
Typical Upper G.I. x-ray	245	2.45
Typical Barium enema x-ray	405	4.05
Typical CAT scan	110	1.10

Table 2. Annual Occupational Dose Limits for Radiation Workers (Source: Code of Federal Regulations Title 10, Part 20)

Category	Dose in mrem	Dose in mSv
Whole Body	5000	50
Pregnant Worker (during gestation period)	500	5
Eye Dose Equivalent	15,000	150
Shallow dose equivalent to the skin or any extremity or organ	50,000	500
Maximum allowable dose for the general public (annual)	100	1.0
For a Minor	500	5.0

Monitoring your radiation exposure

Individuals can be monitored for the radiation dose they receive by use of radiation dosimetry devices (dosimeters). Monitoring dose using a dosimeter can be a way of identifying improper use and at the same time demonstrating proper use. In some locations, dosimetry is required by regulations and in others it is optional. It is normally required when the user could reasonably be expected to receive in excess of 10% of the annual dose limit. Thermo Fisher Scientific recommends that you determine and obey the local regulatory requirements concerning radiation monitoring of occupational workers.

Two common types of dosimeters are whole-body badges and ring badges. Whole body badges are often attached to the user's torso (e.g., clipped to the collar, shirt pocket, or waist as appropriate). A ring badge is worn on the finger as a measure of maximum extremity dose. When worn, the specific location of the dosimeter should be that part of the body that is expected to receive the highest dose. This location will depend on how the analyzer is used and so it may not be the same for all users. Dosimetry services are offered by many companies. Two companies offering dosimetry services in the USA and much of the world are:

Company	Global Dosimetry Solutions	Landauer, Inc.
Address	2652 McGaw Avenue	2 Science Road
City and State	Irvine, CA 92614	Glenwood, IL 60425-9979
Website	www.dosimetry.com	www.landauerinc.com
Phone Number	(800) 251-3331	(800) 323-8830

Note Wearing a dosimeter badge does not protect you against radiation exposure. A dosimeter badge only measures your exposure (at the dosimeter location).

Pregnancy and Radiation Exposure

International guidance documents (e.g., ICRP Publication 60 and NCRP Publication 116*) recommend that the radiation dose to the embryo/fetus of a pregnant woman should not exceed a total of 500 mrem (10% of normal radiation worker limit) during the gestation period. While this dose limit exceeds the dose limit to a trained operator, pregnant workers may want to take special precautions to reduce their exposure to radiation. For more information see the U.S. NRC Regulatory Guide 8.13 "Instruction Concerning Prenatal Radiation Exposure" which can be found on the resource CD.

* The International Commission on Radiological Protection, ICRP, is an independent Registered Charity, established to advance for the public benefit the science of radiological protection, in particular by providing recommendations and guidance on all aspects of protection against ionizing radiation.

* The National Council on Radiation Protection and Measurements (NCRP) was chartered by the U.S. Congress in 1964 as the National Council on Radiation Protection and Measurements.

How to Use the Niton XL2 Analyzer Safely

The Niton XL2 analyzer is designed to be safe to operate provided that it is used in accordance with manufacturer's instructions. Under conditions of normal use, monitored operators seldom receive a measurable dose and have not been known to receive in excess of 10% of the annual occupational dose limits (a criteria that would require monitoring under regulation in the U.S.). In addition to proper use of the analyzer, it is recommended that you follow these precautions to ensure your safety and the safety of those around you.

Know where the beam is

The primary beam is a directed beam out of the front of the analyzer that can have high dose rates. The secondary beam, or scattered beam, has much lower dose rates.

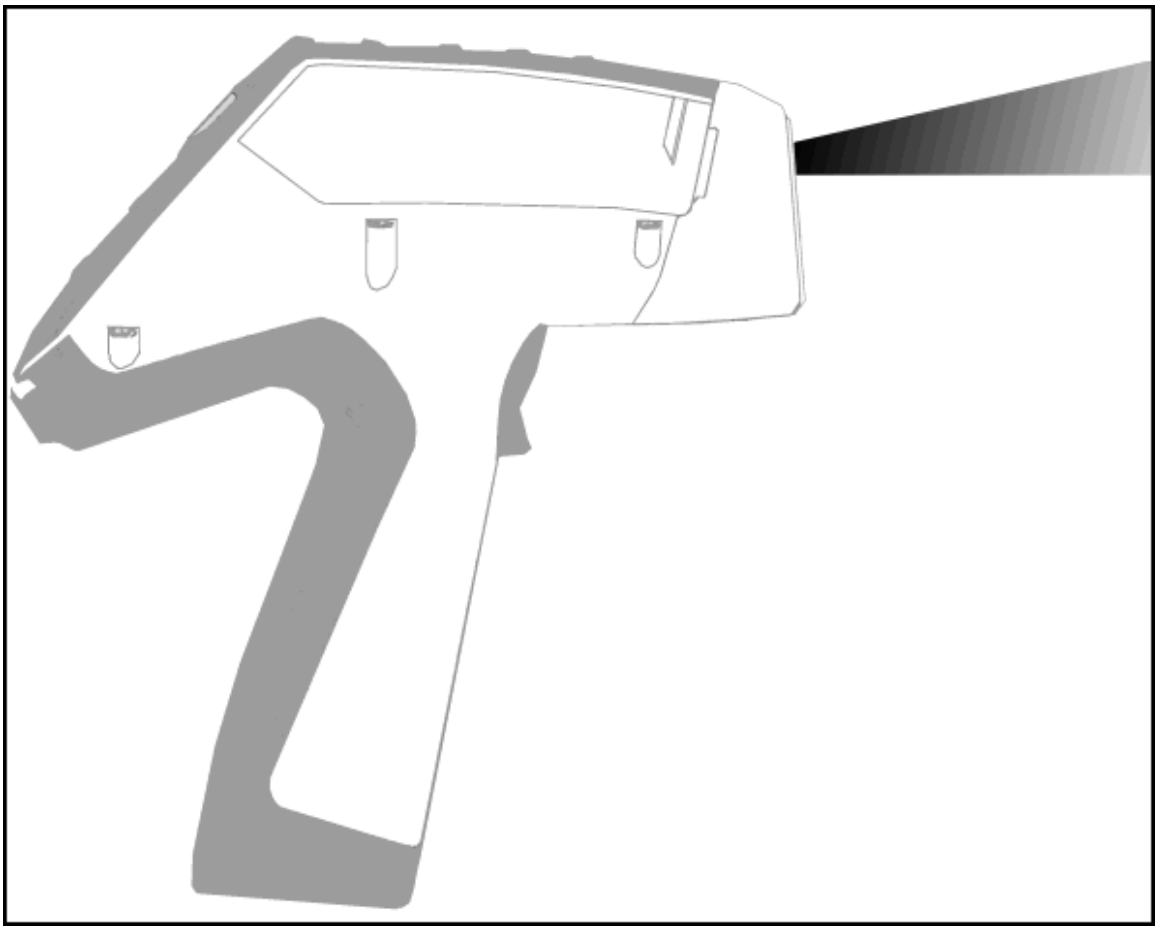


Figure 1. Primary Beam

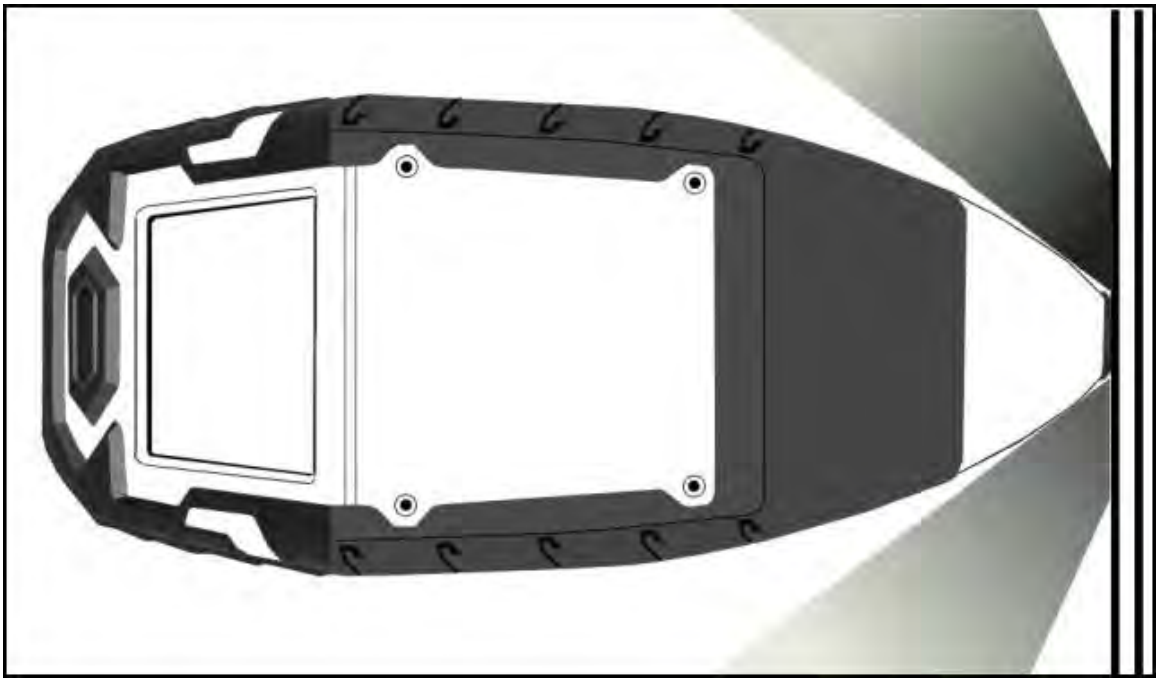


Figure 2. Secondary (Scattered) Beam

The Shutter-Open Indicator Lights

When the lights are flashing, the primary beam is on, and radiation is being emitted from the front of the analyzer.

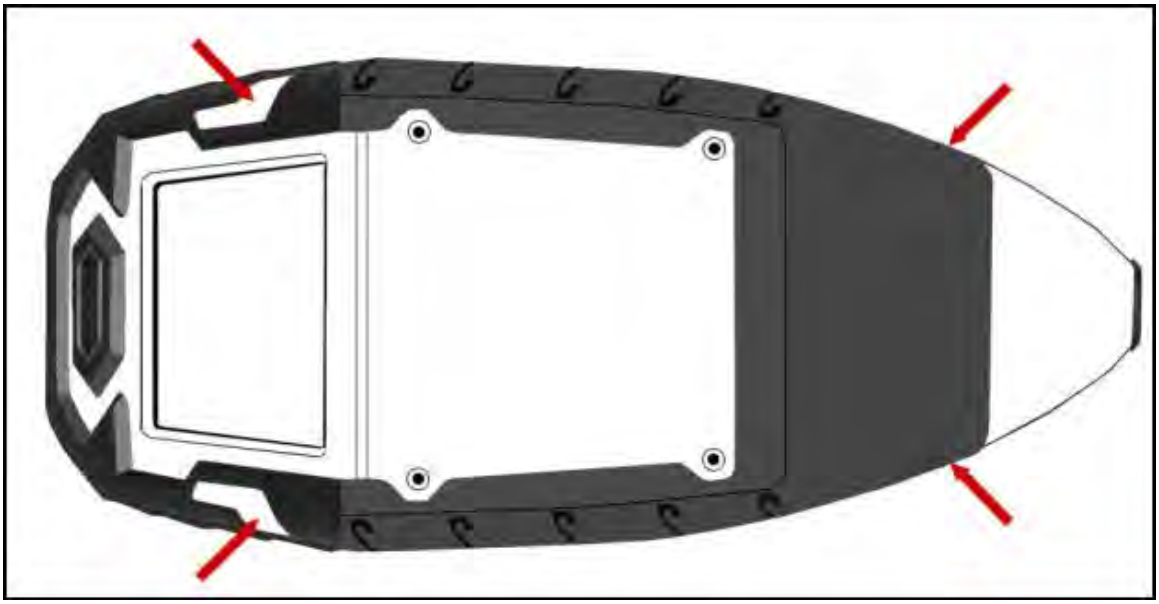


Figure 3. The X-ray Beam Indicator Lights

Handle and Use with Respect

Avoid holding the front of the analyzer when the x-ray tube is energized and the shutter is open. Never point the instrument at yourself or anyone else when the shutter is open and the x-ray tube is energized. Never look into the path of the primary beam.

Follow a Radiation Protection Program

Your organization should establish, document, and follow a Radiation Protection Program. An example of such a program can be found on the resource CD (provided with the instrument).

Take Proper Care of your Niton XL2

Keeping your analyzer maintained in good condition will help minimize the risk of accidental exposure. Mechanical malfunction of the shutter can be avoided by maintaining the measurement window, as described in the User Guide. This prevents foreign objects from entering your analyzer

Avoid Over-Exposures

Direct contact with the window could result in overexposures in the times indicated in Table 3 below.

Table 3. Potential Exposure Limit Times

Location of Dose	Limit	Time to Reach Limit
Deep Dose / Whole Body	5 rem (50 mSv)	2.1 minutes
Shallow Dose / Extremities	50 rem (500 mSv)	0.95 minutes
Member of Public (i.e. untrained operator)	0.1 to 5 rem (1 to 50 mSv)	2.5 to 9.5 seconds

Extremity is defined by the NRC as the hand, elbow, arm below the elbow, foot, knee, or leg below the knee. Whole Body is defined by the NRC as the head, trunk (including male gonads), arms above the elbow, or legs above the knee.

Safe Handling of Samples

As mentioned many times in this chapter, never place any part of your body in the path of the x-ray beam. There is always a safe way to handle samples whether they are small, irregularly shaped, or of low density. Never look into the path of the primary beam.

Small Samples

A small sample would be any sample that is smaller than the measurement window. Small samples present a unique risk because they don't block the entire beam path. The difficulty with placing small samples down on a work surface to analyze them is that you may get readings from the work surface that interfere with analytical results. A test stand is an effective way of analyzing small samples accurately and safely. Never hold samples during analysis or look into the path of the primary beam.

Irregularly Shaped Samples

Irregularly shaped samples may not allow the proximity button to be depressed, or they may not entirely cover the primary beam and cause additional scattering. A back scatter shield is a safe way of reducing your radiation exposure while effectively analyzing an irregularly shaped sample.

Light Materials (such as plastics).

X-rays are attenuated more by denser and higher atomic mass materials, and less through lighter materials such as plastic. This causes higher dose rates in the scattered radiation. If you are frequently handling low density samples, you should consider the use of test stands, backscatter shields, or the equivalent.

Niton XL2 Radiation Profile

Radiation Meter Information

Model: Bicron MicroRem

SN: 2057

Cal Due: 10/10/2009

Background Radiation Level

<0.01 mr/hr

Table 4 - Scatter Measurements off various substrates - Dose Rates in mRem/hr

Table 4. Niton XL2 Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
15	80	Low	Aluminum	<0.01	<0.01	<0.01
15	80	Low	Stainless	<0.01	<0.01	<0.01
15	80	Low	Plastic	<0.01	<0.01	<0.01
15	80	Low	Soil	<0.01	<0.01	<0.01
20	80	Low	Aluminum	<0.01	<0.01	<0.01
20	80	Low	Stainless	<0.01	<0.01	<0.01
20	80	Low	Plastic	3	<0.01	<0.01
20	80	Low	Soil	<0.01	<0.01	<0.01
45	44	Main	Aluminum	1.2	0.017	0.01
45	44	Main	Stainless	1.6	<0.01	<0.01
45	44	Main	Plastic	19	1.2	0.15
45	44	Main	Soil	2.0	0.050	0.025

Table 5 - Scatter Measurements off various substrates - Dose Rates in μSv/hr

Table 5. Niton XL2 Radiation Profile - Scatter Measurements - μSv/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
15	80	Low	Aluminum	<0.1	<0.1	<0.1
15	80	Low	Stainless	<0.1	<0.1	<0.1
15	80	Low	Plastic	<0.1	<0.1	<0.1
15	80	Low	Soil	<0.1	<0.1	<0.1
20	80	Low	Aluminum	<0.1	<0.1	<0.1
20	80	Low	Stainless	<0.1	<0.1	<0.1
20	80	Low	Plastic	30	<0.1	<0.1
20	80	Low	Soil	<0.1	<0.1	<0.1
45	44	Main	Aluminum	12	0.17	0.1
45	44	Main	Stainless	16	<0.1	<0.1
45	44	Main	Plastic	190	12	1.5
45	44	Main	Soil	20	0.50	0.25

Notes:

Scatter measurements were taken at a radius of 5 or 30 cm around the nose of the analyzer with the highest scatter dose rate being recorded.

Table 6 - In Beam Measurements - Dose Rates in Rem/hr

Table 6. Niton XL2 Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
15	80	Low	7.9	230	2.1	0.088
20	80	Low	41	690	19	0.90
45	44	Main	45	150	7.4	0.70

Table 7 - In Beam Measurements - Dose Rates in mSv/hr

Table 7. Niton XL2 Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30cm Deep
15	80	Low	79	2300	21	0.88
20	80	Low	410	6900	190	9.0
45	44	Main	450	1500	74	7.0

Notes:

In beam dose rates were measured using optically stimulated luminescent (OSL) dosimeters.

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

Niton XL2 GOLDD Radiation Profile

Table 8 - Niton XL2 GOLDD Radiation profile- Scatter measurements - mRem/hr

Table 8. Niton XL2 GOLDD Radiation Profile - Scatter Measurements - mRem/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
8	100	Light	Plastic	<0.01	<0.01	<0.01
8	100	Light	Stainless	<0.01	<0.01	<0.01
8	100	Light	Soil	<0.01	<0.01	<0.01
45	44.4	Main	Aluminum	0.5	<0.01	<0.01
45	44.4	Main	Stainless	0.01	<0.01	<0.01
45	44.4	Main	Plastic	5.0	0.4	0.8
45	44.4	Main	Soil	0.9	<0.01	<0.01

Table 9 - Niton XL2 GOLDD Radiation Profile - Scatter Measurements - μSv/hr

Table 9. Niton XL2 GOLDD Radiation Profile - Scatter Measurements - μSv/hr

kV	uA	Range	Substrate	Max @ 5cm	Max @ 30 cm	Max @ Trigger
8	100	Light	Plastic	<0.1	<0.1	<0.1
8	100	Light	Stainless	<0.1	<0.1	<0.1
8	100	Light	Soil	<0.1	<0.1	<0.1
45	44.4	Main	Aluminum	5.0	<0.1	<0.1
45	44.4	Main	Stainless	0.1	<0.1	<0.1
45	44.4	Main	Plastic	50	4.0	8.0
45	44.4	Main	Soil	9.0	<0.1	<0.1

Notes:

Scatter measurements were taken at a radius of 5 or 30 cm around the nose of the analyzer with the highest scatter dose rate being recorded.

Table 10 - Niton XL2 GOLDD Radiation Profile - In Beam Measurements - Rem/hr

Table 10. Niton XL2 GOLDD Radiation Profile - In Beam Measurements - Rem/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30 cm Deep
8	100	Light	0.01	980	0.002	<0.001
45	44.4	Main	39	130	8.1	0.51

Table 11 - Niton XL2 GOLDD Radiation Profile - In Beam Measurements - mSv/hr

Table 11. Niton XL2 GOLDD Radiation Profile - In Beam Measurements - mSv/hr

kV	uA	Range	Contact Deep	Contact Shallow	5cm Deep	30 cm Deep
8	100	Light	0.1	9800	0.02	<0.01
45	44.4	Main	390	1300	81	5.1

Notes:

In beam dose rates were measured using optically stimulated luminescent (OSL) dosimeters.

Reported results are based on measurement results that have been reduced to 2 significant digits by rounding up. For example, a measurement result of 1441 would be reported as 1500.

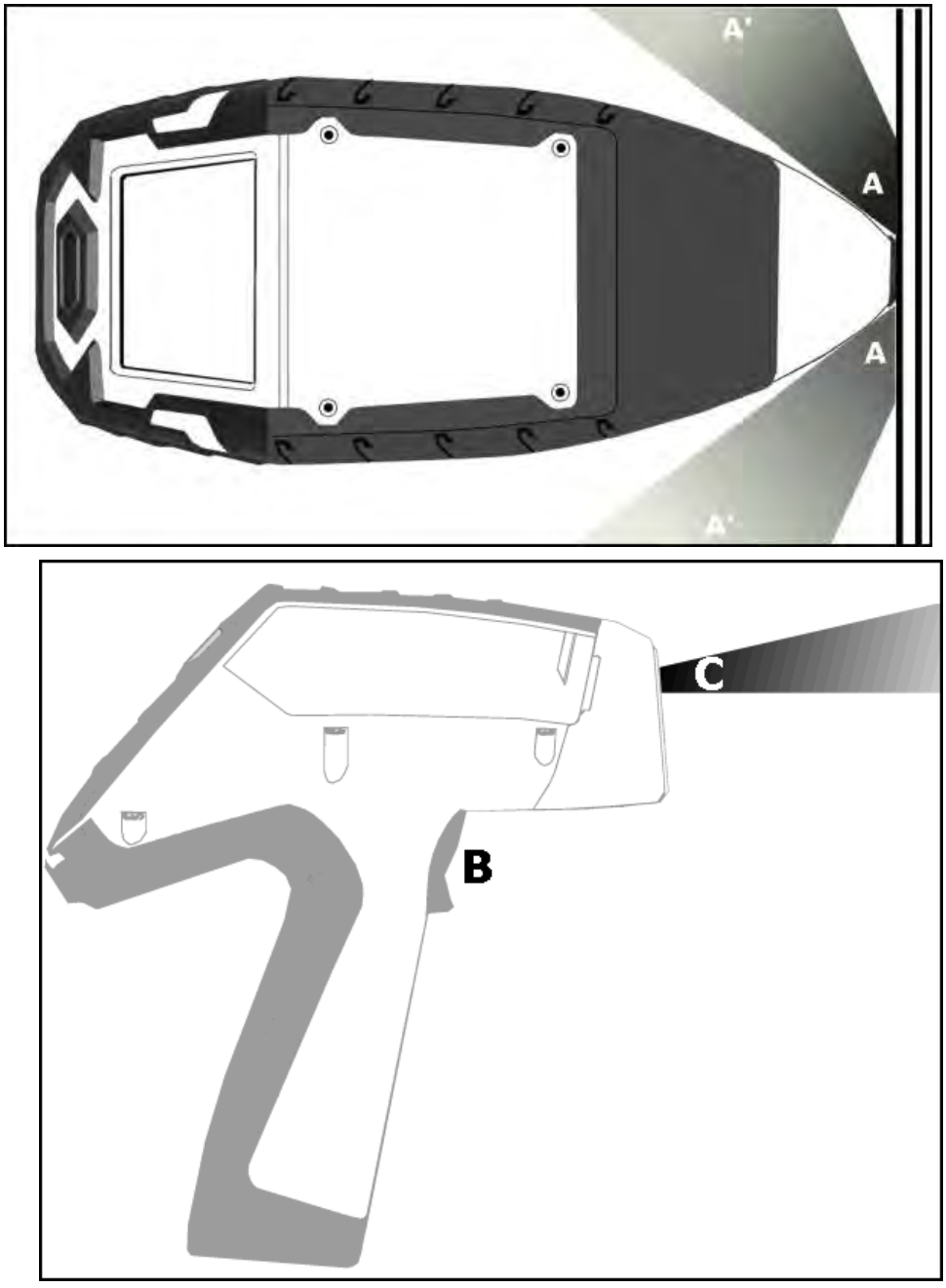


Figure 4. Primary and Secondary Dose Locations (Not to Scale)

Primary Radiation

Primary radiation is radiation that is produced by the analyzer and emitted out through the kapton measurement window. Individuals should never place any part of their body in the primary beam path when the x-ray tube is on. There should always be a sample in contact with the measurement window when the x-ray tube is on. The sample will absorb most of the primary-beam radiation unless it is smaller than the instrument's measurement window or of low atomic mass, low density, and/or very thin. Caution should be taken when analyzing samples that are small, thin, and/or low in atomic mass or density as they may allow much more of the primary beam to escape. In-beam primary radiation dose rates for the Niton XL2 are listed in Table 6 andTable 7 - or Table 10 and Table 11 for the Niton XL2 GOLDD - and their location identified relative to the analyzer in Figure 4 as Dose Point C.

Secondary Radiation

Under conditions of normal and proper use, individuals can be exposed to secondary (or "scattered") radiation. Secondary radiation is low-level radiation that emanates from the sample being analyzed as a result of primary beam radiation scattering in the sample or primary beam radiation inducing fluorescent x-rays in the sample. Dose points A, A' and B in Figure 4 are examples of where you can encounter secondary radiation. The magnitude of this secondary radiation is sample dependent. Higher atomic mass and density samples such as steel will emit the lowest levels as they absorb most primary and secondary radiations. Lower atomic mass and density samples such as aluminum, wood, and especially plastic, will produce higher levels of secondary radiation. Secondary radiation dose rates for the Niton XL2 are listed in Table 4 and Table 5 - or Table 8 and Table 9 for the Niton XL2 GOLDD - for a few common sample types over a wide range of densities.

The operator is reminded that one should never hold samples during analysis, doing so will result in higher than necessary exposure to secondary radiation and could expose the operator directly to the much higher primary-beam dose rates.

Deep and Shallow Dose

You will find in Table 6, Table 7, Table 10, and Table 11 that shallow dose rates are listed for some dose points. All dose rates listed in these four Tables are deep dose unless they are specifically identified as shallow dose. Deep dose is dose from penetrating radiation that is delivered to both skin and underlying tissues and organs and is the type most commonly referred to when describing external radiation hazards. Occupational deep dose is limited to a maximum of 5 rem (50 mSv) per year in the United States and most countries internationally. Deep dose is measured at 1.0 cm below the skin surface.

Shallow dose is often referred to as "skin dose" because it is a result of low penetrating radiation that only interacts with the skin. Shallow dose is limited to a maximum of 50 rem (500 mSv) per year in the United States and most countries internationally. Shallow dose is listed for primary in-beam dose points only because the low penetrating radiation that causes shallow dose is nearly all absorbed by a sample and does not produce any significant secondary radiation. Shallow dose is measured at a point 0.007 cm below the surface.

Proper and Improper Operation

Storage and Transportation

Storage

Regulations in nearly all locations will require that you store your analyzer locked in a secured area to prevent access, use, and/or removal by unauthorized individuals. Storage requirements will vary by location, particularly with regard to storage at temporary job sites or away from your primary storage location such as hotels and motels and in vehicles. You should contact your local Radiation Control Authority to identify the specific storage requirements in your jurisdiction.

Transportation

There are no X-ray tube specific US Department of Transportation (DOT) or International Air Transport Association (IATA) radiation regulations regarding shipping the Niton XL2 analyzer. It is recommended that you ship the analyzer in its carrying case and an over-pack to protect the sensitive measuring equipment inside the analyzer. Do NOT ship the analyzer with the battery pack connected to the analyzer.

Lost or Stolen Instrument

Note THIS PAGE CONTAINS EMERGENCY CONTACT INFORMATION THAT SHOULD BE AVAILABLE TO THE OPERATOR AT ALL TIMES.

If the Niton XL2 analyzer is lost or stolen, notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately. Your company's RSO, as well as other important emergency contacts, are listed below. Your company RSO may need to notify the x-ray tube regulatory authority and the local police. It is also recommended that a notification is made to Thermo Fisher Scientific.

Damaged Instrument

Minor Damage

If the instrument is intact but there is indication of an unsafe condition such as a cracked case, a shutter mechanism failure, or the lights remain flashing after a measurement is terminated, follow these steps:

1. Stop using the instrument
2. Remove the battery. The x-ray tube can not produce radiation when the battery is disconnected. The instrument is now safe to handle.
3. Place the instrument securely in the holster.
4. Place the instrument in the carrying case that came with the instrument.
5. Notify your Radiation Safety Officer (RSO) or the equivalent responsible individual at your company or institution immediately.
6. You or your RSO should call Thermo Fisher Scientific at one of their contact numbers listed below for additional instructions and guidance.

Major Damage

If the instrument is severely damaged:

1. Perform the same steps as described above for minor damage. There will be no radiation hazard as long as the battery is removed from the instrument.
2. Place all components in a plastic bag and contact Thermo Fisher Scientific.

Emergency Response Information

Please Complete the Following Emergency Response Information and Keep with the Analyzer at All Times

NITON ANALYZER EMERGENCY CONTACT INFORMATION

The Company RSO is:_____

RSO Telephone Number:_____

Regulatory Agency Emergency Number:_____

Local Fire Department:_____

Local or State Police Department:_____

Thermo Fisher Scientific's Niton Analyzer Contact Numbers

Main Number (USA): (800) 875-1578

Additional Radiation Emergency #'s: (978) 790-8269 or (617) 901-3125

Outside the USA - Local Niton Service Center:_____

Europe

Niton Analyzers Europe

Munich, Germany

Phone: +49 89 3681 380

Fax: +49 89 3681 3830

Email: niton.eur@thermofisher.com

Asia

Niton Analyzers Asia

Hong Kong

Phone: +852 2869-6669

Fax: +852 2869-6665

Email: niton.asia@thermofisher.com

Registration and Licensing

As a user of a Niton XL2 analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. See the Safety and Compliance Web Hub for much more information.

Regarding Safety Devices for the Open Beam Configuration:

In the US, you may be required to file for an exemption, "variance letter", with your state if there is a requirement for a safety device that would prevent entry of an extremity into the primary beam. If you need assistance with the exemption letter, you may contact the radiation safety group.

Registration and Licensing FAQ

See the "Registration and Licensing FAQ" on page 433

How to Analyze

To analyze samples, from the main menu select sample type, and then click on the appropriate Mode icon. Once in the Selection Screen you have a number of sub-modes to select from. depending on how your instrument is calibrated. See the Example Path below.

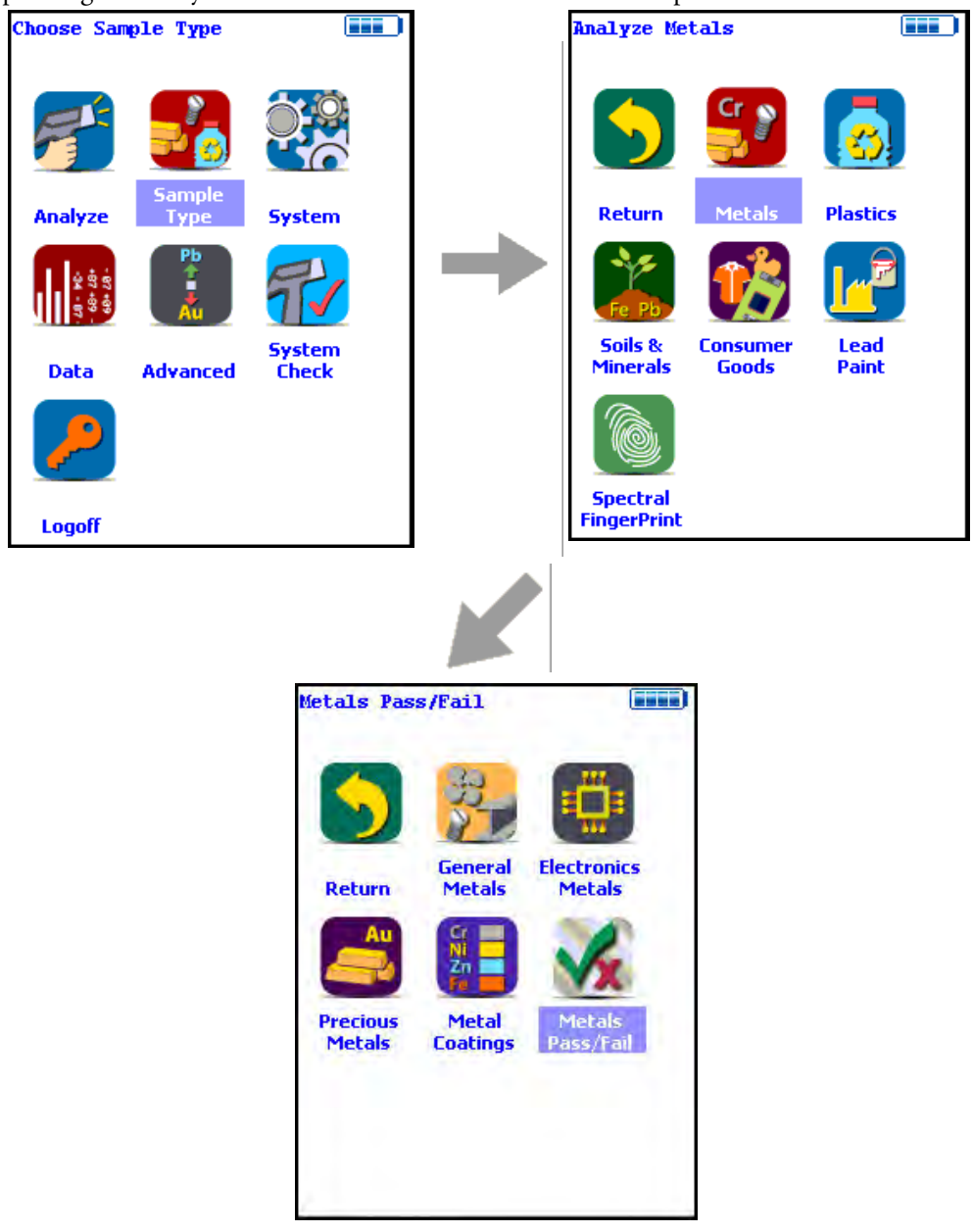


Figure 5. The Metals Analysis Menu Path (Example)

Element Ranges and Lists

From the Element Range Screen, select the Element List Button to display the Element List for the Range you want to use. This list shows the elements that the Range is best designed to detect. See Adjusting the Element Range for details.

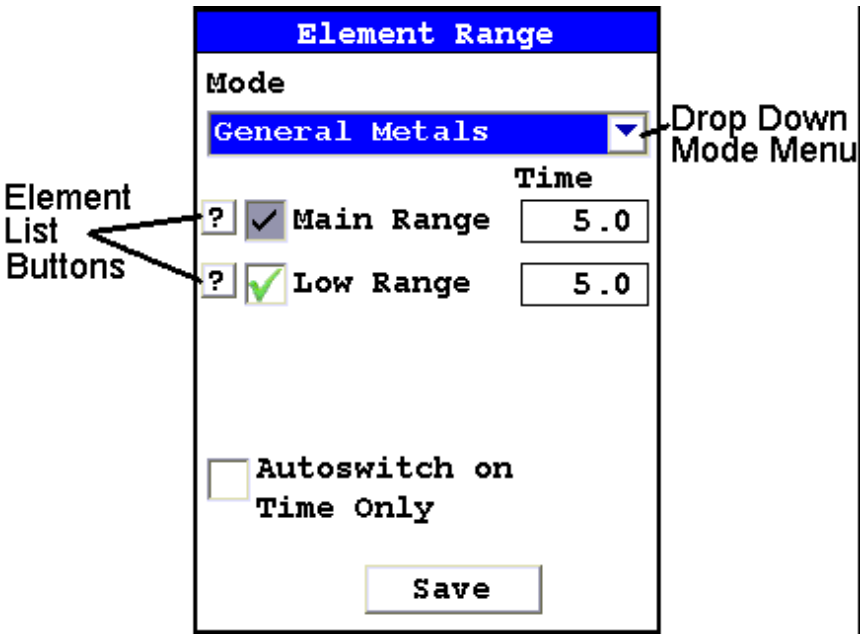


Figure 6. The Element Range Screen

General Analysis

Note Each user should read the Thermo Scientific Niton XL2 User's Guide carefully before initiating measurements with the system. Users are strongly urged to attend the Thermo Scientific Niton XRF Analyzer Radiation Safety and Operations Training courses offered regularly, or the web-based trainings. For more information, visit www.thermo.com/niton.

PREPARATORY TASKS

Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and "Log On" as the operator using either the default password or a custom one as designated by the user in an NDU file.

Wait five (5) minutes before using the analyzer, allowing the instrument electronics to stabilize.

Verify that the date is set properly for data tracking purposes.

From the Main Menu, select the System icon, then the Specs icon. The date will be displayed for verification. If the date is incorrect, correct it prior to proceeding. This can be done by "Closing" out of the Specs screen and selecting the Date & Time icon. Detailed information on this procedure is available in Setting the Date and Time.

(Optional) Connect the analyzer to a computer via the included serial cable, USB cable, or Bluetooth™ wireless module. (Consult "Using Your Analyzer With Your PC" on page 109for details, if necessary.)

During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.

From the Main Menu, select System Check icon then the Yes button. (Figure 1.)

System Check calibrates the detector and verifies it is operating to specifications. After starting the process, no further user interaction is required during this operation. When the instrument is finished performing the check, the unit will show either "System OK" or one of the failure errors.

If the unit shows a failure error, then perform a second System Check by clicking Recheck. If the unit still does not show a "System OK," please contact Thermo Scientific Niton Analyzers toll-free in the USA at (800) 875-1578, +1 978 670-7460, niton@thermofisher.com, or contact your local Niton Analyzers representative for assistance.

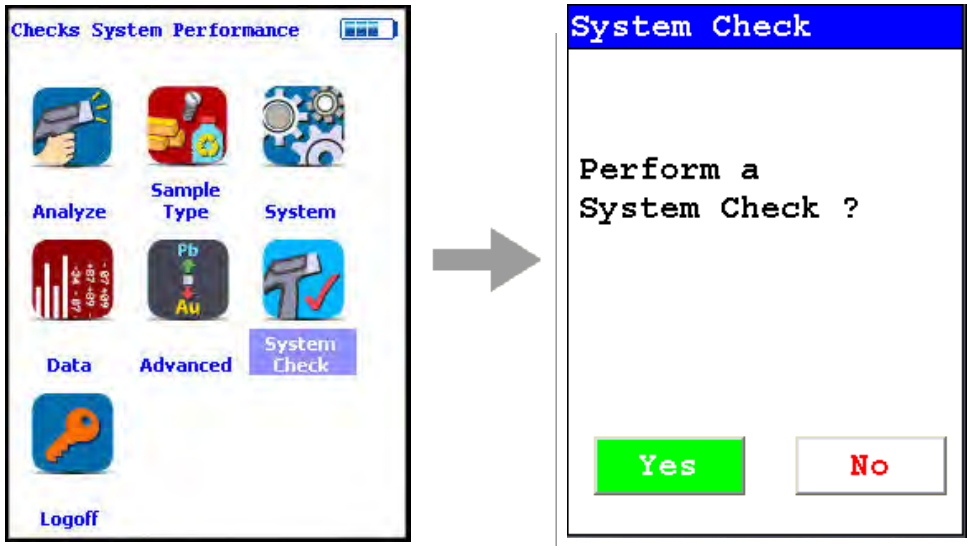


Figure 7. System Check Menu Path

Thermo Scientific Niton XL2 analyzers are equipped with excitation filters that optimize the analyzers' sensitivity for various elements. The "Main Range" filter provides optimum sensitivity for the elements manganese (Mn) through bismuth (Bi). The "Low Range" filter is used to optimize the sensitivity for the elements from titanium (Ti) through chromium (Cr). Note that the main range filter can be used to analyze Ti, V and Cr, but the sensitivity is not as good as when using the low filter. The "Light Range" filter is available only with He-purged and GOLDD technology analyzers, and is typically used in light element analysis. The amount of time that the analyzer spends in each filter position is user definable, but the default settings should be used unless there is reason to change them. Please note that the analyzer will continue alternating excitation filters until the user selectable maximum analysis time is reached or the operator terminates the measurement.

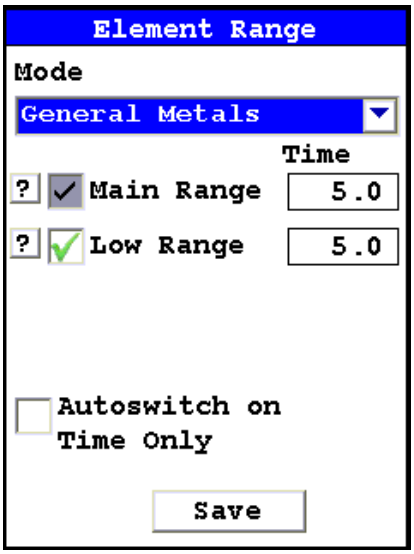


Figure 8. Setting Element Ranges

Verify instrument measurement accuracy using the supplied reference material (RM) supplied with the analyzer.

Test the factory-supplied reference standard (or other approved check sample) based on a 30s measurement using main range filter only. If the sample is correctly identified and all major elements read within calculated acceptance limits (within the low and high values of factory readings found on the QC sheet, proceed to General Testing Protocol section

If the analyzer reports values outside the acceptance tolerance ranges specified in the tables, repeat the detector calibration then repeat the reference sample analysis.

If the analyzer again fails to meet the acceptance tolerance ranges specified in the tables, please contact Thermo Scientific Niton Analyzers or your local representative for assistance.

GENERAL TESTING PROTOCOL

Good surface preparation is essential for obtaining accurate test results. All non- representative material (e.g., paint, coating, scale) must be removed prior to testing. An approximately 2-inch-square section of surface should be cleaned down to the material to be analyzed. See the Resource Guide for information on Sample Preparation.

The analyzer will often display a correct alloy identification and/or accurate chemistry result before the specified time interval. If the accuracy meets the user's requirements, it is not necessary to measure for the full time.

Longer measurements might be necessary if low concentrations of elements must be determined.

INSTRUMENT QC

Measure the supplied reference calibration check sample AT LEAST once a shift. If correct, continue work. If incorrect, redo System Check and re-take the past 2 hours of results.

UNDERSIZED OR NON-CONTACT SAMPLES

(Samples that do not make contact with or that do not fully cover the measurement aperture)

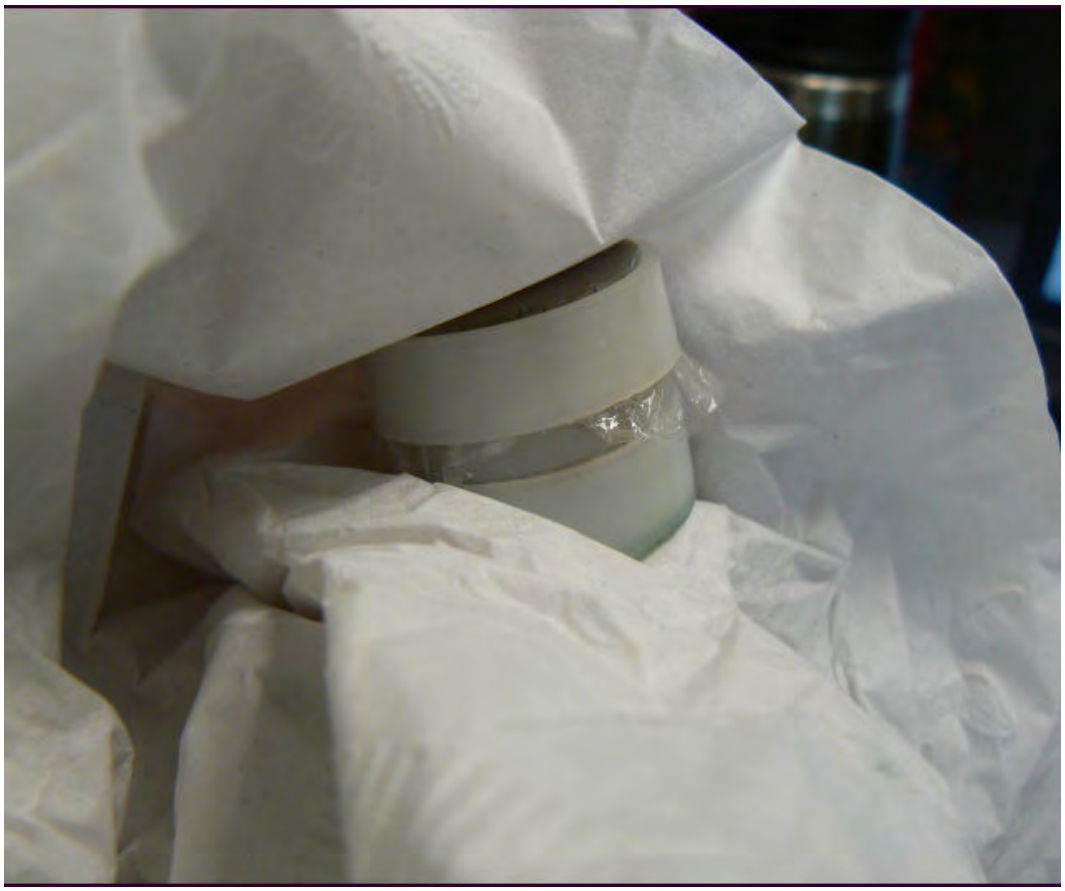
For samples that do not fully cover the measurement aperture, increase the testing time by increasing the time in inverse proportion to the decrease in percentage of aperture covered. For example: a rod only covers ½ of the aperture, so increase the measurement time by two (e.g., from 10 to 20 seconds per filter for alloy chemistry).

The best procedure to measure undersized samples is to use the Thermo Scientific Niton portable test stand (optional), which is shielded to prevent radiation exposure to the operator.

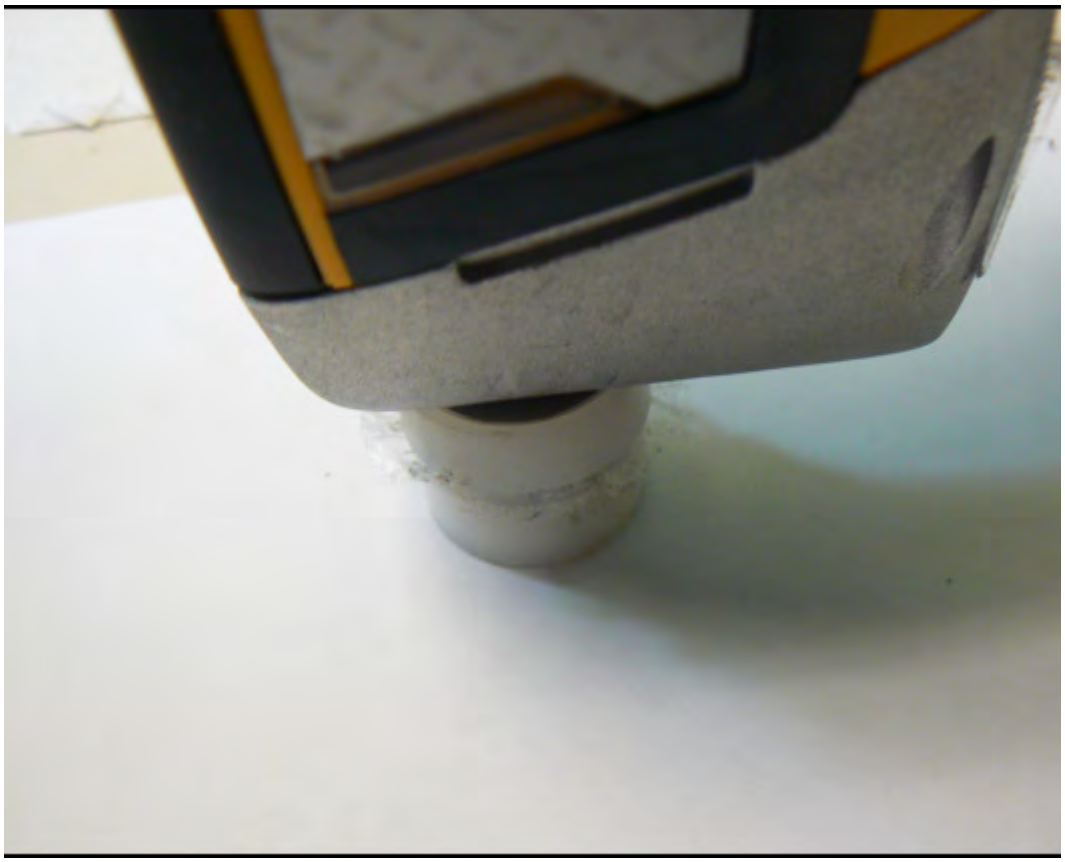
An undersized sample may alternately be measured while lying on another material. Results may be affected by the signal coming from the underlying material itself. Use only pure aluminum, pure plastic, or clean wood and employ the Disable Al feature. Use the Tools Menu, then select Disable Al, and check the underlying surface itself to be sure no metals are present. Be sure to use the Tools Menu and select Enable Al before testing aluminum alloys.

Basic Operation

Taking a Sample Analysis



1. Clean the sample to be analyzed so it is free of all surface contamination.



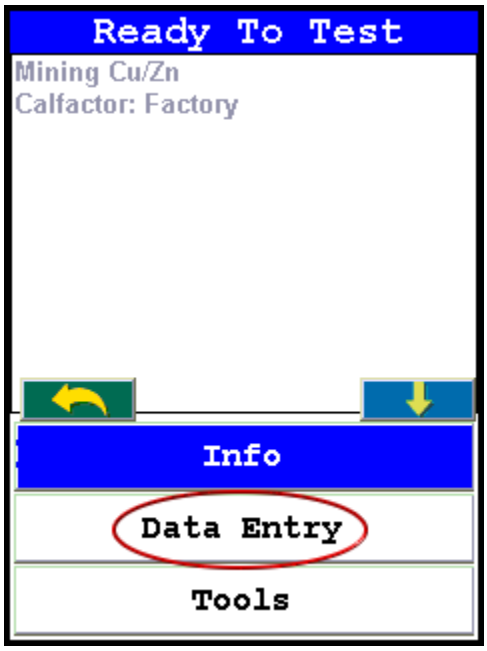
2. Place the analyzer so the sample is covered by the analysis window.

3. Select the Sample Type Icon.

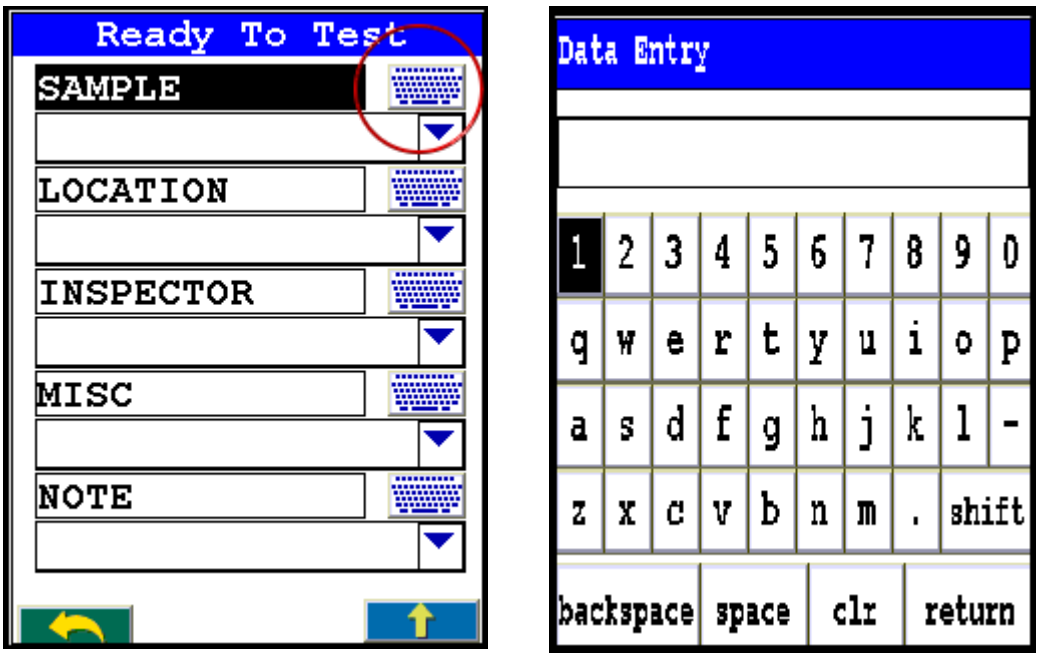
4. Select the proper Mode (in this case Mining Cu/Zn) from the Mode Menu.

Note See "Analysis Modes" on page 37. for more information on the Modes available.

5. Select the Analyze Icon.



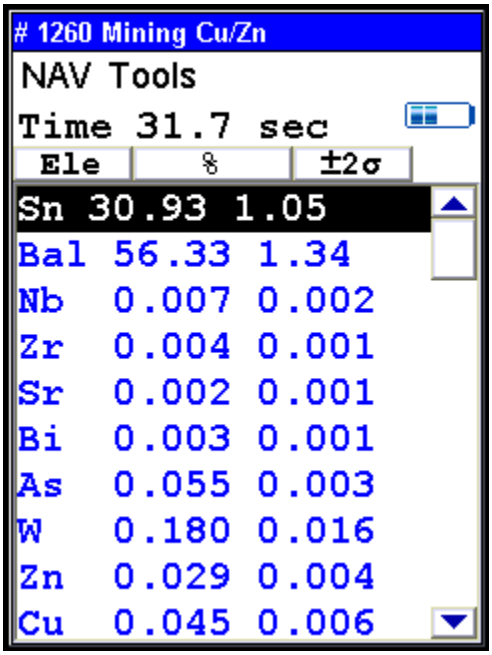
5a. Select Data Entryif you wish todo any data entry.



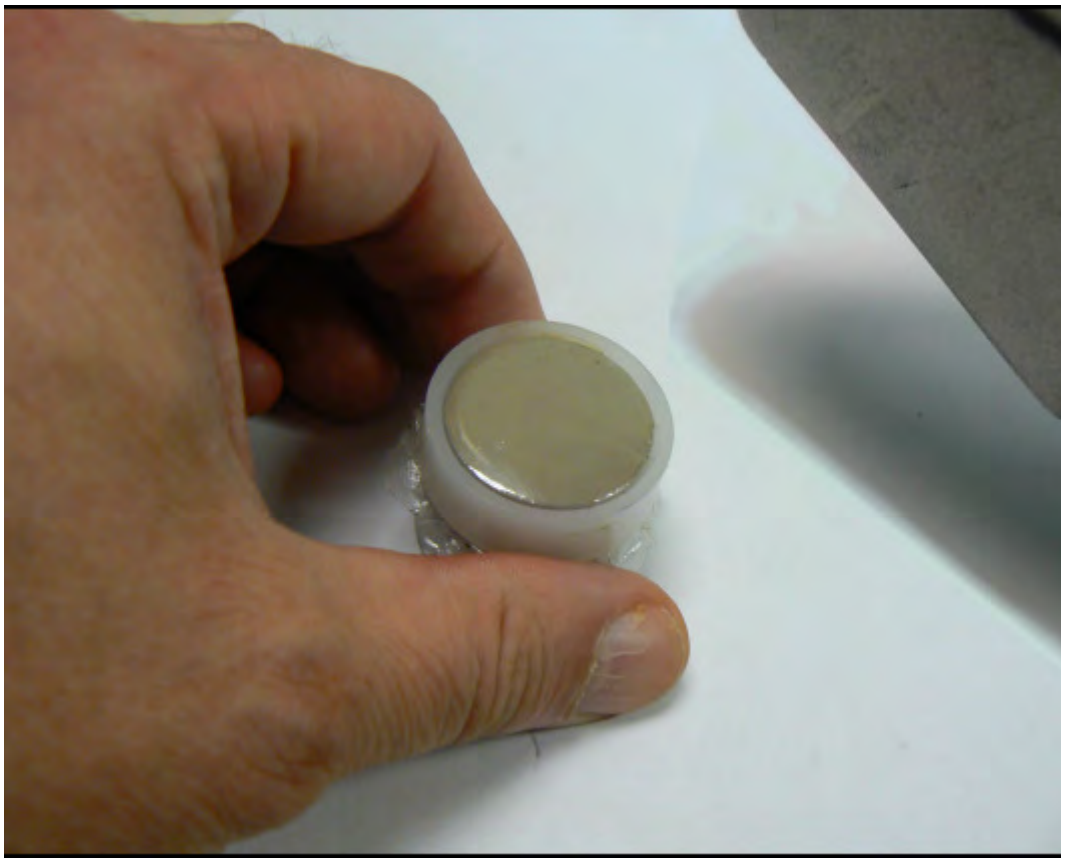
5b and 5c. Enter the data on the sample using the Virtual Keyboard.

6. Initiate a Reading by pressing the trigger.

7. When the sample has been sufficiently analyzed, release the trigger.



8. View the composition returned.



9. Remove the sample.

Analysis Modes

Your analyzer has several Analysis Modes. Which Analysis Mode you should use depends on the nature of the sample you are attempting to analyze.

General Metals Mode

Use this mode to analyze samples entirely composed of metal alloys. This mode will attempt to return an Alloy Grade Identification by matching the analyzed composition of the sample with the nominal composition of alloys in the analyzer's Alloy Grade Library. It will also return an elemental composition of the alloy as analyzed. Alloy Composition is output by default in terms of percent of composition by weight.

See “Using General Metals Mode” on page 39.

Electronic Metals Mode

Use this mode to analyze electronic component samples - circuit boards, chips, etc. This mode will attempt to return an Alloy Grade Identification by matching the analyzed composition of the sample with the nominal composition of electronic alloys in the analyzer's Alloy Grade Library. It will also return an elemental composition of the electronic alloy as analyzed. Electronic Metal Composition is output by default in terms of percent of composition by weight.

See “Using Electronic Metals Mode” on page 40.

Precious Metals Mode

Use this mode to analyze samples composed primarily of precious metals. This mode will attempt to return an Alloy Grade Identification by matching the analyzed composition of the sample with the nominal composition of alloys in the analyzer's Precious Alloy Grade Library. It will also return an elemental composition of the precious metal sample as analyzed. Precious Alloy Composition is output by default in terms of parts per million.

See “Using Precious Metals Mode” on page 40.

Plastics Mode

Use this mode to analyze samples composed primarily of plastic. This mode will return an elemental composition of the plastic sample as analyzed. Plastic Composition is output by default in terms of parts per million.

See “Using Plastics Mode” on page 42.

Soils Mode

Use this mode to analyze samples composed primarily of soil and rock. This mode will return an elemental composition of the soil sample as analyzed. Soil Composition is output by default in terms of parts per million.

See “Using Soils Mode” on page 42.

Mining Cu/Zn Mode

Use this mode to analyze samples composed of potential metal ore - rock containing high proportions of metal - and containing Cu and/or Zn. This mode will return an elemental composition of the ore sample as analyzed. Ore Composition is output by default in terms of percent of composition by weight.

See “Using Mining Cu/Zn Mode” on page 43.

Mining Ta/Hf Mode

Use this mode to analyze samples composed of potential metal ore - rock containing high proportions of metal - and containing Ta and/or Hf. This mode will return an elemental composition of the ore sample as analyzed. Ore Composition is output by default in terms of percent of composition by weight.

See “Using Mining Ta/Hf Mode” on page 44.

TestAll Mode

Use this mode to analyze samples composed of unknown and/or mixed composition, such as toys and consumer products. This mode will attempt to return a general Material Identification by comparing the analysis with other general types of materials. It will select the proper sub-mode for analysis and return an elemental composition of the sample as analyzed. Material Elemental Composition is output by default in terms of parts per million.

See “Using TestAll Mode” on page 44.

TestAll Geo Mode

Use this mode to analyze powder, mineral, and ore samples without first determining whether the samples would best be analyzed with Mining or Soil Mode. This mode uses both the Compton Normalization calibration (Soil) and the Fundamental Parameters calibration (Mining) to determine whether the soil calibration is acceptable or whether the total metal content is too high for Compton mode. It will then return an elemental composition of the sample as analyzed. If the sample can be analyzed via soil mode, then the analyzer will display results from both Soil and Mining Modes in one unified list. If both calibrations contain the same element, then the mode that has the lower detection limit will be displayed. Material Elemental Composition is output by default in terms of both parts per million (mg/kg) and percent of composition by weight, with 0.10% being the cutoff point.

Note Due to the nature of this mode, your analyzer will only use factory calibrations. User modified Cal Factors will not be available.

See “Using TestAll Geo Mode” on page 44.

Using General Metals Mode

1. Clean the sample to be analyzed so it is free of all surface contamination, grinding the surface if appropriate.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select General Metals from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Electronic Metals Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Electronic Metals from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Precious Metals Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Precious Metals from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Plastics Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.
2. Place the analyzer so that the sample covers the analysis window.
3. Select the Mode icon.

a. Select Plastics from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.
6. When the sample has been sufficiently analyzed, stop the analysis.
7. View the composition returned.
8. Remove the sample.

Using Soils Mode

1. Pack the sample into a Sample Cup.

a. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.
3. Select the Mode icon.

a. Select Soils from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.
6. When the sample has been sufficiently analyzed, stop the analysis.
7. View the composition returned.
8. Remove the sample.

Using Mining Cu/Zn Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select Mining Cu/Zn from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Using Mining Ta/Hf Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.
2. Place the analyzer so that the sample covers the analysis window.
3. Select the Mode icon.

a. Select Mining Ta/Hf from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.
6. When the sample has been sufficiently analyzed, stop the analysis.
7. View the composition returned.
8. Remove the sample.

Using TestAll Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.
2. Place the analyzer so that the sample covers the analysis window.
3. Select the Mode icon.

a. Select TestAll from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.
6. When the sample has been sufficiently analyzed, stop the analysis.
7. View the composition returned.
8. Remove the sample.

Using TestAll Geo Mode

1. Clean the sample to be analyzed so it is free of all surface contamination.

2. Place the analyzer so that the sample covers the analysis window.

3. Select the Mode icon.

a. Select TestAll Geo from the Mode Menu.

4. Select the Analyze icon.

a. Select the Data Button if you wish to do any data entry.

b. Enter the data on the sample using the Virtual Keyboard.

5. Initiate the analysis.

6. When the sample has been sufficiently analyzed, stop the analysis.

7. View the composition returned.

8. Remove the sample.

Common Operations

Metal Sample Prep

Up until recently, sample preparation was not a big worry for XRF metals analysis, as the LOD of the analyzer was seldom low enough for any but the most heavy contamination to be intrusive; but recent developments such as He-purged analysis have brought analysis to a level where even light surface contamination can skew an analysis.

You should always prepare your samples before analysis, especially when using He-purged analysis, as these analyzers will see even trace amounts of contaminants. Oils from fingerprints and other body contact, lint, oxidation materials, and abrasive materials used in cleaning can all skew readings if not removed. Sample preparation is simple and not time consuming, and usually well worth the effort.

The following is a list of problems that need correction before testing:

- Oxidation or Rust may produce an increase or decrease in one or more element test values unless we remove the rust or oxidation and expose the raw metal.
- Paint may contain several elements which need to be tested at lower levels within metal alloys (Ti & Zn in white paint, Fe in red paint, Cr in green paint).
- Oil, grease or lubricates may contain high levels of the following elements: lithium, aluminum, barium, strontium, molybdenum or calcium.

Plated surfaces may have high levels of the following elements: zinc, chromium, nickel, or copper.

CAUTION Anything on the metal surface will become part of your test results!

Sample Analysis Preparation

You need to clear the surface of your samples of any paint, plating, or any oxidation such as rust or verdigris before analysis. In order to accomplish this, you need the following:

- Isopropyl alcohol - not rubbing alcohol, which contains oils.
- Lint-free paper.
- Diamond paper - P/N 179-1202- cut into 1 inch/2.5 cm squares. Never re-use this paper, as it may transfer contaminants to the surface of the sample from previous cleanings. Depending on the state of the sample, several squares may be needed per sample.

- A Sample Grinder for removing deeper surface contamination. Choice of grinding wheel media also may be important, depending on what you are testing for. Never re-use grinding media, as contaminants can be transferred from sample to sample on the media itself.

For light contamination on hard metal reference standards, remove the oxidation by scrubbing the dry sample lightly with the diamond paper square, using the fingers to maintain pressure. If the diamond paper begins to load up with material, discard it and use a fresh square. When the oxidation is removed, wipe the sample with lint-free paper soaked with isopropyl alcohol to remove any oils or dust. Let the sample dry before attempting analysis.

For soft metal reference standards, wipe the sample with lint-free paper soaked with isopropyl alcohol, then remove the oxidation by scrubbing the wet sample lightly with the diamond paper square, using the fingers to maintain pressure. If the diamond paper begins to load up with material, discard it and use a fresh square. When the oxidation is removed, wipe the sample again with lint-free paper soaked with isopropyl alcohol to remove any oils or dust. Let the sample dry before attempting analysis.

Oils, lint and dust can be removed by wiping the sample with lint-free paper soaked with isopropyl alcohol. Let the sample dry before attempting analysis.

Surface Oxidation

With the exception of a limited number of metal types, most metal alloys form an oxide covering on the surface when exposed to oxygen or air. This oxide covering is visible in carbon and low alloy steel as a red colored substance called rust. Other metal alloys form oxidation which is not always visible, but that does not mean that it is not present. If the test results for low concentration elements are higher or lower than expected, remove the oxide coating by grinding and retest. Follow proper safety procedures when changing discs or grinding materials.

During a recent case study the effects of sample preparation became apparent. A customer asked for low detection limits of nickel, chromium and copper in carbon steel pipe. The reported chemistry of the purchased material is listed on the first line in the chart below. The test results of a hand held Niton XL2t 900S GOLDD instrument appears in the second line of the chart. The results from a test on the unground surface appear in the bottom line of the chart. Note the values for nickel and copper in this carbon steel alloy in the chart below. The oxidation on the surface of this pipe was not visibly egregious. We need to always be wary of the presence of even low levels of oxidation and their possible effects on analytic accuracy.

Table 1. Comparative test results with and without grinding

Sample	% Mn	% Ni	% Cr	% Mo	% Cu
Reported Chemistry	0.650	0.090	0.070	0.030	0.040

Table 1. Comparative test results with and without grinding

Test Results with Ground Surface	0.67	0.089	0.070	0.033	0.039
Test Results with Unground Surface	0.61	0.178	0.081	0.033	0.514

Painted Surfaces

Paint is a mixture of several items that are combined into a liquid which is applied to the surface of materials such as metal. Once applied this liquid dries with time and adheres to the surface of metal. Paint is used to protect or decorate the metal item. Paint can also be used to identify or mark the metal during the manufacturing process.

Components of paint are divided into classifications of pigments, binders, solvents, additives and fillers. The inorganic elements in pigments will contribute to increases in displayed values for those elements if paint on the metal surface is not removed prior to testing. Be especially careful of the presence of heavy elements, which can also act to shield x-rays from lighter elements in the metal sample.

The following is a list of some of the most common components of paint:

White Paint

- Antimony (Sb)
- Lead (Pb)
- Titanium (Ti)
- Zinc (Zn)
- Cobalt (Co)

Red Paint

- Iron (Fe)
- Lead (Pb)
- Green Paint
- Chromium (Cr)

An experiment was conducted to determine the effect and severity of surface problems on XRF results. Results from analyses of a 1541 alloy steel sample are shown below, before and after surface grinding. The sample had painted markings, of light to medium thickness, on the surface, as well as light rust. Note the change in titanium, zinc and cobalt levels after surface grinding.

Table 2. Prepped and unprepped painted metal analysis

Sample	Mn	Ni	Cr	Mo	Ti	Zn	Co
Ground Surface	1.49	0.04	0.03	0.004	0.011	0.0001	0.03
Unground Surface	1.34	0.01	0.04	0.011	2.507	1.751	0.21

Oil, Grease & Cutting Oils

Oil and grease contain a number of elements combined into a viscous substance and applied to moving parts in order to reduce friction. Grease coatings can remain on component surfaces after it has been removed from service. Grease can also be applied to a metal's surface by accidental contact with other materials coated in heavy grease. Metals can also be coated in oil as a result of cutting and machining processes in manufacturing.

Grease and oil may contain the following elements:

- Aluminum (Al)
- Zinc (Zn)
- Molybdenum (Mo)
- Sodium (Na)
- Calcium (Ca)

An experiment was performed to show how grease on metal surfaces affects XRF results. A carbon steel sample was cleaned and ground as a control surface for the experiment. XRF tests were performed on the control surface, and again after light and heavier layers of automotive wheel bearing grease were applied to the surface of the steel sample. Results are shown below. Note the elevated levels of molybdenum, cobalt and zinc from the grease.

Table 3. Clean and greased sample metal analysis

Sample	Mn	Ni	Cr	Mo	Cu	Co	Zn
Clean Surface	1.18	0.001	0.041	0.004	0.001	0.001	0.019
Light Grease	1.07	0.001	0.001	0.067	0.033	0.322	0.416
Heavy Grease	0.96	0.001	0.001	0.500	0.062	1.760	3.430

If a sample's surface contains lubricants or cutting oil, use a solvent and a clean towel or rag to remove them before analysis. You may then need to grind the surface to insure good results. Clean first, grind second, test last.

Remember to follow safe techniques for handling and disposing of solvents and cleaning rags

Anodized, Plated and Galvanized Surfaces

Anodizing is the process of polarizing the metal surface into a passive state which protects it against corrosion. This process is most often applied to aluminum alloys.

Galvanized steel is one of the most common of the coated surfaces. In this process, steel is passed through a molten bath of a zinc alloy. Zinc reacts with the steel metal to form a bonding layer on the steel surface. The zinc layer does not separate from the steel and forms a protective layer that protects the steel from oxidation.

Galvanized layers are relatively thick compared to other plating elements and methods. When grinding to remove the zinc coating, you will find increased zinc values even when you can see the steel surface. Grind a little further and zinc values will disappear. Zinc clings to the surface of the sanding disc, so you will need to frequently change discs.

Electroplating is another common practice of applying a coating which not only protects the surface from oxidation, but also improves the base material's wear resistance, lubricity and improves the overall aesthetics of the product. The electroplated coating is generally thinner and more evenly applied than galvanizing. Electroplating has a wide range of elements and in some situations there may be two or more different coatings on the same part.

The following is a partial list of elements that are used to plate the surface of base metals:

Ni, Cr, Cadmium (Cd), Tin (Sn), Zn, Al

Cordless Right Angle Drill

This style of drill is recommended for most surface preparation in the field because it gives the operator the greatest amount of control, and thus safety, when grinding samples. When moving a sanding disc on a conventional drill over a sample, forces tend to produce movement the operator may find difficult to control. Control and stability are important in grinding from effectiveness and safety perspectives.

A cordless right angle drill similar to the one pictured below is recommended for light to medium surface removal. For materials with heavy oxidation such as carbon and low alloy steel, an angle grinder, explained in the next section, is recommended. A kit with the drill, batteries and charging units, can be purchased from ThermoFisher, or companies such as DeWalt, Hitachi, Makita, Milwaukee or Ryobi.

Figure 1. Example of Right Angle Drill

A disc holder is needed with the drill to hold the sanding disc. (In the US, we recommend a 3.0 inch disc holder. It has a 0.25 inch shank to insert into the chuck of the drill.) If sanding discs are ordered from a local supplier, attention should be paid to the method of attaching the sanding disc to the disc holder. There are three types of connections: metal snap-on, plastic twist and plastic snap-on.

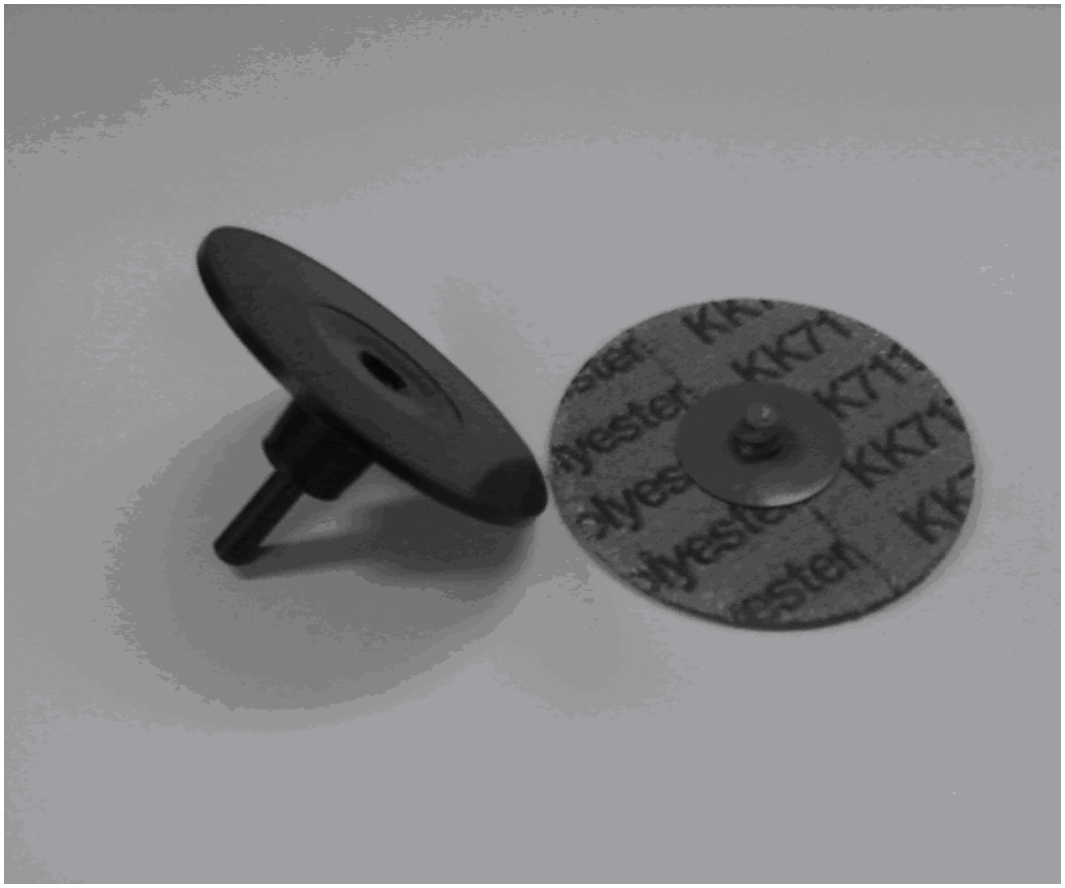


Figure 2. Sanding Disc

Before attaching the grinder and sanding disc as pictured below, first remove the battery to disable the grinder. Then insert the shaft of the disc holder into the drill and securely tighten the chuck. Next, attach the appropriate sanding disc. The method of attachment will vary depending upon the type of fastener on the sanding disc (snap-on or twist connectors). Reinstall the battery and prepare for use.

Figure 3. Attaching the Sanding Disc 1

Figure 4. Attaching the Sanding Disc 2

Cordless Angle Grinder

A cordless angle grinder similar to the one pictured below will successfully remove medium to heavy oxidation or paint. This grinder (which uses a 4.5 inch sanding disc with a rubber backup pad) can be purchased from ThermoFisher or industrial tool manufactures like DeWalt, Makita or Milwaukee.

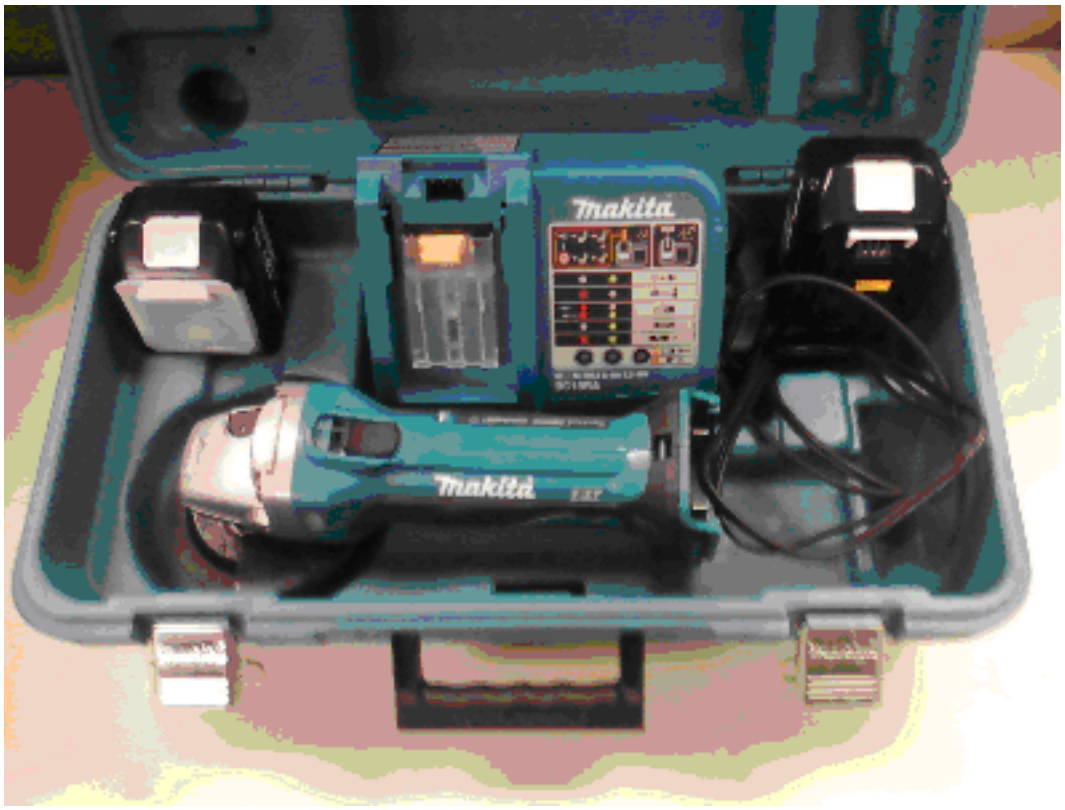


Figure 5. Cordless Angle Grinder Kit

A grinder kit typically contains the grinder, a battery, and charging unit. If the kit contains a grinding stone wheel, remove and dispose of it. Grinding stones are not to be used for XRF sample preparation. A rubber backup pad and a retaining nut are needed to use with sanding discs. (See picture below).

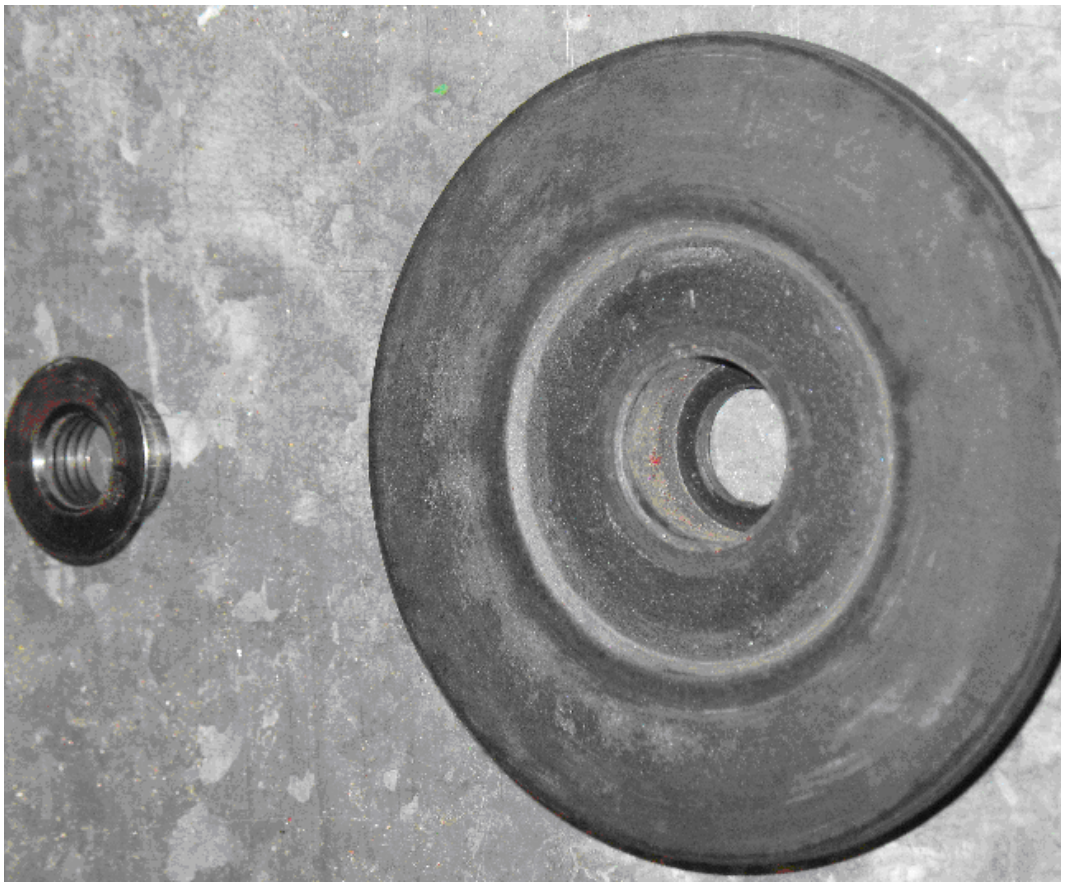


Figure 6. Rubber Backing Pad and Nut

In the US, sanding discs are 4.5 inch diameter and can be purchased in various grit sizes of 36 to 120. The surface abrasive can be one of the following materials: aluminum oxide, silicon carbide or zirconia alumina. The selection of sanding discs is covered in the next section.

Figure 7. Assembling the Grinder

Remove the battery before assembling the grinder, backup pad and sanding disc. Start by installing the backup pad onto the drive shaft of the grinder, or, with some backup pads. You will need to screw it onto the threaded shaft.

Next place the sanding disc over the drive shaft onto the backup pad. Hold the locking button on the reverse side of the grinder while tightening the retaining nut into the hole of the sanding disc.

Once the backup pad, sanding disc and locking nut are secured, reinstall the battery. The grinder is now ready for use.

Sanding Discs

It has been well tested and determined that samples can be easily contaminated by the abrasive material contained in and on a sanding disc. An example would be the increase in aluminum content of carbon steel after grinding the sample with a new aluminum oxide sanding disc. Aluminum from the aluminum oxide disc imbeds itself in the surface of the steel sample and an XRF would show an unusually high aluminum concentration.

Aluminum oxide is the most common abrasive surface used today. For most applications it will be safe to use aluminum oxide discs. But if test results for aluminum in any metal alloy are significantly higher than expected, switch to another type of abrasive disc. Also, when grinding aluminum, aluminum oxide discs tend to trap aluminum from the metal surface into the disc surface. Once this happens, the disc looses its efficiency and cross contaminates the next sample.

Silicon Carbide

Silicon carbide discs are a good alternative for aluminum oxide and the cost of a disc is only slightly higher than aluminum oxide. This adhesive type is best for grinding aluminum, copper and other soft metals.

Zirconia Alumina

Zirconia alumina discs are more expensive than aluminum oxide or silicon carbide but they last much longer and so may be the best investment. Few metal alloys have low additive levels of zirconium, so it is one of the safest abrasive types for general use.

One exception is the Aluminum alloy Al 7050 which is a near twin to alloy Al 7075 except for the ~0.1% Zr in 7050. Therefore, if 7075 is ground with Zr grinding paper it may be erroneously identified as Al 7050 due to the Zr transferred from the grinding disk to the surface of the Al 7075. s

Diamond Sanding Paper

Do not use diamond sanding paper for surface preparation in the field. Even after extensive and aggressive sanding with diamond paper, a metal surface will not be prepared properly. Diamond sanding paper is only recommended for removal of very light oxide coatings on flat surfaces such as analytical reference standards.

- Nickel, cobalt, and steel alloys should be ground using 36, 40, 50 or 60 grit discs. The selection of a grit size of 100 or la
- inum, copper alloys, and other softer metals should be ground using 60 or 80 grit discs.
- Grinding stones are not recommended because they will absorb surface material and transfer them onto the next surface ground.

Safety Rules

When using a grinder, follow these safety rules:

- When changing sanding discs, always remove the grinder battery to prevent accidental activation of the grinder.
- Allow the grinder to stop spinning before placing it on a flat surface.

- Replace any damaged or torn sanding discs immediately.
- Always wear impact eye protection to prevent eye damage from flying debris.
- Place small samples or standards in a clamping device when grinding to prevent accidental contact between the spinning disc and your hand.
- Use proper techniques and safety precautions when grinding beryllium, beryllium copper, lead, or titanium alloys.
- Always follow the safety instructions outlined by the grinder's manufacture as mentioned in the instruction manual..

Soil Sample Prep

Examine the site for differences in surface characteristics before sampling. Valid results depend on a sufficient and appropriate selection of sites to sample. Incorrect sample collection may give rise to misleading or meaningless results, regardless of the analysis method. Delineate sections with different characteristics and treat them as different areas. It may be desirable to subdivide larger areas even if they have the same characteristics to ensure a thorough examination. Make certain to label each bag thoroughly. Common information included on each bag includes the person and/or the company who collected the sample, the location and area where the sample was taken, and the date the sample was collected.

Prepared sample analysis is the most accurate method for determining the concentration of elements in a bulk medium using the instrument. Sample preparation will minimize the effects of moisture, large particle size, variations in particle size and sample non-homogeneity.

Note More sample preparation (drying, milling and sifting) will yield greater accuracy. The drier, finer, and more homogeneous the particles, the better the measurements.

Preparing Bulk Soil Samples

We recommends establishing a specific sample protocol. Following this protocol for preparing and testing samples is vital for achieving a level of accuracy comparable with laboratory results. The equipment you need to prepare samples is included in your kit. Among these are a mortar and pestle, several different sized metal sieves, and cups to hold the samples

.

CAUTION All test equipment must be kept clean to prevent contaminationof samples.

Cleaning Your Equipment:

The mortar, pestle, and grinding mill may be cleaned with dry paper towels. You can also clean the mortar, pestle, and the mill's container with water, but be sure each is absolutely dry before using them on another sample. The mortar and pestle may be cleaned by grinding clean, dry sand in the mortar. Use the short bristle brushes (included in your Soil Testing Kit) to clean the sieves. If you have an electric soil grinder in your kit, when the soil grinder blades wear out, unbolt the worn blades and replace them. Call the Thermo Sales Department at 1-800-875-1578 for replacement blades.

Note Using the soil grinder may artificially increase the amount of Fe in soil samples.

Sample Preparation

Prior to analysis, the material should be dry and well homogenized. Ideally, the entire sample should be dried to constant weight, sifted to remove gravel and debris, and ground or milled to a fine powder. Dry the sample if it is moist and cohesive. The sample can be dried in any of several ways. Choose one of the following:

- Oven dry the sample for approximately 2 hours at 150° C, until the sample reaches a constant weight. Note: Oven drying is inappropriate when volatile compounds may be present in the sample. For example, lead present as tetraethyl lead would be driven off by the heat of drying. Some forms of mercury and arsenic are volatile. Air drying will preserve more of these volatile substances.
- Air dry the sample overnight at room temperature in a shallow pan.
- Stir gently and warm the sample in a pan over a hot plate or burner.

Coning and Quartering

You may need to divide your sample at various times during preparation. Coning and quartering is a method for dividing the sample into homogenous quarters.

- Pour the dry material slowly and carefully onto a flat sheet or pan, forming a symmetrical cone. Divide the cone into equal piles using a flat thin-bladed tool, such as a knife or ruler. Divide these in half again.
- Now you have four samples, each one-quarter the size of the original and each more homogenous than the original.
- Grind the sample to break up dirt clods and/or paint chips.

WARNING Grinding and sifting dried samples produces dust. Even clean soil contains silica, which may be hazardous when airborne. Prepare all samples in a ventilated area; wear a mask, gloves, and an apron; and spread a drop cloth.

Sift using the #10 (2mm) mesh and separate out the larger pieces (stones, organic matter, metallic objects, etc. Examine the larger particles by eye but do not include in the sample. Grind the sample again so its particles will be finer and more homogenous. Use mortar and pestle, or an electrically powered grinding mill. Sift at least 10 grams of the sample through #60 (250 ?m) and #120 (125 ?m) mesh. Re-grind the un-passed material until the entire fraction is able to pass. Mix the resulting sample.

Placing the Sample in an XRF Sample Cup

Note The sample container should be a sample cup of a type that can be filled from the rear; that is, the side opposite the window (e.g. Thermo NITON Part Number 187-466). Thermo recommends using a 1/4 mil Polypropylene film (e.g. Thermo NITON Part Number 187-461). A supply of cups and films are included.

The container used to hold the sample will affect the accuracy of the measurement. Use a container with as thin-walled a window as is convenient and use the same kind of container and window for each sample. Consistency and careful attention to detail are keys to accurate measurement.

PLACE FILM

Place a circle of polypropylene film on top of an XRF sample cup. This film goes on the end of the cup with the indented ring. Thermo recommends preparing the cup ahead of time, if possible.

SECURE FILM

Secure the film with the collar. The flange inside the collar faces down and snaps into the indented ring of the cup. Inspect the installed film window for continuity and smooth, taut appearance.

FILL CUP

Set the cup on a flat surface film-window-side down. Fill it with at least five grams of the prepared sample, making sure that no voids or uneven layers.

TAMP SAMPLE

Lightly tamp the sample into the cup. The end of the pestle makes a convenient tamper.

PLACE FILTER

Place a filter-paper disk on the sample after tamping it.

STUFF CUP

Fill the rest of the cup with polyester fiber stuffing to prevent sample movement. Use aquarium filter or pillow filling as stuffing. A small supply of stuffing comes with your bulk sample kit.

CAP CUP

Place a cap on your cup.

LABEL CUP

Place a label on teh cup. Using a pen with indelible ink, write identifying information on the cup. Keep a record of the sample designation, the site and location, the date of the sample, and any other relevant comments.

Cup is ready for testing.

Preparing Liquids and Sludge

Liquids

Fill an XRF sample cup with the liquid to be tested (do not pad the sample with cotton). The cup must be full so it is best if some liquid is allowed to overflow when the cap is put on.

Sludge

Sludge can be placed directly into an XRF cup for screening. This is considered in-situ testing because no attempt has been made to prepare the sample. For more accuracy, the sludge can be dried, sieved, and ground. Prepare in an XRF sample cup and test the same way you would with a soil sample. For risk analysis, it is advisable to use a 60-mesh sieve to isolate and test only fine particles.

Preparing Mining Samples

Examine the site for differences in surface characteristics before sampling. Valid results depend on a sufficient and appropriate selection of sites to sample. Incorrect sample collection may give rise to misleading or meaningless results, regardless of the analysis method. Delineate sections with different characteristics and treat them as different areas. It may be desirable to subdivide larger areas even if they have the same characteristics to ensure a thorough examination. Make certain to label each bag thoroughly. Common information included on each bag includes the person and/or the company who collected the sample, the location and area where the sample was taken, and the date the sample was collected.

Prepared sample analysis is the most accurate method for determining the concentration of elements in a bulk medium using the instrument. Sample preparation will minimize the effects of moisture, large particle size, variations in particle size and sample non-homogeneity.

Note More sample preparation (drying, milling and sifting) will yield greater accuracy. The drier, finer, and more homogeneous the particles, the better the measurements.

Specimen Preparation - Fused Glass Disk

The samples need to be predried for 2-6 hours in 105°C depending on the moisture content.

1. Grind the dried samples to ~200mesh (74 ?m).

2. Calcination (Ashing) the sample

a. About 4-6 g of dry pulverized sample is calcinated in an alumina or platinum crucible in a muffle furnace at 1000°C for 1 hour.

b. The sample is cooled in a dedicator and loss on ignition (LOD) is calculated from weight difference before and after Calcination.

3. Weight 1.0g of calcinated sample into fusion crucible add 5.0 g of lithium tetraborate and 0.3 lithium fluoride, and 10-20 mg lithium bromide as a nonstick agent.

4. Fuse in a fluxer for at least 4 min in the flame.

5. The resulting disk is released from the mold, cooled, then presented to the spectrometer.

Specimen Preparation - Pressed Powder Briquette Preparation

1. Thoroughly remix the sample in its jar by rotating in a figure-eight motion with two hands

2. Weight 7.0g of sample into weighting boat by taking several separate gram-size portions then fine grind sample using a swing mill.

3. Add 2 small drops of propylene glycol on the top of the powder sample in the mill as a grinding aid, grind 4min at 1000rpm to obtain 10 ?m particle size.

4. Add 0.5g binder to the sample and continue grinding for 30sec more.

5. Brush the finely grounded samples into 31 mm aluminum sample cap and press at 50,000psi for 1 min.

CAUTION All test equipment must be kept clean to prevent contamination of samples.

Setting Up Beep Times

Selecting the Measurement Parameters icon allows you to set up Beep Times, enabling changes to the beep settings for various modes. This option allows you to change the beep settings for different modes independently. Select Mode you want to change, then the Measurement Parameters icon to set up your preferred beep times.

First Beep

This option allows you to change the seconds of delay before the First Beep. Select the screen button labeled with the number of seconds of delay for the First Beep. The Beep One Time editor will open. Clear the current number of seconds with the "C" button, then select the E button to enter the information.

Second Beep

This option allows you to change the seconds of delay before the Second Beep. Select the screen button labeled with the number of seconds of delay for the Second Beep. The Beep Two Time editor will open. Clear the current number of seconds with the "C" button, then select the E button to enter the information.

Third Beep

This option allows you to change the seconds of delay before the Third Beep. Select the screen button labeled with the number of seconds of delay for the Third Beep. The Beep Three Time editor will open. Clear the current number of seconds with the "C" button, then select the E button to enter the information.

Beep on Grade Match

Selecting this option will enable a special beep when the reading chemistry matches an alloy grade, and put a check mark in the box. Selecting the box again will remove the check mark and turn the beep off

Sorting the Custom Element Display

Select the Custom Element Display icon to configure sorting criteria used for analysis display. Select the mode you wish to change, then selecting the Custom Element Display icon opens up the Custom Element Display Screen.

On the left of the display are elements, each with its currently selected display option beside it to the right. The element list is ranked by importance, with the most important element on top, and each one lower down of less importance than the one above it.

By selecting an element and using the arrow buttons to the right of the list, you can change its ranking. Use the Up Button to move an element one rank closer to the top with each click. Use the Dn Arrow Button to move an element one rank closer to the bottom with each click.

Display Options

The Display Options Drop Down Menu allows you to change the display status of any element to one of three states:

- Normal - The standard state. Element displays only when the elemental value is greater than the limit of detection.
- Always - Always display the results for this element. Use this state for elements critical to all of your analyses.
- Never - Never display the results for this element. Use this state for elements which are unimportant to your work. This makes your instrument display less complex.

Select the element you want to change, then select the menu option corresponding to your choice of display status. The currently selected element is displayed in white on black.

Select the Save Button to save your current status as the new default. Select the Reset button to reset the settings to the previously saved state. Select the Close button to exit the screen without saving.

Report Settings

Under Electronics Metals, Plastics, and Test All Modes, A field called Report Settings is available. Selecting the triangle next to the Report Settings Field will open a pop up menu allowing you to choose between the three Report Settings Modes. Select the mode you wish to edit.

Changing the settings for one analysis mode will not affect the settings for other modes, and the configurations can be saved independently.

RoHS Option

When the RoHS Option is selected, clicking on the Pass and Fail values works as it does in any other Mode.

Detection Option

When the Detection Option is selected, Selecting the Pass/Fail field for that element acts as an On/Off Toggle, which will switch Pass/Fail mode between On and Off for the selected element. Selecting it again will reverse the toggle.

Consumer Products Option

When the Consumer Products Option is selected, clicking on the Pass and Fail values works as it does in any other Mode. In addition, the total of Cl+Br is also calculated and used for Pass/Fail Testing.

Max Measure Time

Under the Method Setup -> Measurement Parameters option is a field called Max Measure Time. Here you can set up the maximum time your analyzer will continue to analyze the sample. Select the Max Measure Time field, and a Virtual Numeric Keypad will pop up, allowing you to input a new Maximum Measurement Time in seconds. The default Max Measure Time is set to 300 seconds.

Minumum Test Time

Under the Method Setup -> Consumer Goods option is a field called Minimum Test Time. Here you can set up the minimum time your analyzer will continue to analyze the sample when using the Detection Option only. Select the Minimum Test Time field, and a Virtual Numeric Keypad will pop up, allowing you to input a new Minimum Test Time in seconds. The default Minimum Test Time is set to 60 seconds.

Virtual Keyboard

Whenever you see the Keyboard Icon, you can select it to bring up a Virtual Keyboard on your touch screen. Generally, selecting the keys on the Virtual Keyboard will type the corresponding character into the field. The exceptions are the meta-keys Del, Clear, Left, Right, Shift, Backspace, Cancel, and Enter.

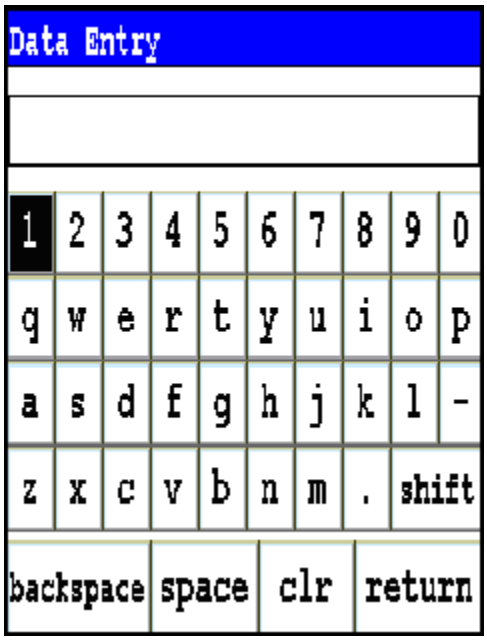


Figure 8. Virtual Keyboard

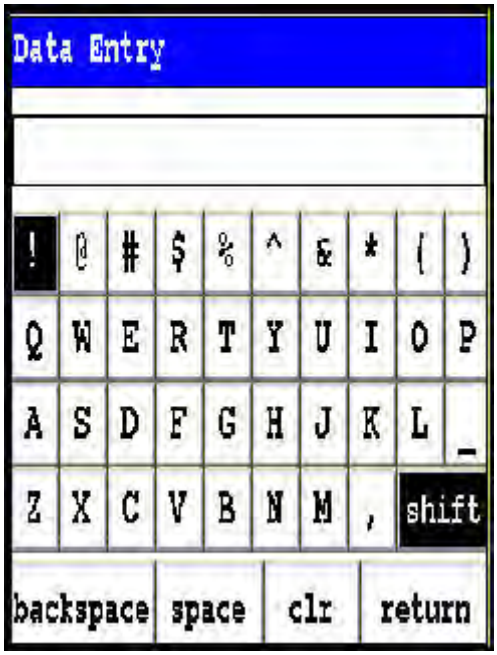


Figure 9. Shifted Virtual Keyboard

Del

Del is the Delete Key. Selecting this key will delete the character to the left of the cursor.

Clear

Clear is the Clear Key. Selecting this key will clear all characters from the field.

Left

Left is the Left Cursor Key. Selecting this key will move the cursor one space to the left.

Right

Right is the Right Cursor Key. Selecting this key will move the cursor one space to the right.

Shift

Shift is the Shift Key. Selecting this key will bring up the alternate, shifted keyboard. See Figure 1-1B. Selecting the Shift Key on the shifted keyboard will bring up the normal keyboard. See Figure 1-1A.

Backspace

Backspace is the Backspace Key. Selecting this key will delete the character to the right of the cursor.

Cancel

Cancel is the Cancel Key. Selecting this key will return you to the normal screen without inputting your changes into the field.

Enter

Enter is the Enter Key. Selecting this key will return you to the normal screen, replacing the former contents of the field with the changes you have made.

Setting Display Units

Select the Display Units radio buttons on the Set Display Units page to choose between ppm (parts per million) and percentage (hundredths of whole) displays when taking readings, and to change the Sigma value you want for the reading.

In the Display Units area, you can select between Percent composition and Parts per Million as the units displayed in a measurement, and you can change this setting independently for any mode. You can also change the Sigma for each of these modes independently. When you have changed the display units to the appropriate values, select the Close button to save these settings for use.

Changing Precision (Sigma Value)

Sigma is the symbol used for Standard Deviation, a measure of how much a set of numbers deviates from the mean. For example, each of the three data sets {0, 0, 14, and 14}, {0, 6, 8, and 14} and {6, 6, 8, 8} has a mean of 7. Their standard deviations are 7, 5, and 1, respectively. The third set has a much smaller standard deviation than the other two because its values are all close to 7. In a loose sense, the standard deviation tells us how far from the mean the data points tend to be. The number of standard deviations between the process mean and the nearest specification limit is given in sigmas. As process standard deviation goes up, or the mean of the process moves away from the center of the tolerance, the sigma number goes down, because fewer standard deviations will then fit between the mean and the nearest specification limit.

Confidence Intervals

Confidence intervals assume that the data are from an approximately normally distributed population - generally, sums of many independent, identically distributed random variables tend towards the normal distribution as a limit. Using this assumption, about 68 % of the values must be within 1 standard deviation of the mean, about 95 % of the values must be within two standard deviations, about 99.7 % must lie within 3 standard deviations, and about 99.99% of the values must lie within 4 standard deviations.

The greater the sigma value of the test, the more confident you can be that the sample is as it appears, but the more difficult and time consuming the testing must be to verify this. That's why it's important to use the most appropriate sigma value for the test. By adjusting the sigma value for each type of test, you can optimize the process for your needs.

Adjusting the Sigma Values

The sigma values are listed in the column headed with the Greek letter "sigma". The default value is 2 sigma. You can change this value by selecting the down arrow next to the value, which opens up a drop-down menu from which you can select the desired sigma value by clicking on it.

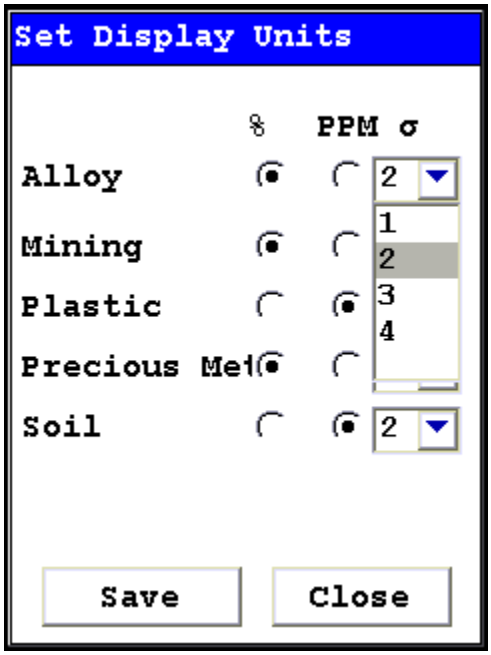


Figure 10. Selecting the Sigma Value

When you have changed the sigma values to the appropriate number, select the Close button to save these settings for use.

Adjusting the Element Range

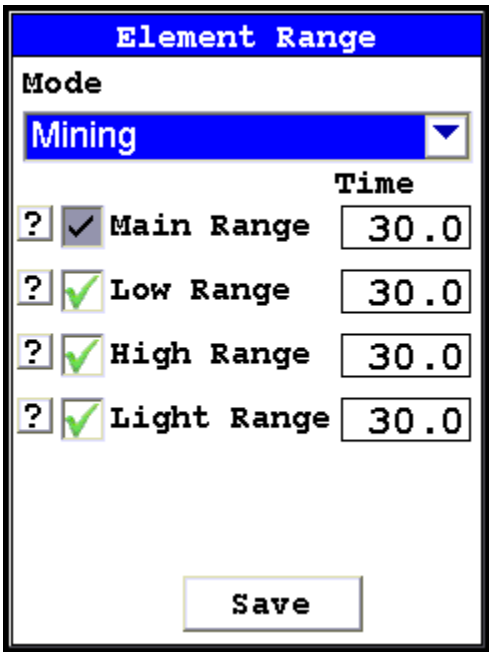


Figure 11. Adjusting the Element Range

Multi-Range tests are used to either preferentially excite specific elements for increased sensitivity, or to cover a wider element range than one Range alone can provide. Most modes, when enabled, will use several Ranges in sequence to produce a combined analysis result. In typical Metals analysis applications, Main Range is used for the analysis of most elements, Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr, High Range is is used to optimize the sensitivity for the elements from Palladium (Pd) through Barium (Ba), and Light Range is typically used in light element analysis. Multi-Range switching can be set to activate off time alone, or, when time switching is disabled, off settings in the General Metals grade library. In most modes, Low and Light Range add the capability to analyze light elements which cannot be efficiently excited by Mid Range.

Select the mode you wish to configure from the Mode Menu. You can set different configurations for different modes.

The Element Range Screen enables you to directly enable or disable any Range, or control the time that a Range alters the irradiation of the sample before auto-switching to another Range.

Select the checkbox next to the Range you want to use to determine exactly which of the Ranges contained in your Analyzer is used for sample testing. Selecting an empty checkbox will enable that range and place a check into the box as an indicator. Selecting a checked box will disable the Range and clear the box.

In typical metals analysis applications, Main Range is used for the analysis of most elements. You cannot deselect the Main Range in metals analysis.

Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr.

Select the Element List Button - labeled with a question mark - to display the Element List for that Range. This list shows the elements that the Range is best designed to detect.

Select the Range Time field for the intended range to change the switch time for that range. The Range Time Editor will appear. This enables you to set the number of seconds each enabled range is allotted before auto-switching will occur when needed during sample testing. Your analyzer will auto-switch from one range to another when the testing time for that range is greater than or equal to the time you have chosen, and the identified alloy is flagged as needing the switch in the Niton Alloy Library.

Select the C button to clear the current time, then from the virtual numeric key pad, select each digit you want to input, then select the E button to enter.

Setting the Date and Time

Figure 12. Setting the Date and Time

From the System Menu, select the Date & Time icon from the System Screen to set the date and time as needed for different time zones, daylight savings time, or any other reason. The date and time are factory preset prior to shipping. The clock is a 24 hour clock, so add 12 to PM hours - i.e. 1:13 PM would be 13:13.

Figure 13. The Date & Time Screen

When the Date & Time button is selected, the Date & Time Screen comes up on your analyzer's LCD Screen. You may change the Month, Year, Date, Hour, and Minute on your analyzer.

Changing the Month

To change the month, select the downward pointing triangle button next to the month displayed. A drop down menu will appear, listing the months of the year in order of appearance.

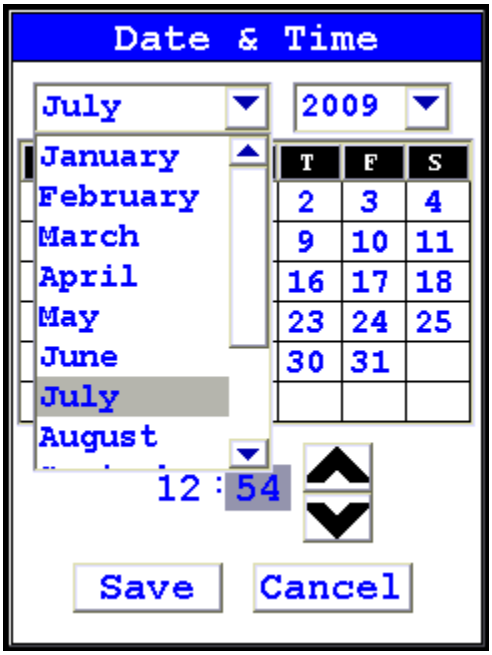


Figure 14. Month Drop Down Menu

Select the month you want from the drop down menu, using the vertical slider button to display hidden months. The display will change to show the month you selected.

Changing the Year

To change the year, select the downward pointing triangle button next to the year displayed. A drop down menu will appear, listing the years in order of appearance.

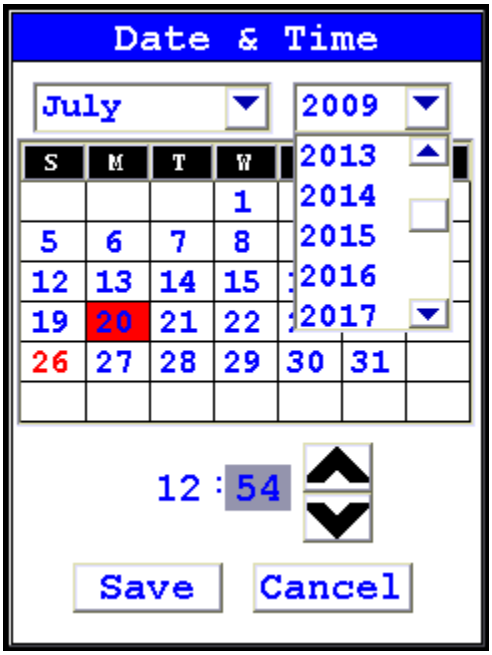


Figure 15. Changing the Year

Select the year you want from the drop down menu, using the vertical slider button to display hidden years. The display will change to show the year you selected.

Changing the Date

To change the date, select the date you want from the Date Selection Screen. The date you selected will be highlighted in red, while the old date will be shown in red numbers.

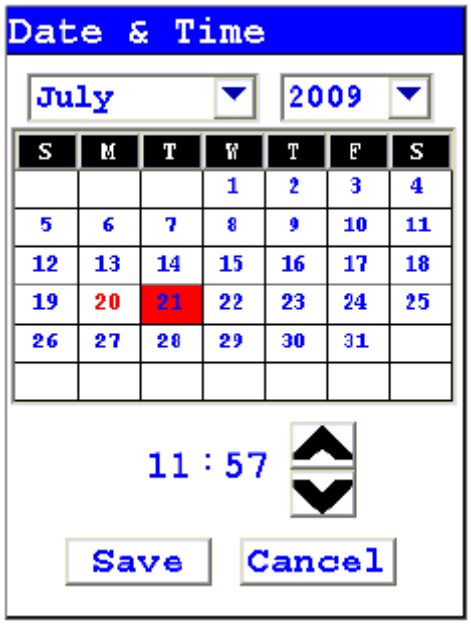


Figure 16. Selecting the Date

Changing the Hour and Minute

To change the hour, select the hour numbers. The hour numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the hour, or the Downward Pointing Chevron Button to decrement (decrease) the hour.

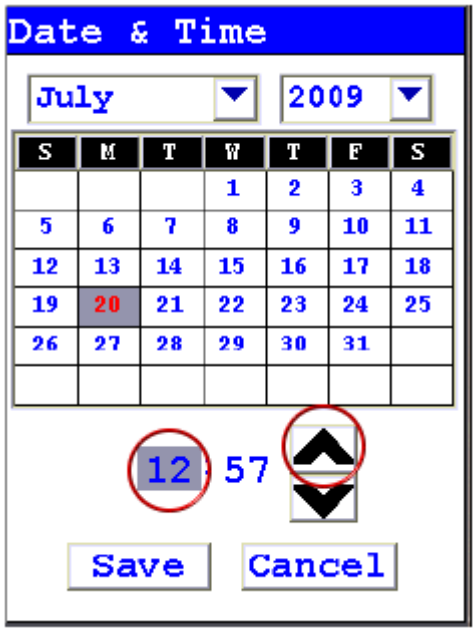


Figure 17. Changing the Hour

To change the minute, select the minute numbers. The minute numbers will be highlighted in gray. Then select the Upwards Pointing Chevron Button to increment (increase) the minute, or the Downward Pointing Chevron Button to decrement (decrease) the minute.

Figure 18. Changing the Minute

Saving Your Changes

To save your changes, select the "Save" screen Button. The display will return to the previous screen and the Date and Time will be saved.

Exiting Without Saving

To exit the screen without saving changes, select the "Cancel" Screen Button. The display will return to the previous screen and the Date and Time will not be saved.

Calibrating the Touch Screen

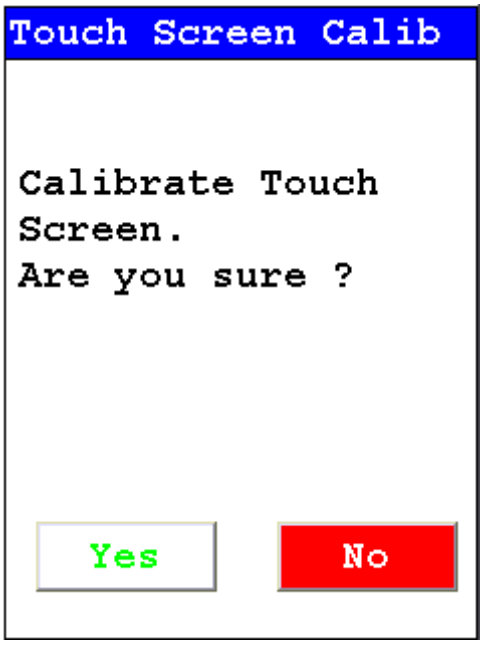


Figure 19. Initiating Touch Screen Calibration

Select the Calibrate Touch Screen button from the System Screen to re-calibrate the analyzer's touch screen display. This procedure establishes the display boundaries for the touch screen interface.

1. Select the Touch Screen icon.

2. The display will show a message asking you to confirm whether or not you want to calibrate your Touch Screen. Select the Yes button.

3. The display will show the message: "Calibrate Touch Screen". There will be a small cross in the upper left-hand corner of the display.

4. Tap on this cross with the stylus, and the cross will disappear and reappear in the upper right-hand corner of the screen.

5. Tap on the cross again, and it will reappear in the lower right-hand corner of the screen.

6. Tap on the cross again and it will reappear in the lower left-hand corner of the screen.

7. Tap on the cross once more, and you will be presented with a Confirmation Screen.

8. Select the Yes Button to confirm that the parameters are good. Select the No Button to start the process again.

9. Once you have confirmed the parameters, the System Menu will be displayed. The screen is now calibrated.

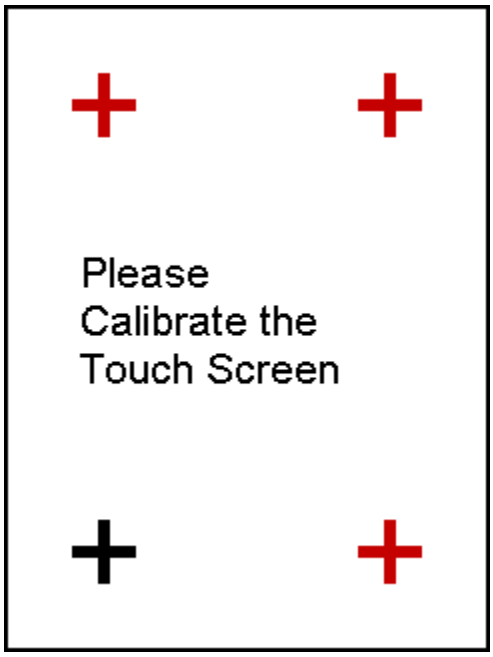


Figure 20. Touch Screen Calibration Crosses

The Touch Screen can be calibrated - and the system operated - with a USB mouse plugged into the USB ports in the rear of teh analyzer.

Data Management

Viewing Data

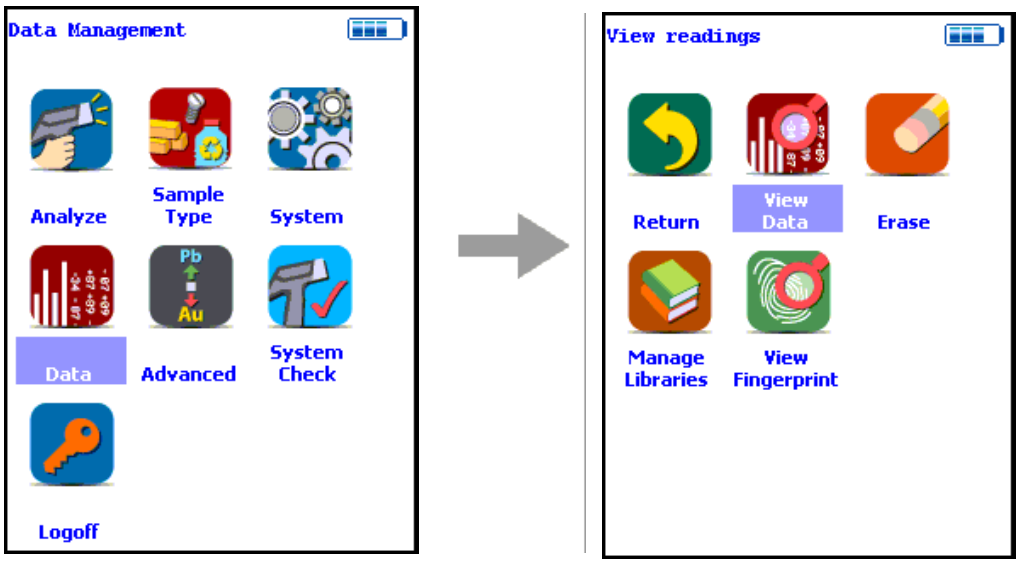


Figure 21. The View Data Menu Path

Use the Data Screen to view previously taken test result readings. When the View Data icon is selected, the Results screen of your most recent test is shown on the Touch Screen.

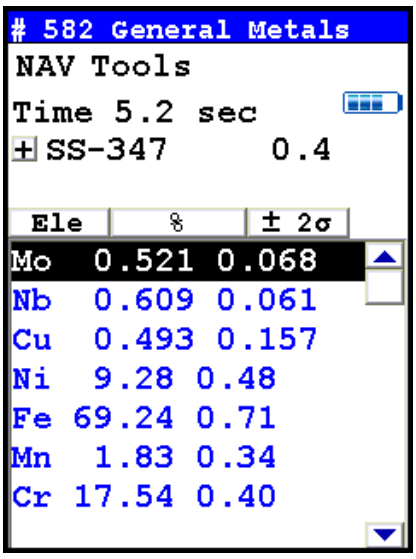


Figure 22. The View Data Screen

Using the buttons on the control panel, you may view different readings or additional data for individual readings. Your analyzer will display the standard screen analysis. Pressing the Down Button on the 4-way touch pad will display a complete scrolling elemental chemistry listing. Each press of the Down Button scrolls the screen down to the next element. You can also use the scroll bar along the right side to scroll or page through the elements.

Scrolling Down Through the Complete Listing of Elements

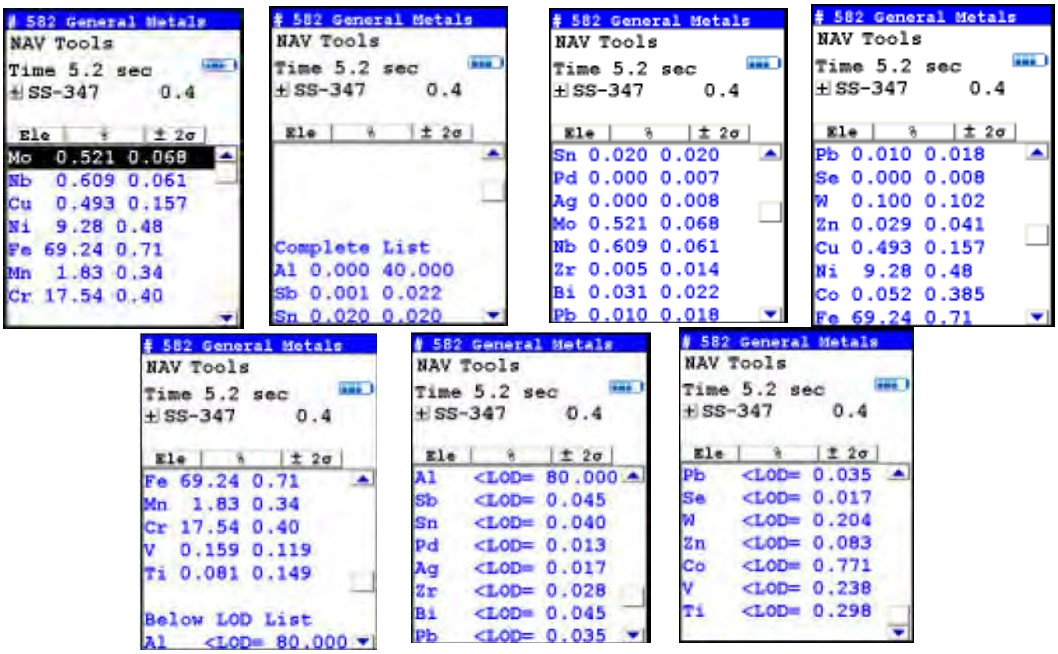


Figure 23. Complete Listing of Elements

Pressing the Left Button on the 4-way touch pad of your analyzer will display the previous reading, or if the first reading is currently displayed, the last reading. Pressing the Right Button on the 4-way touch pad will display the next reading, or if the last reading is currently displayed, the first reading in memory. Your analyzer can store up to 10,000 readings. You can also look at the complete x-ray spectra for each reading stored in the analyzer's memory.

Sorting Elements

You can sort element rows by various criteria in order to view your data in the manner you prefer. The Sort Buttons, which double as column headings, can be used to re-sort the data in different ways. The Data Screen always begins as a Standard Sort, as you have defined it. Selecting the appropriate Sort Button once toggles the sort order to High-to-Low. Selecting the Sort Button again toggles the sort order to Low-to-High. To return to the Standard Sort, select the Sort Button a third time.

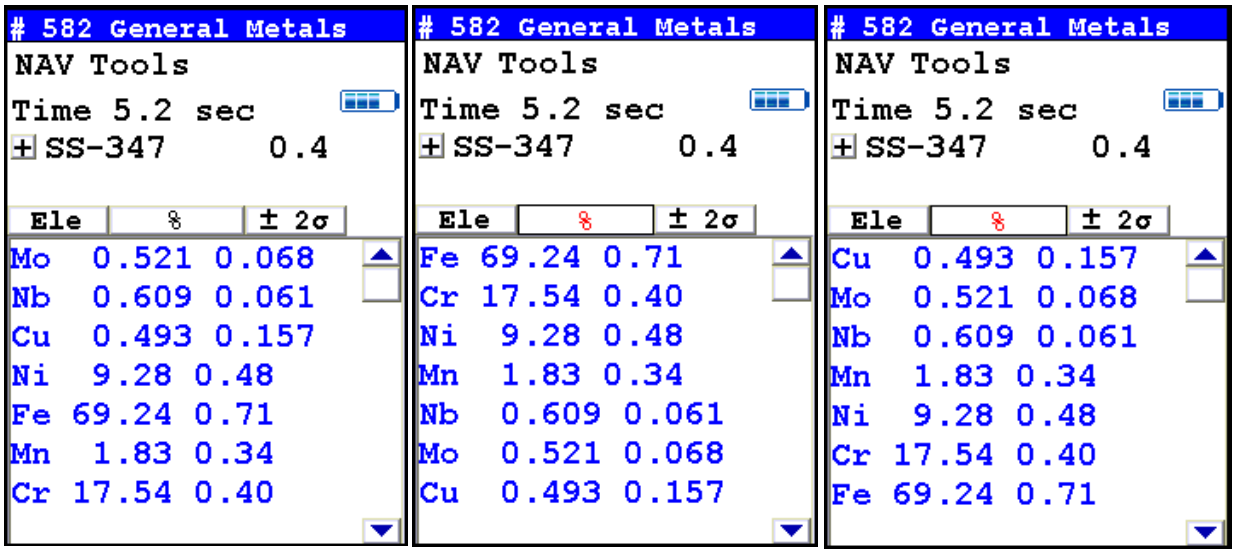


Figure 24. Standard, High-to-Low, and Low-to-High Composition Sorts

Element Sorts

Element sorts are performed alphabetically based on the element symbol.

Composition Sorts

Composition sorts are performed numerically based on the percentage of composition, i.e. from lowest to highest concentration, or by toggling again, from highest to lowest.

Error Sorts

Error sorts are performed based on the size of the error in the reading, i.e. from largest to smallest error, or by toggling again, from smallest to largest.

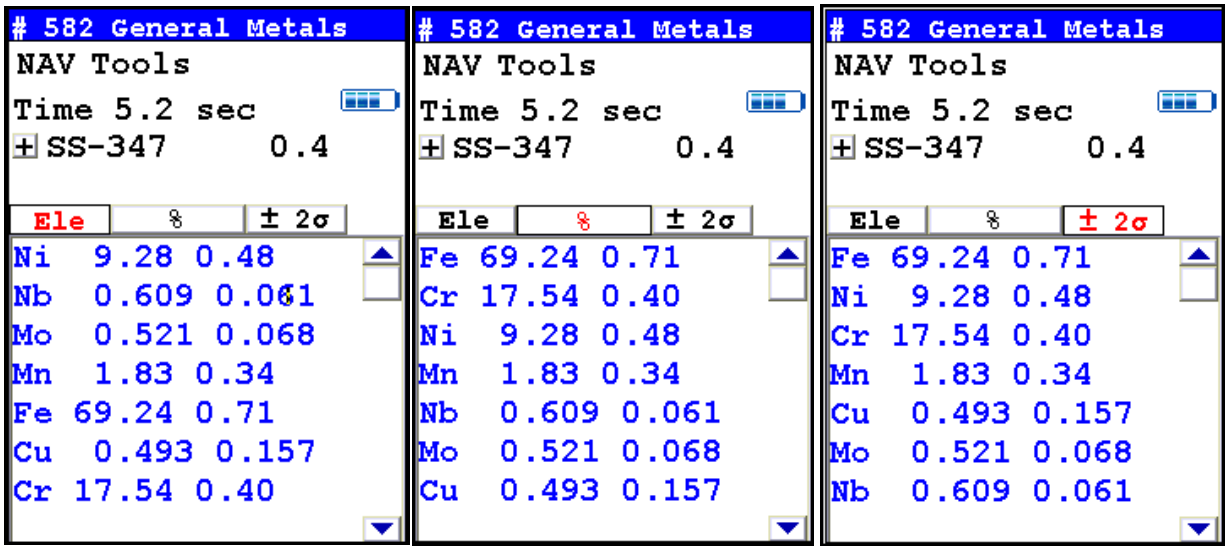


Figure 25. Element, Composition, and Error Sorts

Spectrum Graph

For any reading result, simply use the NAV Menu to gain access to the reading's spectrum graph. Selecting Spectra will show a graphed spectrum of this reading, called SpectraView. SpectraView can be a useful tool for rapid, qualitative analysis of a sample. See Viewing the Spectrum for details.

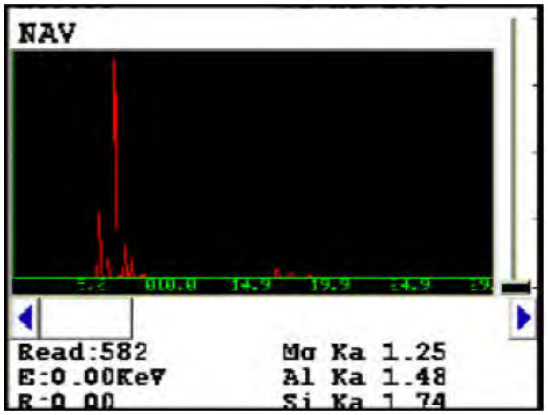


Figure 26. The SpectraView Screen

Viewing the Spectrum

SpectraView

SpectraView enables you to to visually inspect the fluorescent x-ray peaks obtained from any sample and qualitatively identify them using the on-board software. In SpectraView Mode, the spectrum is displayed using a linear energy scale along the x-axis, with the count rate autoscaled logarithmically on the y-axis so that the highest peak on the screen reaches the top of the scale.

How to Use SpectraView

You can access the SpectraView screen after taking a measurement in any mode, or while viewing a previous measurement, by selecting Spectra from the NAV Menu. Once you are in SpectraView, you can use the up and down positions of the 4-way touch pad to scroll through the spectrum, or you can tap on the spectrum display with the stylus to place the cursor at the point you tapped. The vertical cursor line indicates the current position along the spectrum.

Viewing the Information in SpectraView Mode

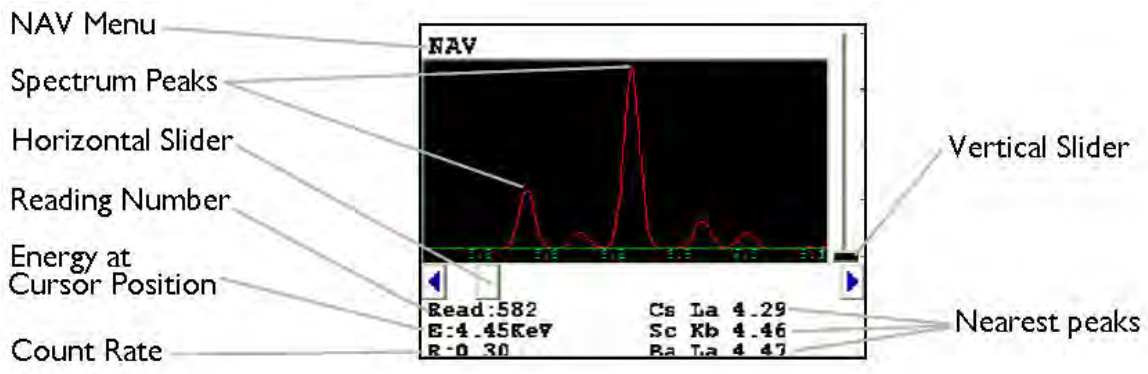


Figure 27. The SpectraView Screen

By default, the following information is shown along with the spectrum:

- The Reading number (Bottom Left) in the form "Read:x", where x is the Reading number.
- The position of the cursor on the energy scale (Bottom Left, under the Reading number), in the form "E: x.xx KeV", where KeV is kilo electron volts.
- The count rate (Bottom Left, under the energy position), in the form “R:x.xx”.

- Ka, Kb, La, Lb, and/or Lg peaks of the three elements closest to where your cursor is positioned on the energy scale (Bottom Right). This information is written with the element symbol first, followed by either Ka (K shell alpha peak), Kb (K shell beta peak), La (L shell alpha peak), La (L shell beta peak), or Lg (L shell gamma peak). An example would be "Al Ka 1.48." To determine if a given element is present, look at the count rate at that cursor position.

Note SpectraView cannot be used to determine exact element percentages in a sample.

Fitting the Spectrum

By using the touch screen, you can select parts of the displayed spectrum and zoom in. Touch and hold the stylus to the screen immediately before the area of the spectrum you wish to enhance, then - still holding the stylus to the screen - sweep it across the area of the spectrum you wish to see closer, lifting the stylus from the screen when you pass the end of the area of interest. The screen will display vertical lines to either side of the area of interest, delineating the boundaries of the area.

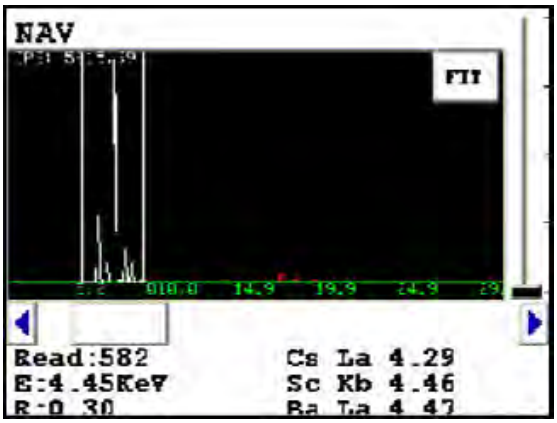


Figure 28. Delineating the Area of Interest

Select the FIT button in the upper right hand corner of the Spectrum to fit the area of interest to the display area.

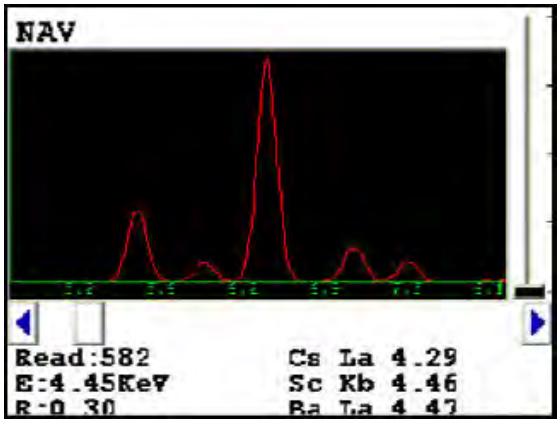


Figure 29. Area of Interest Fit to the Display

The view of the spectrum will change to show only the area of interest.

Multiple Ranges

SpectraView can display any individual spectra, including those obtained from multiple Ranges (filters) if you are using more than one Range. Use the NAV Menu to select which spectrum to view.

The Spectra1 choice will display the spectrum produced by the first Range.

The Spectra2 choice will display the spectrum produced by the second Range.

SpectraView Navigation

Use the left button on the 4-way touch pad to expand the spectrum, centered on the position of the cursor.

Use the right button on the 4-way touch pad to contract the spectrum, centered on the position of the cursor.

Viewing Fingerprints

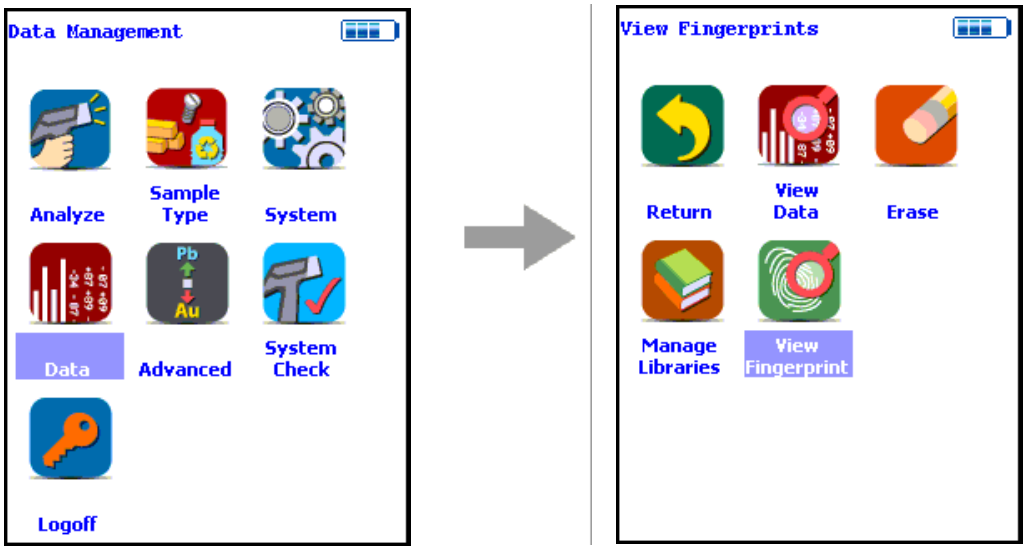


Figure 30. The View Fingerprints Menu Path

Select the View Fingerprints icon to view data saved as reference sample Fingerprints in Teach Fingerprint Mode. When the View Fingerprints icon is selected, the Results Screen of your most recent Teach Fingerprint is shown on the Touch Screen display.

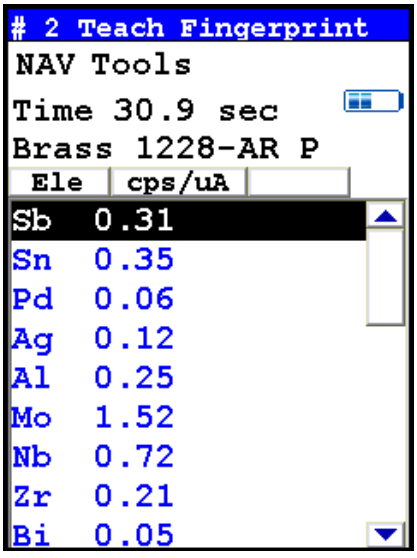


Figure 31. The View Fingerprints Screen

Erasing Data

The Erase All Data Screen

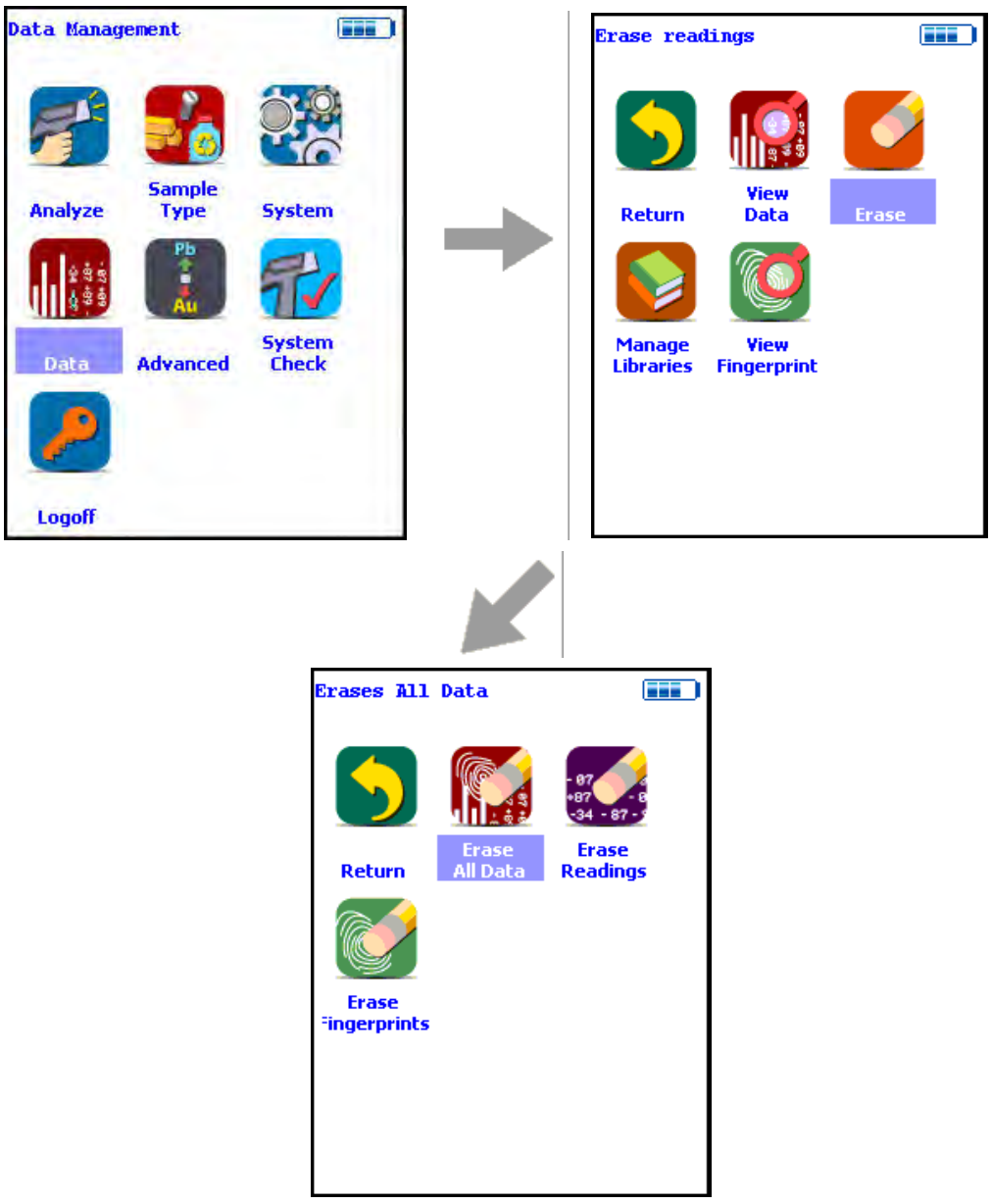


Figure 32. The Erase All Data Menu Path

Select the Erase All Data icon to erase all data, including signatures and readings, from your analyzer. Selecting the Erase All Data icon will bring up a confirmation screen asking you "Are you sure?" with options to select "YES" or "NO". Selecting the Yes Button will erase all data from your analyzer. Selecting the No Button will return you to the Erase Menu.

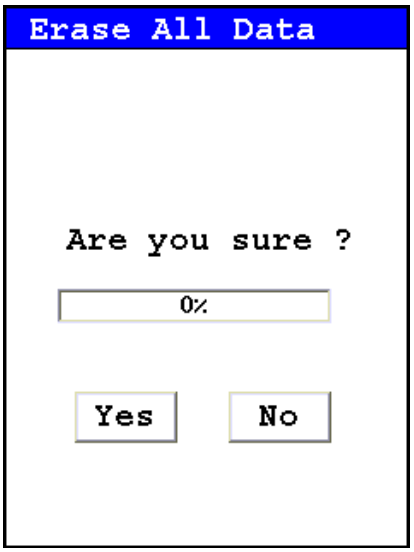


Figure 33. The Erase All Data Confirmation Screen

CAUTION Never turn off the analyzer while data is being erased!

WARNING Do not attempt to take measurements while downloading readings! This will generate an error requiring a system reset, and may corrupt your stored readings, requiring all stored readings to be erased.

The Erase Readings Screen

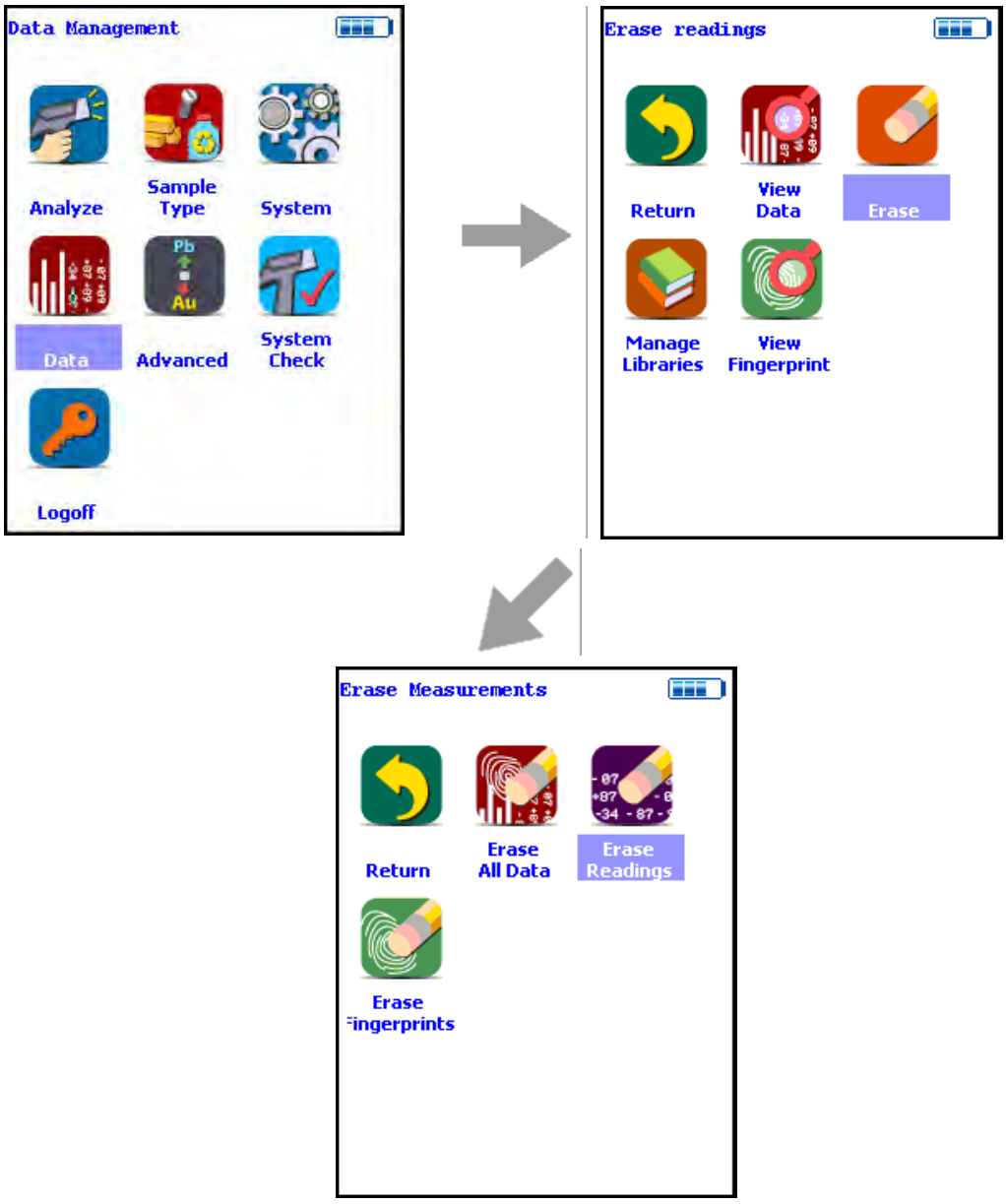


Figure 34. The Erase Readings Menu Path

Select the Erase Readings icon to erase all accumulated test readings from your analyzer. Selecting the Erase Readings icon will bring up a confirmation screen asking you “Are you sure?” with options to select “YES” or “NO”. Selecting the Yes Button will erase all test reading data from your analyzer. Selecting the No Button will return you to the Erase Menu.

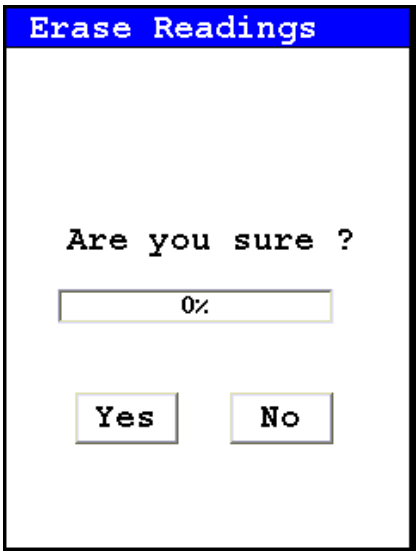


Figure 35. The Erase Readings Confirmation Screen

Note - We recommend that you download all your readings into an NDT file for recording purposes before erasing all data.

The Erase Fingerprints Screen

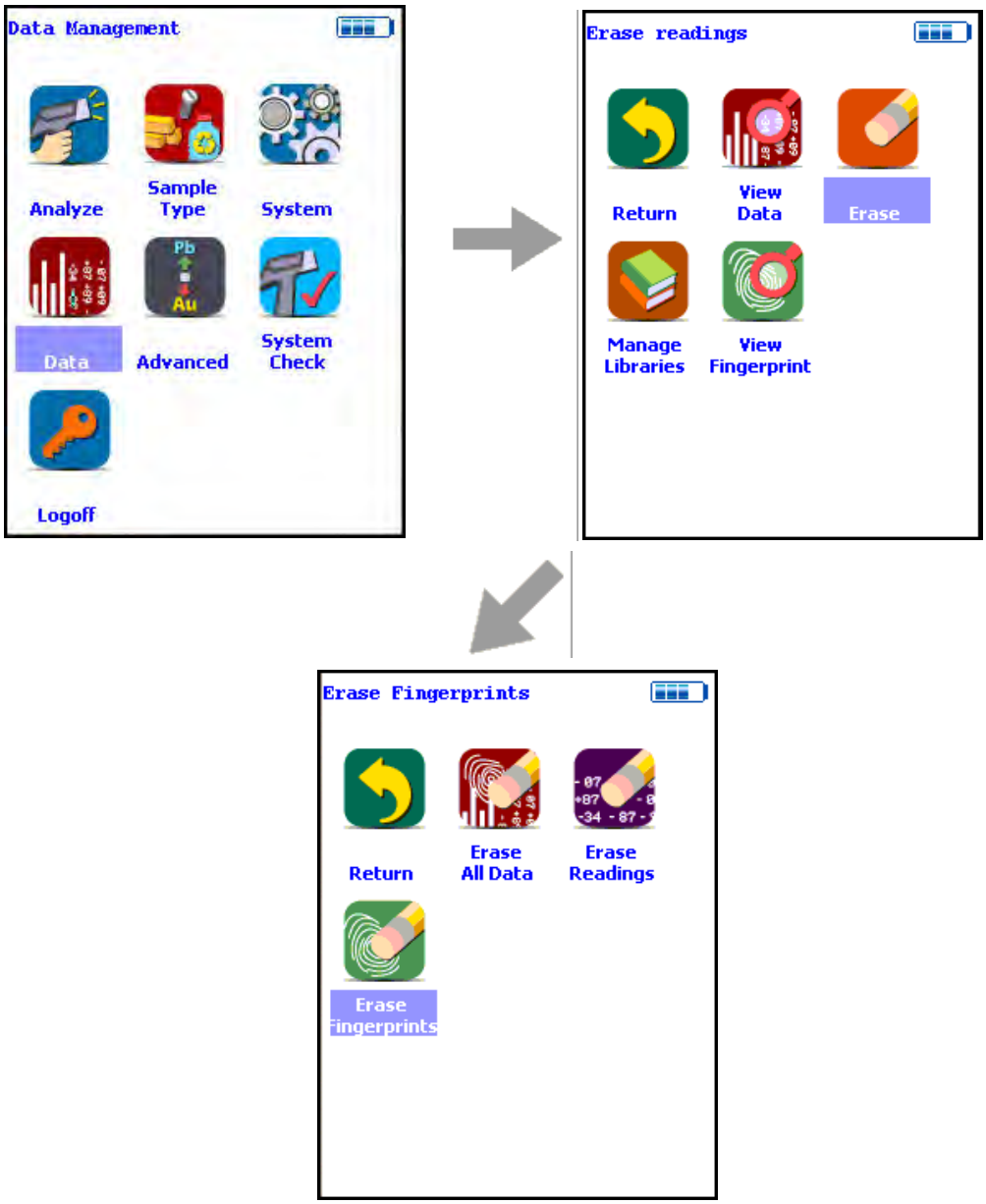


Figure 36. The Erase Fingerprints Menu Path

Select the Erase Fingerprints icon to erase all accumulated alloy fingerprints from your analyzer. Selecting the Erase Fingerprints icon will bring up a confirmation screen asking you "Are you sure?" with options to select "YES" or "NO". Selecting the Yes Button will erase all fingerprint data from your analyzer. Selecting the No Button will return you to the Erase Menu.

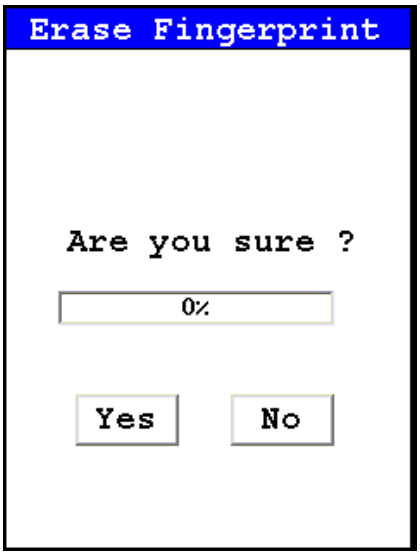


Figure 37. The Erase Fingerprints Confirmation Screen

Managing Libraries

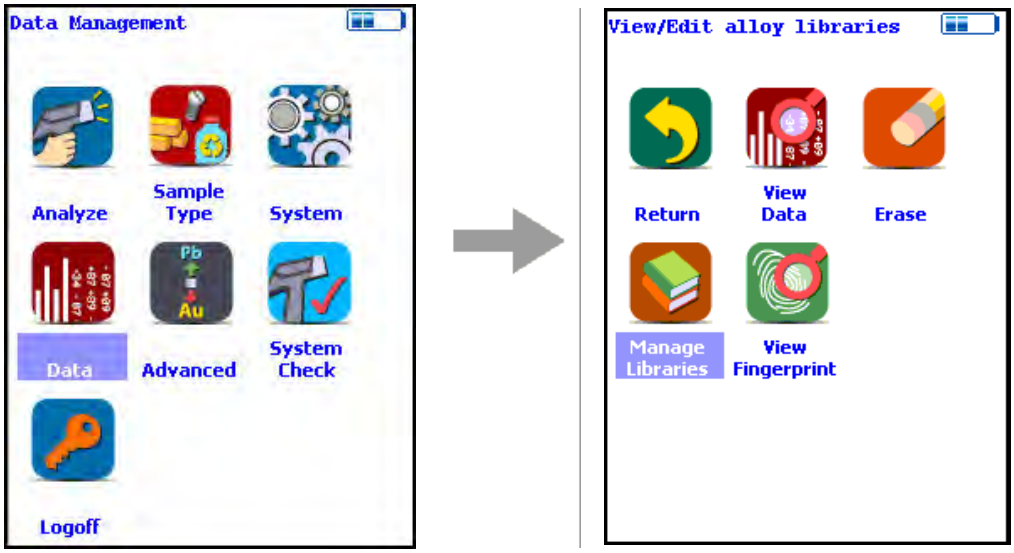


Figure 38. The Manage Libraries Menu Path

Select the Manage Libraries icon to access the Library Management Menu. The Library Management Menu allows you to view and modify data in the Primary Library as well as the currently loaded alternate libraries. Just select the library you wish to view or editfrom the list on screen.

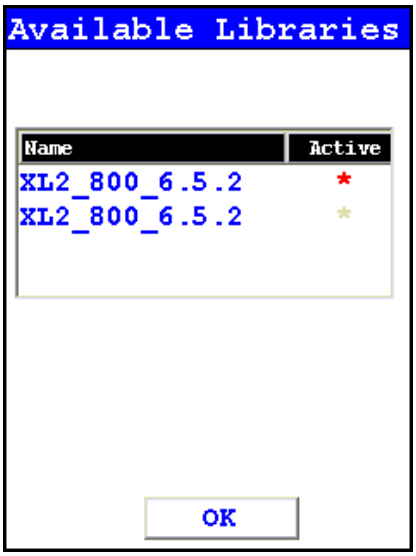


Figure 39. Viewing the Libraries

The entries in the Grade Library serve as a reference for chemistry based analysis. The library entries allow the analyzer to work properly "out of the box" without needing time-consuming pre-analysis.

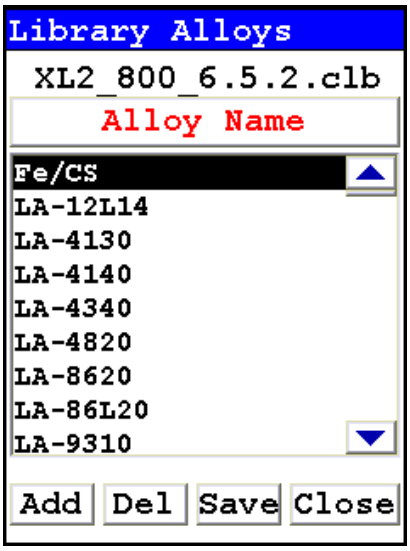


Figure 40. The Library Editor

Using the Library Editor

The Library Editor enables you to edit any library to conform to your specifications.

Alloy Name Button

Selecting the Alloy Name Button sorts the alloy list alphanumerically.

(Name in List)

Selecting the actual name of the alloy - i.e. “Fe/CS” - will bring up the Element Specification Screen.

Add Button

Selecting the Add Button will add a new alloy to the Library. First the Alloy Name Editor will appear, enabling you to enter the name of the new alloy.

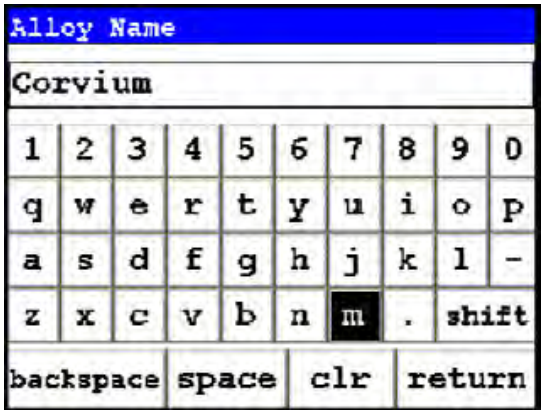


Figure 41. The Alloy Name Editor

The Alloy Name Editor is a standard Virtual Keyboard. Use it as you would any Virtual Keyboard. Hitting the return key enters the name into the alloy list. Select the name of the new alloy to bring up the Element Specification Screen and enter the specification of the alloy.

Del Button

Selecting the Del Button will delete the currently selected alloy. First a confirmation screen appears.

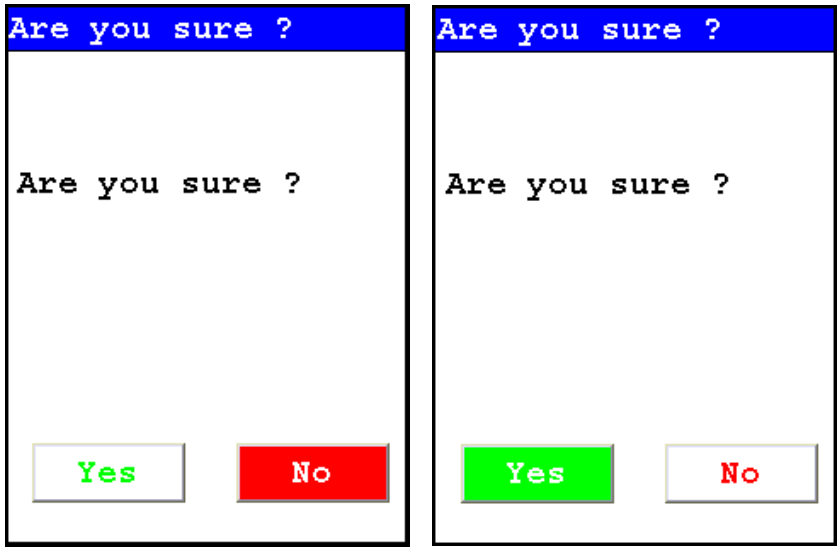


Figure 42. Confirmation Screen

Selecting the Yes Button will delete the alloy from the list. Selecting the No Button will return you to the Alloy List.

Save Button

Selecting the Save button will save the current Library.

Close Button

Selecting the Close button will close the current Library without saving it.

The Element Specification Screen

The Element Specification Screen allows you to edit the elemental content of any alloy.

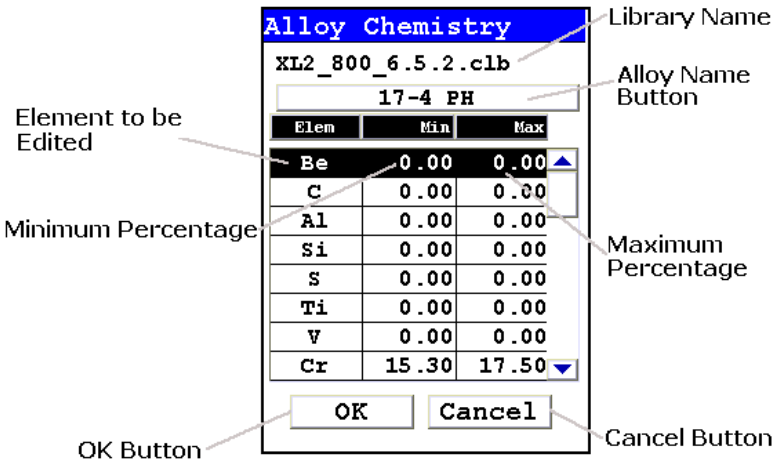


Figure 43. The Element Specification Screen

Library Name

This is the name of the library you are editing. Make sure you are editing the correct library before proceeding further.

Alloy Name

This is the name of the alloy you are editing. Make sure you are editing the correct alloy before proceeding further.

Element to be Edited

This is the element you need to edit for this alloy.

Minimum Percentage

This is the lowest amount of the element in question you want to be in the alloy. If the element in the analyzed sample is any lower, the sample will not be recognized as this alloy. Selecting the element minimum will open the Minimum Editor.

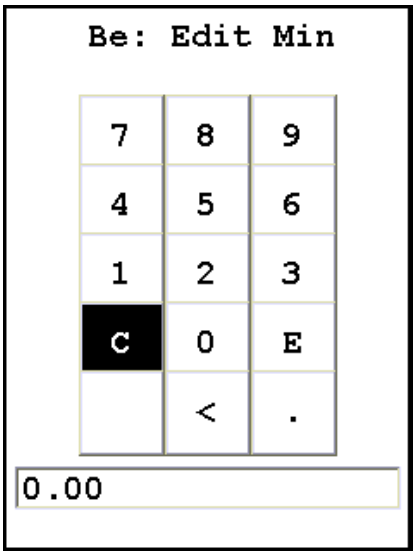


Figure 44. The Minimum Editor

This is a standard Virtual Numerical Keypad. The C Button will clear the current display, The < Button will backspace one space, and the E Button will enter this number as the minimum. After selecting the E Button, you will be returned to the Element Specification Screen.

Maximum Percentage

This is the highest amount of the element in question you want to be in the alloy. If the element in the analyzed sample is any higher, the sample will not be recognized as this alloy. Selecting the element maximum will open the Maximum Editor.

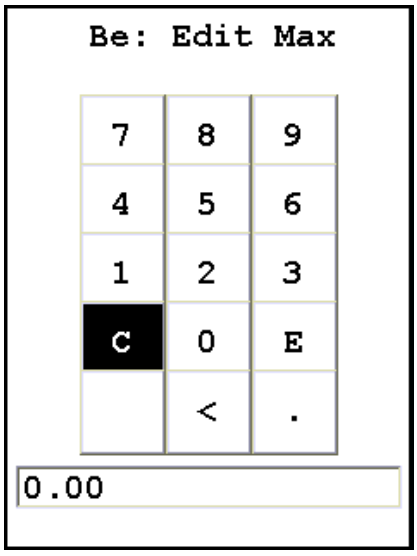


Figure 45. The Maximum Editor

This is a standard Virtual Numerical Keypad. The C Button will clear the current display, The < Button will backspace one space, and the E Button will enter this number as the minimum. After selecting the E Button, you will be returned to the Element Specification Screen.

OK Button

Selecting the OK button will save the edited library.

Cancel Button

Selecting the Cancel button will exit the Element Specification Screen for this alloy, returning you to the Library Editor.

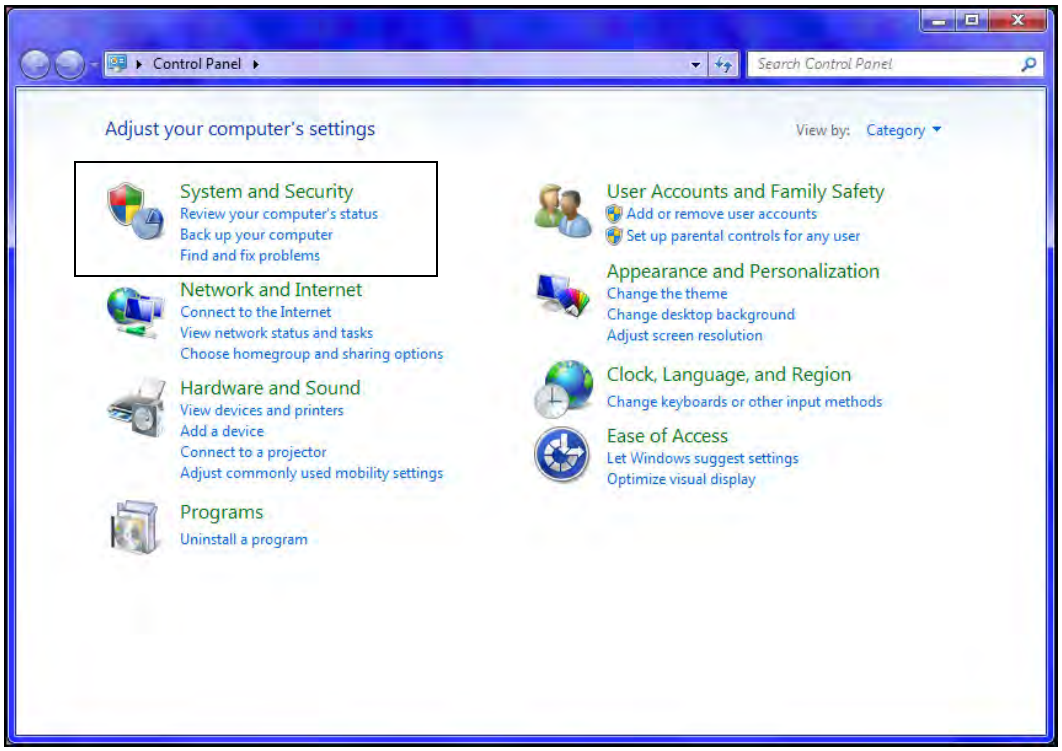
Connectivity

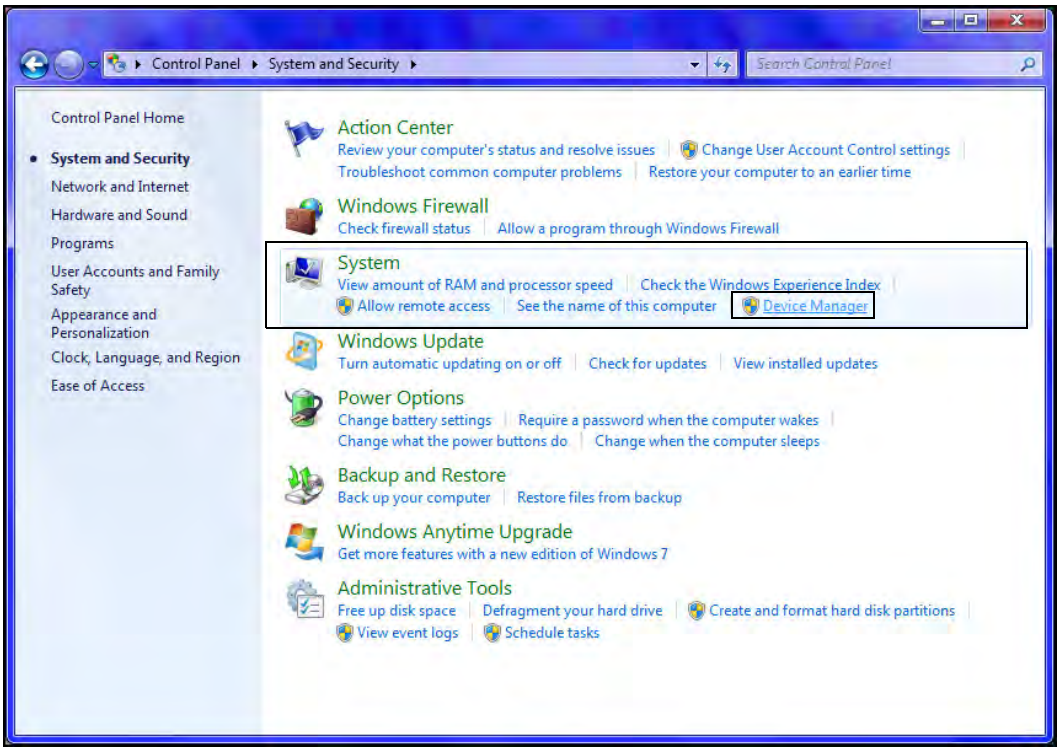
This section discusses how to connect your computer and your analyzer, for data transfer and integration, translation to other formats, data storage and security, as well as controlling your analyzer remotely through your computer. Connection can be achieved via USB, serial, and/or Bluetooth wireless means. Your analyzer comes with software which facilitates these uses, and works together with your analyzer to increase your productivity and efficiency.

Installing the Windows 7 USB Driver

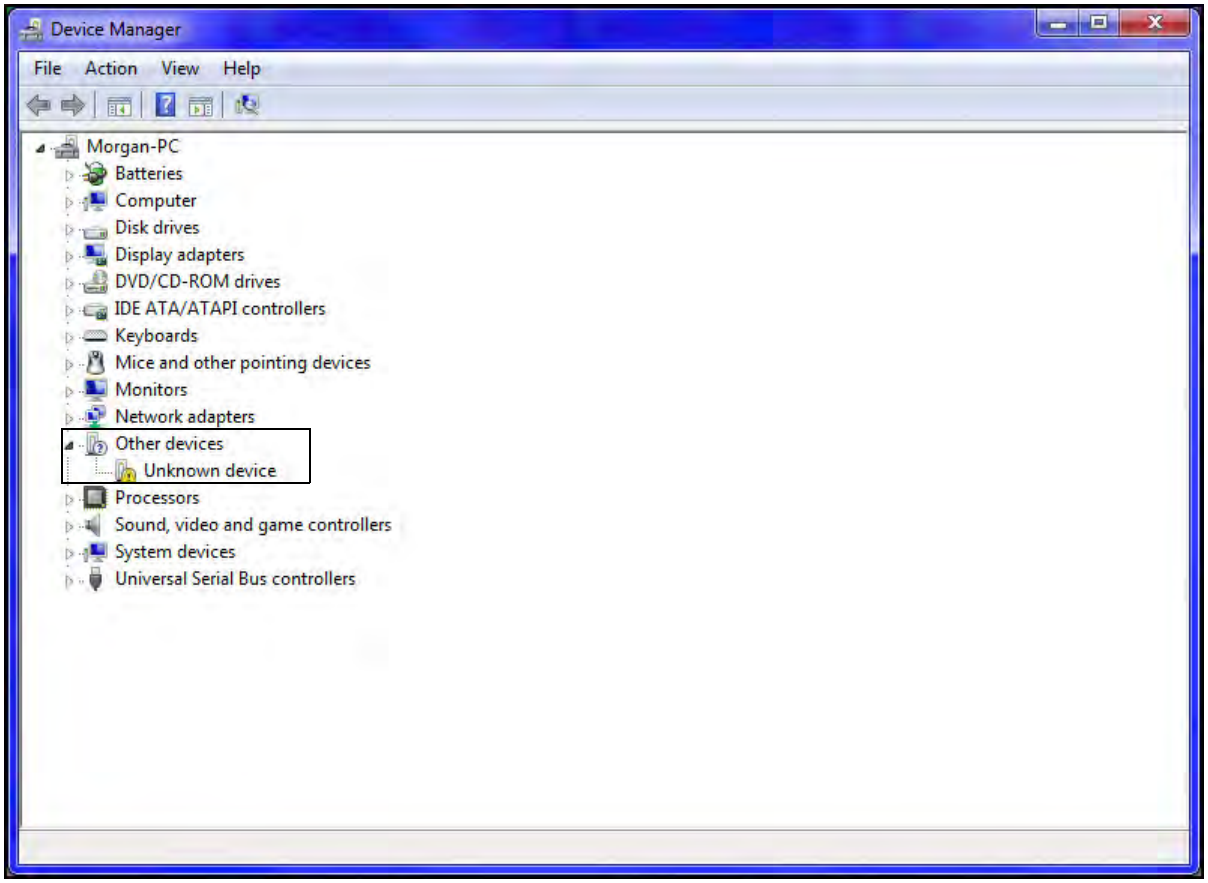
1. Insert the NDT CD and close out any dialogue box that pops up. The driver is located on this disk.

2. Click on “Control Panel” and locate the “Device Manager”. If it is not available directly under “Control Panel”, look under “System and Security” then “System”.



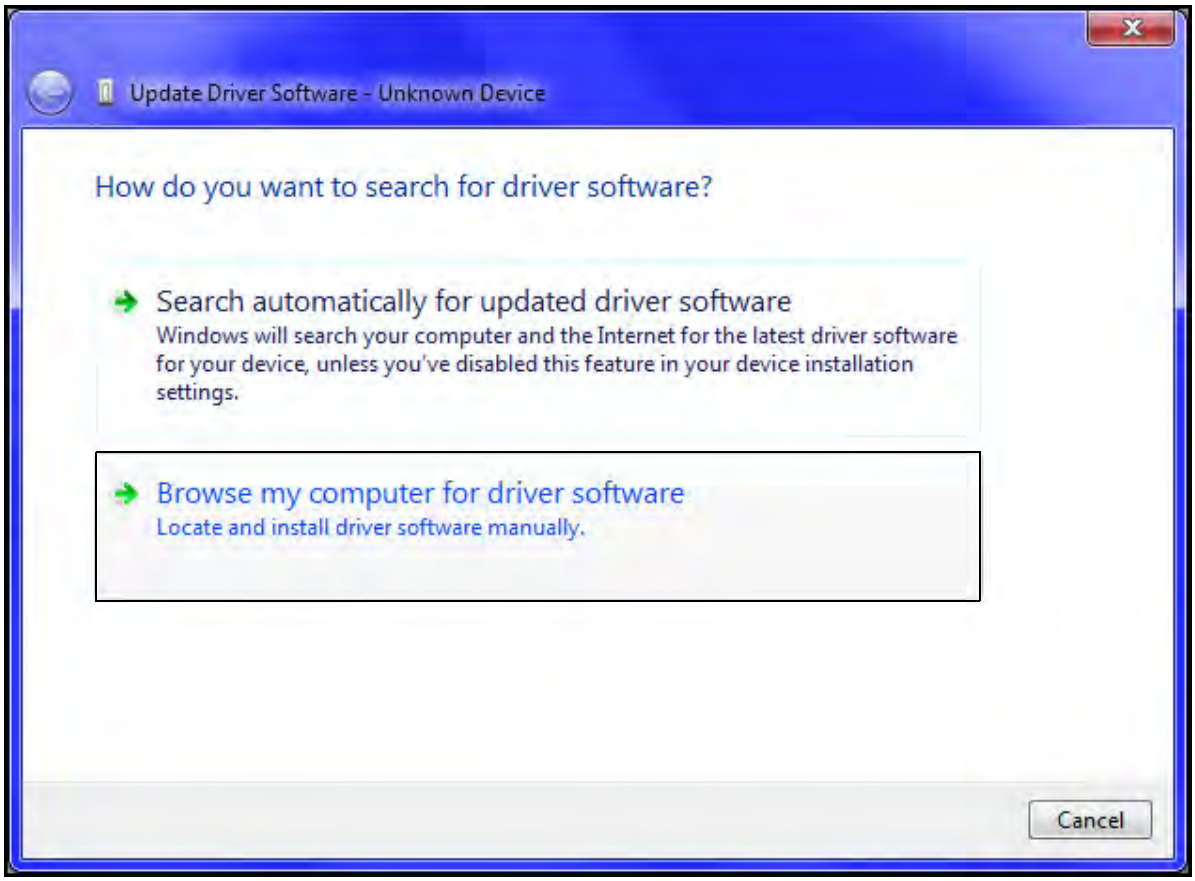


3. Open “Device Manager”
4. Plug in instrument using the USB cable provided
5. Message will appear “Device Driver Software Not Successfully Installed”
6. In “Device Manager”, “Unknown Device” will appear under “Other Devices”

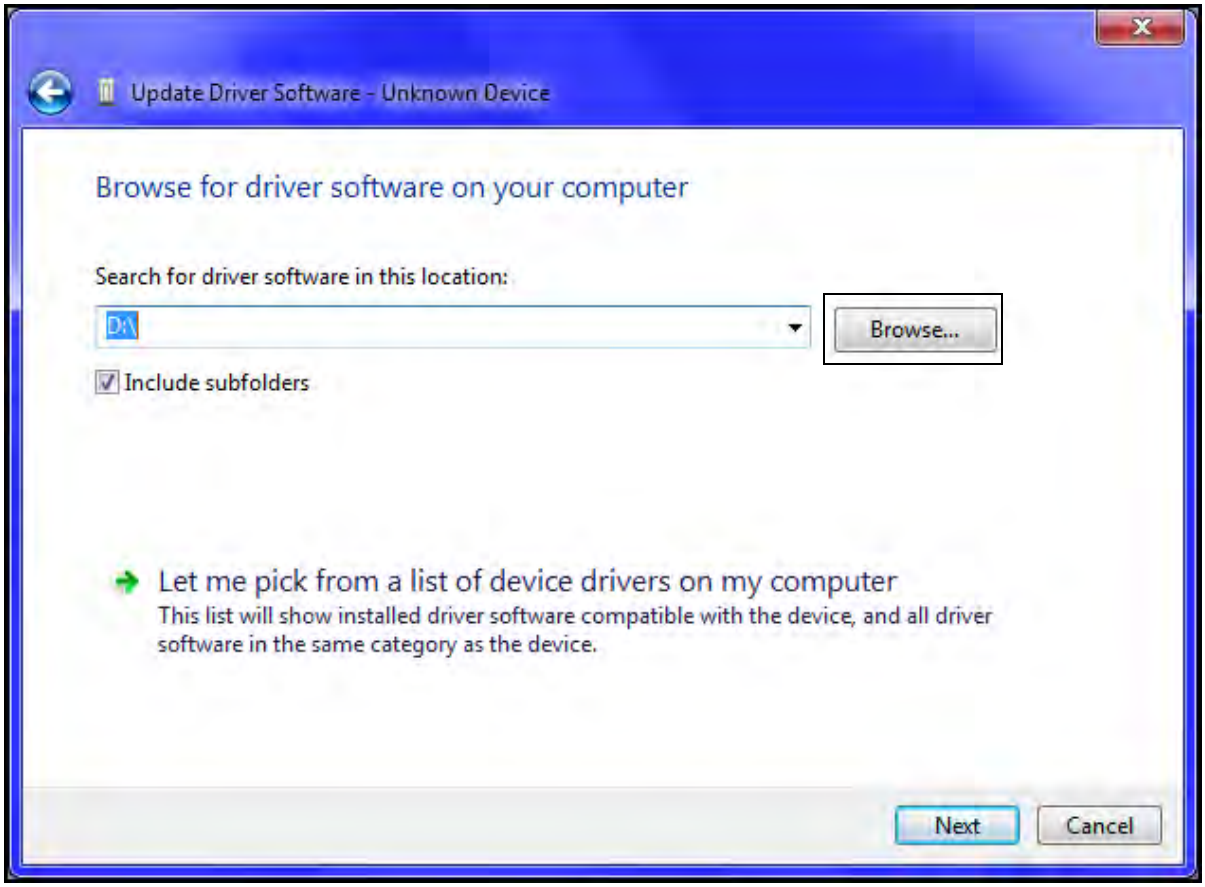


7. Right click on “Unknown Devices”; select “Update Driver Software”

8. Click on “Browse My Computer for Driver Software”

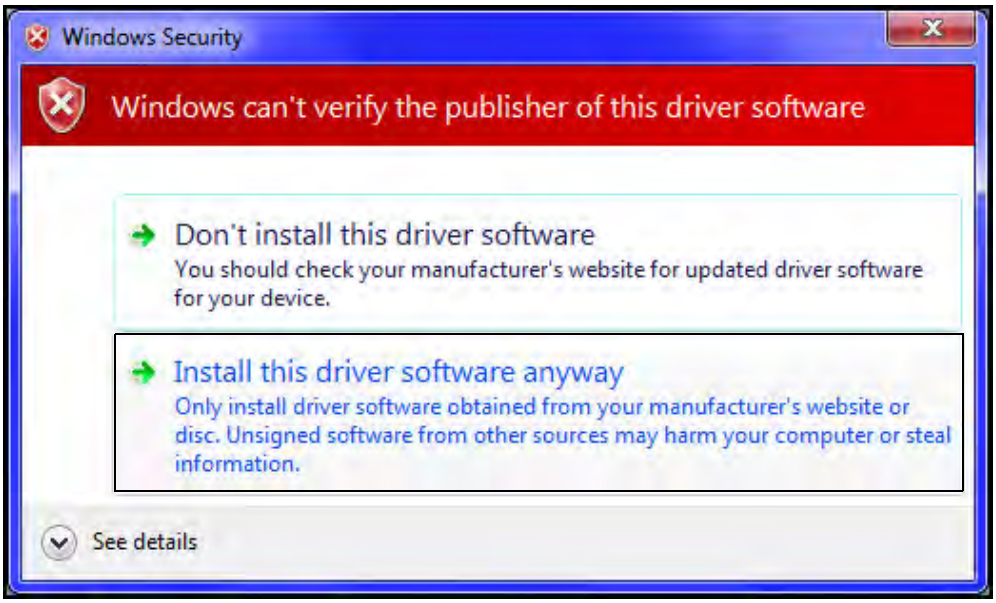


9. Click “Browse” button; select CD drive or the location of the driver if you are not installing from the NDT CD (recommended).



10. Click “OK”
11. Click “Next”

12. A Security Dialog Box will appear. Select “Install This Driver Software Anyway?”



13. Driver will install; close out.

Using Your Analyzer With Your PC

Using the Wireless (Bluetooth) USB Adapter to Connect the XRF Analyzer

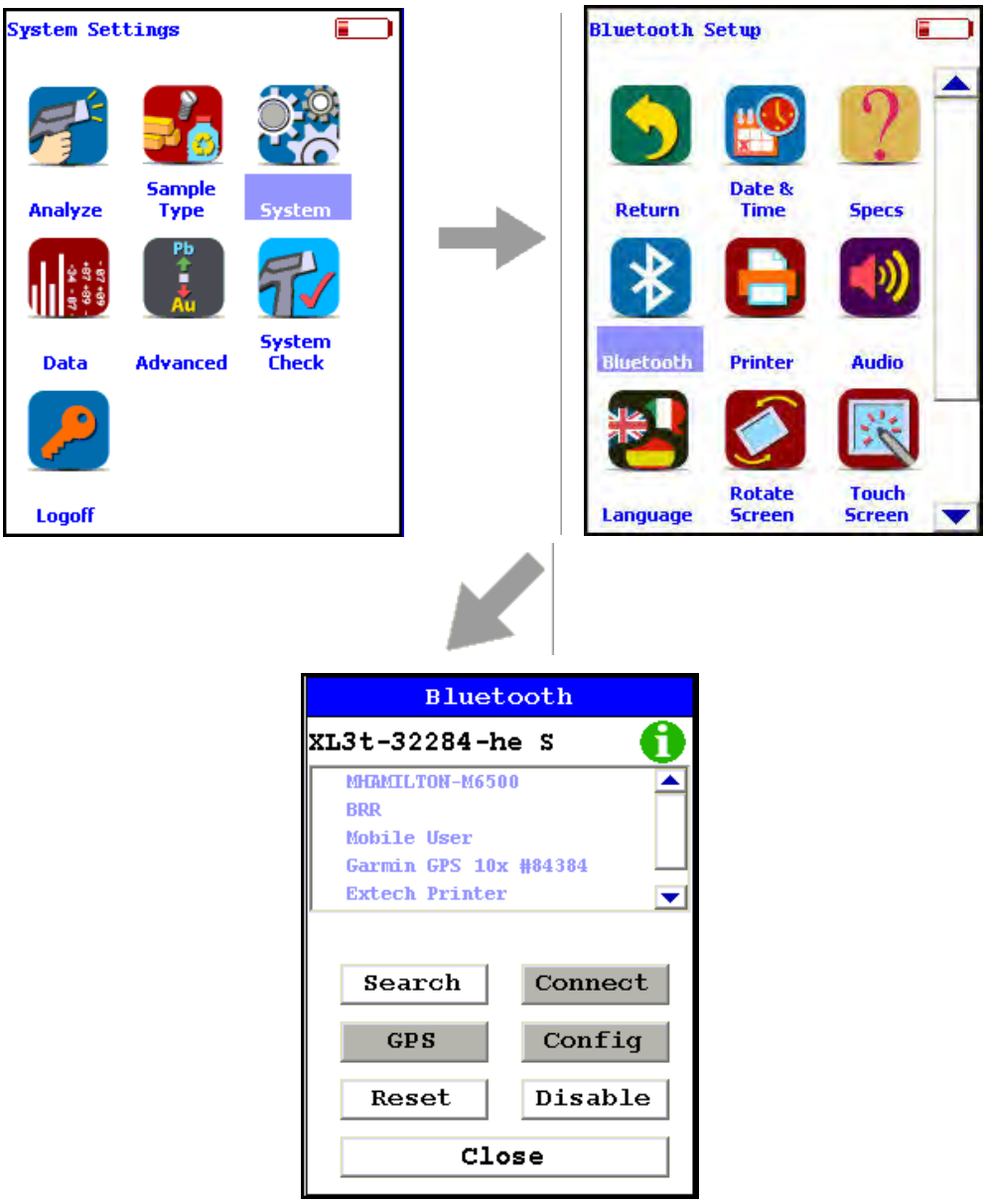


Figure 46. The Bluetooth Setup Menu Path

The USB adapter provided by Niton uses Bluetooth wireless technology. See Setting up Bluetooth to set up Bluetooth.

Select the Bluetooth icon from the System Screen to set up your analyzer for Bluetooth wireless connection.

i Icon

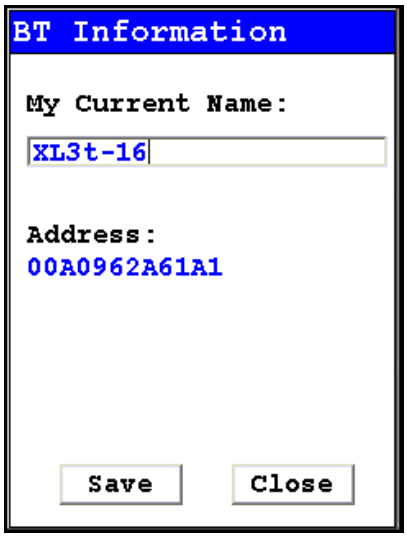


Figure 47. BT Information Screen

Selecting the i Icon in the top right of the Bluetooth Setup Screen will open the Bluetooth Information Screen. The Bluetooth Information Screen will supply the current name as well as the MAC address of your analyzer

Search Button

Selecting the Search Button will initiate a search for currently available Bluetooth devices in the area.

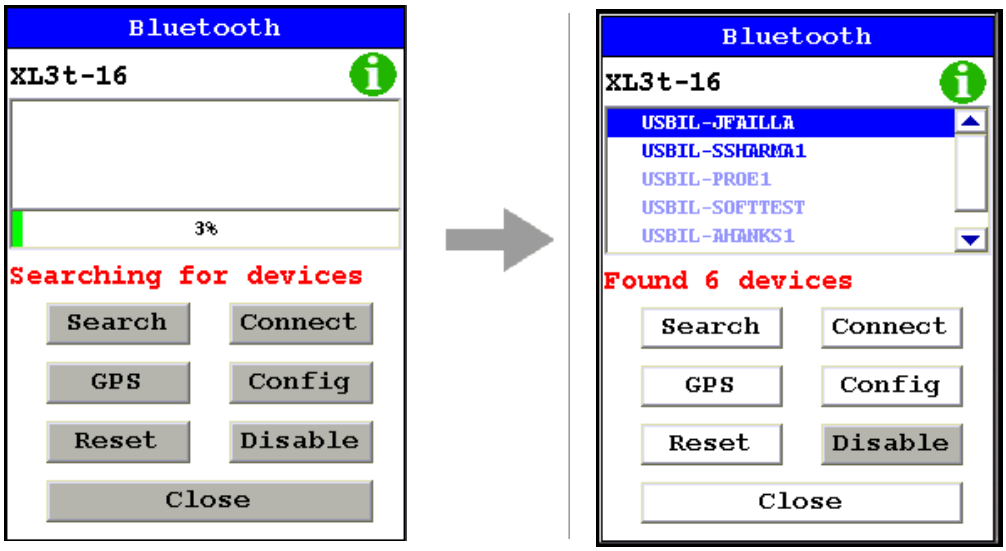


Figure 48. Bluetooth Searching

After the search, your analyzer will report which Bluetooth devices it has found in the main window of the screen.

You can connect your PC and analyzer two different ways, from the analyzer to the PC, and from the PC to the analyzer.

Connecting From Your Analyzer to Your PC

After searching, select the PC you would like to connect to from the main window of the screen.

Select the Connect Button. Your analyzer's screen will show connection progress.

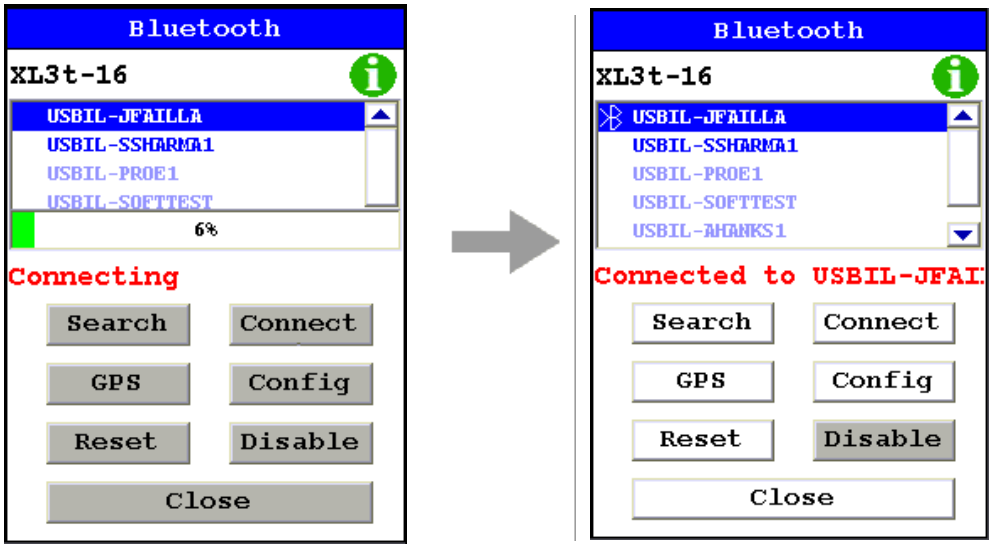


Figure 49. Connecting Via Bluetooth

Open the program you are attempting to connect to. Here we are connected to NDTr, running the analyzer remotely over COM 5

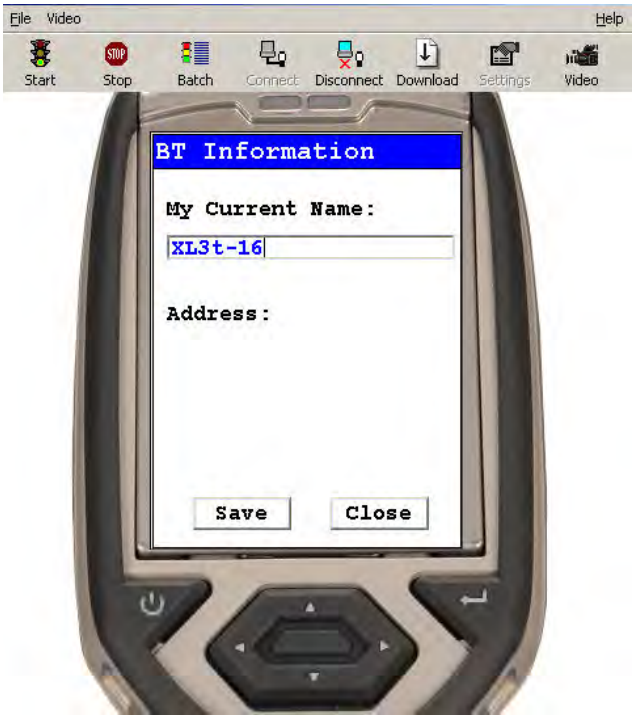


Figure 50. Connecting Via Bluetooth

Connect Button

Select a located Bluetooth device from the Search List, then select the Connect Button to connect to that device.

GPS Button

Select the GPS Button to download GPS data from a connected GPS Device.

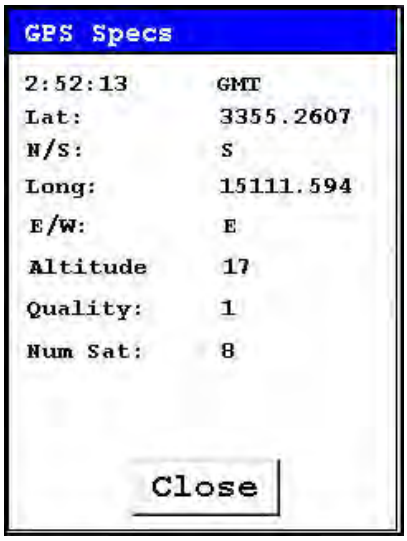


Figure 51. Bluetooth GPS Data Screen

Reset Button

Selecting the Reset Button initiates a Bluetooth reset.

Config Button

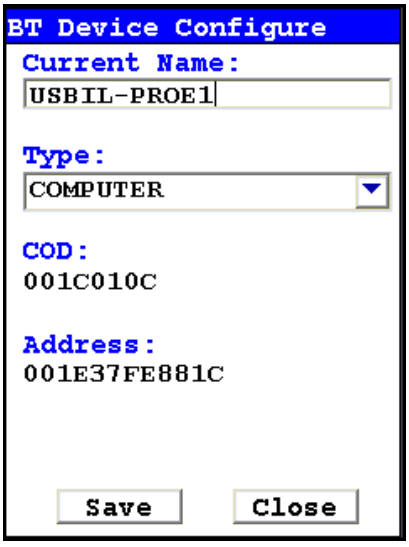


Figure 52. Bluetooth Config Screen

Selecting the Config Button will load the Config Screen. This screen enables you to se the name of the currently selected Bluetooth device, change the type of device, see that device's COD Number, and see that device's MAC Address.

Changing the Bluetooth Device Type

Select the Down Arrow button next to the Type field to change the type of device connected. Select the proper type from the drop down menu. Select the Save button to save this configuration. Select the Close Button to exit without saving.

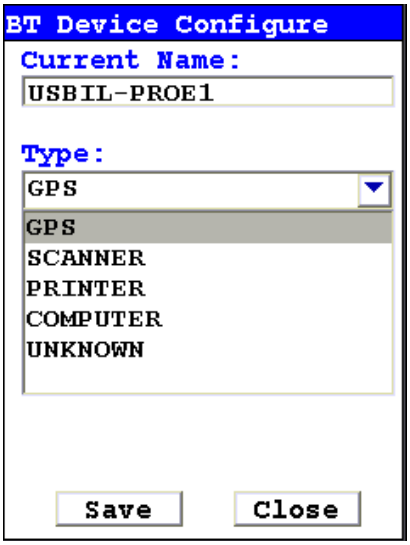


Figure 53. Changing Bluetooth Device Type

Disable/Enable Button

Selecting the Disable Button will disable Bluetooth neetworking and change the button to Enable. Selecting the Enable Button will restart Bluetooth networking and change the button back to Disable.

Close Button

Select the close button to exit from Bluetooth setup.

Using a USB Cable to Connect Your Analyzer

To connect the XL2 XRF Analyzer to your PC using the USB cable:

1. Insert the Standard USB connector on the USB cable into a USB port on your computer.

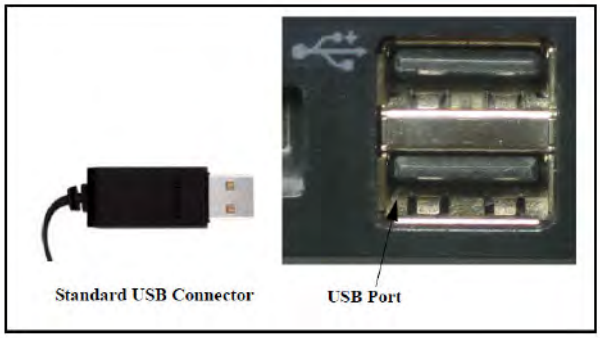


Figure 54. Standard USB Connector

2. Open the Port Cover on the XRF Analyzer.
3. Turn on the analyzer and insert the mini USB connector on the USB cable into the USB port in the handle of the XRF Analyzer.
4. Upon initial installation, insert the NDT disk located behind the foam in your case. Follow the prompts and install the USB driver located on the disk.

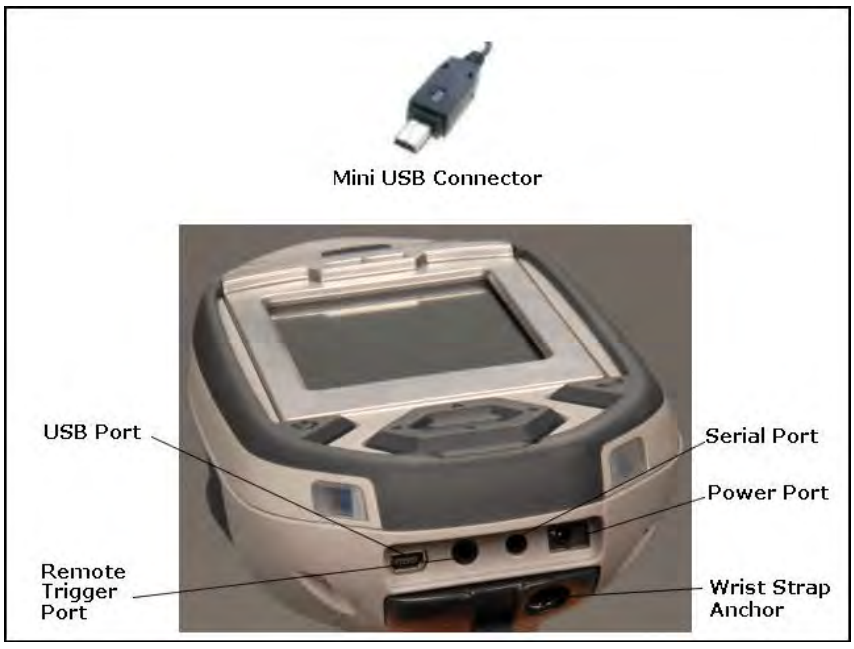


Figure 55. Mini USB Connector

Downloading Data

Standard Download

To download data you have collected offline:

1. Make sure that the XRF Analyzer is connected to your computer. See "Using Your Analyzer With Your PC" on page 109for more information.

2. Turn on the XRF Analyzer. See the manual for the XRF Analyzer for more information.

Note Wait at least 30 seconds after turning on the XRF Analyzer to begin downloading files. The System Start screens do not allow downloading.

3. Start Niton Data Transfer.

4. Click the Download button. The Download dialog box will open.

Figure 56. The Download Button

5. In the Download dialog box, Select the Test button to test the serial connection to the Analyzer.

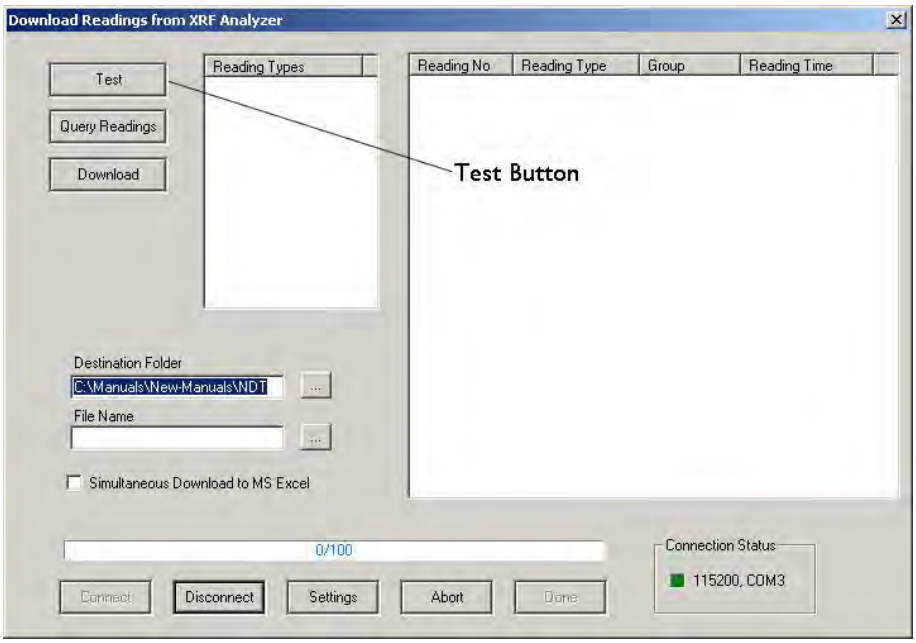


Figure 57. The Test Button

6. You should get a pop-up window informing you that the connection tested successfully. If the test fails, there is a problem with your serial port setup.

Figure 58. Connection Success Window

7. In the Download dialog box, click the Connect button.

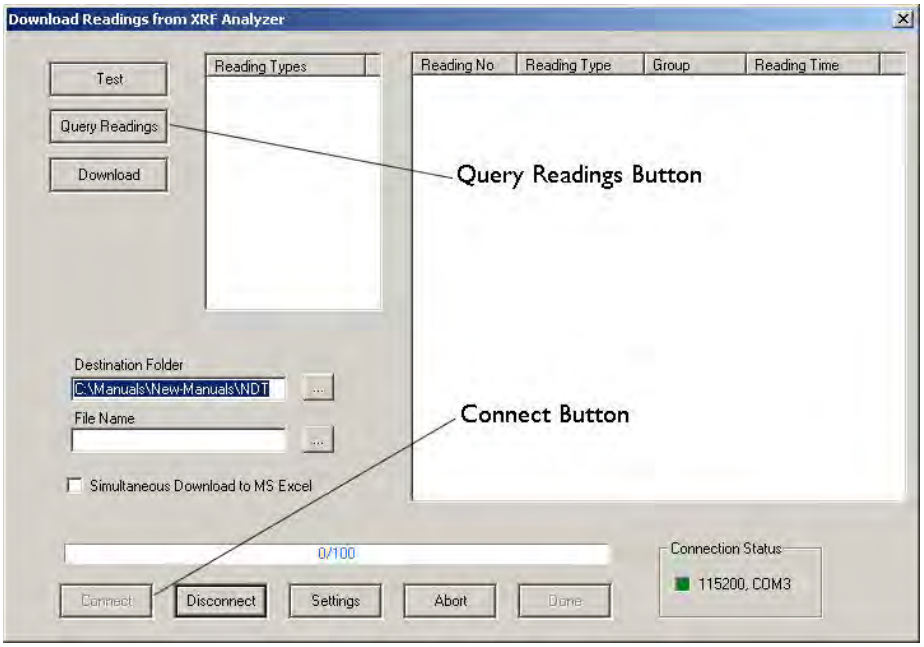


Figure 59. The Connect Button

8. Click the Query Readings button. This will return a list of all current readings on your analyzer. The list appears in the large white box in the Download dialog box.

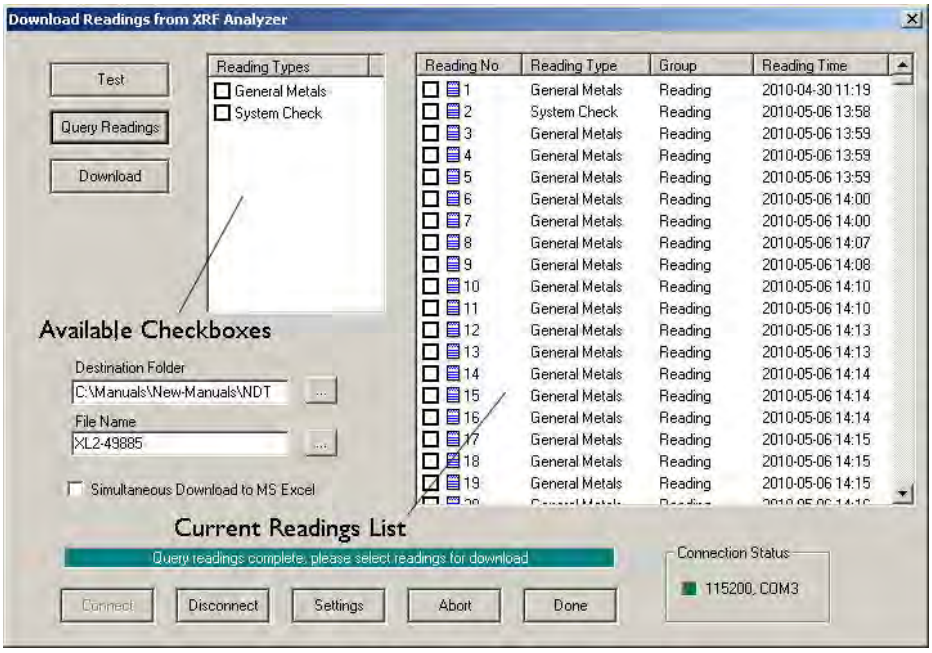


Figure 60. Current Reading List

9. Select the readings that you want to download. There are two ways to do this.

   a. Click the boxes next to each of the reading numbers to select or de-select individual readings. You can select a range of readings by pressing the shift key, then selecting the first and last reading in the range. All readings from the first reading selected to the last will then be selected.

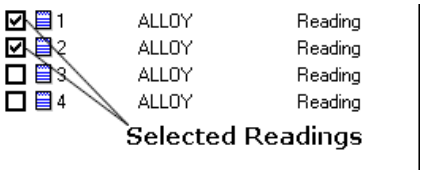


Figure 61. Selecting Readings

   b. Click the boxes on the left to select or de-select all the readings of a specific type. You can also use the Shift-Click method of selecting a range of readings as described above.

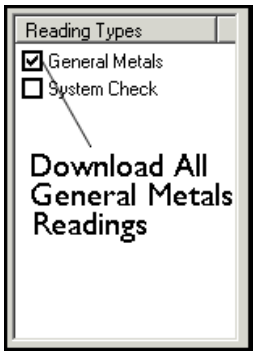


Figure 62. Using Check Boxes

10. The download generates a data file containing the selected readings. To save the file for later use:

c. Enter the path for the file in the Destination Folder field. You can use the ... button to browse.

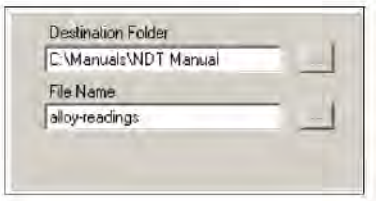


Figure 63. The Browse Button

d. Enter a name for the file in the File Name field.

CAUTION Some characters are not allowed in the file name. Characters such as the "#" sign will cause an error. Niton recommends using only alphanumeric characters "-", "_" and the space character when naming a file.

e. Click the Download button.

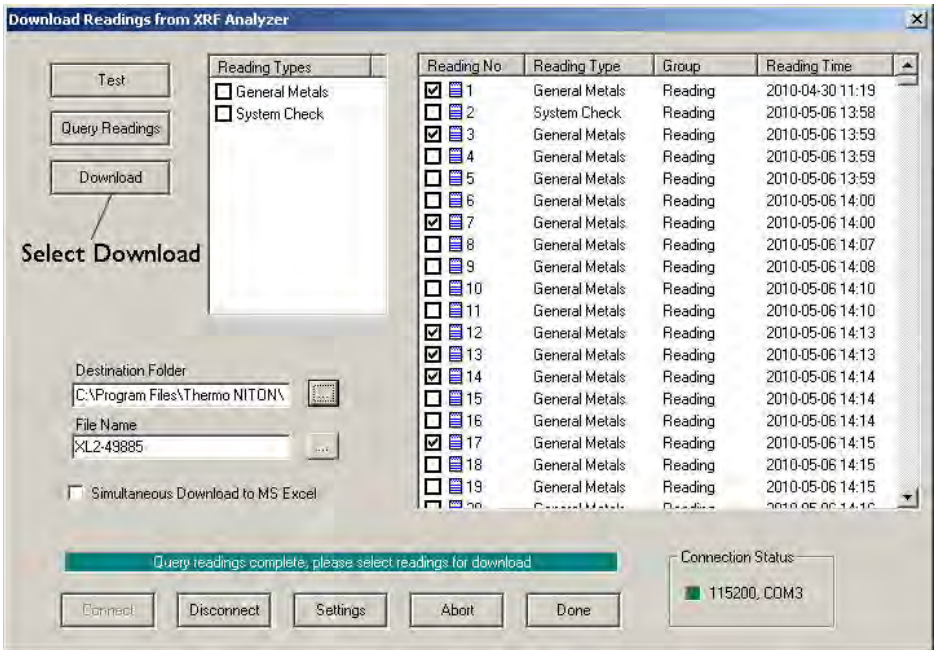


Figure 64. The Download Button

When the progress bar shows that all the readings are downloaded, click the Done button.

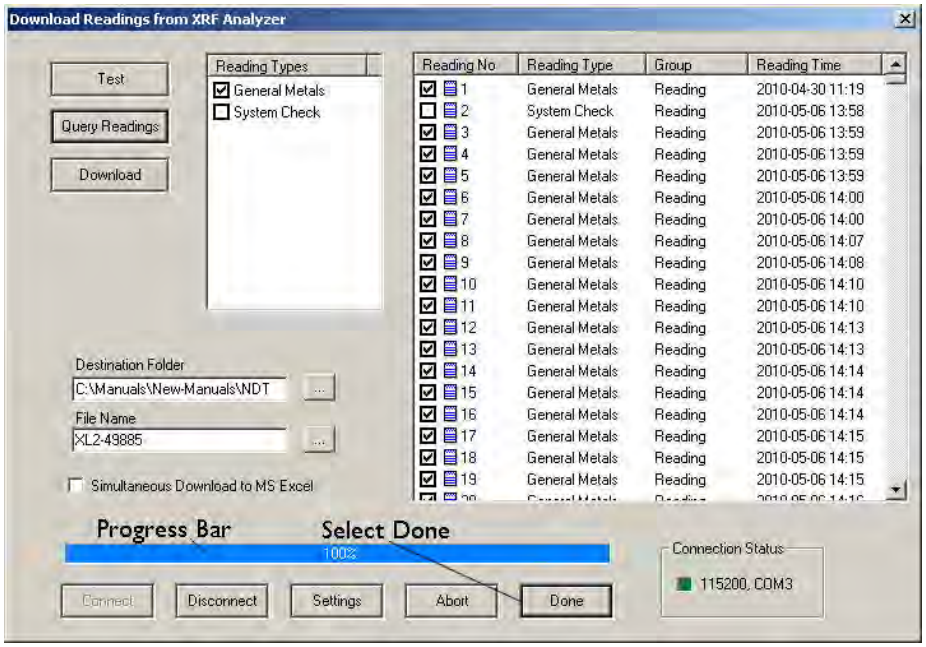


Figure 65. The Progress Bar

You should now see the readings you selected for download displayed, one reading per horizontal line. The data has been saved to the folder and filename you indicated prior to downloading. If an error message has appeared, see the following section.

You can also automatically save reports in .csv format for importing into Excel or other programs.Simultaneous Save as CSV File

Table 4: Error Messages while Downloading

Error Message	Action
Couldn't open \\.\COM7 Error Code: 2	Select another COM port.
The port \\.\COM2 is in use	Select another COM port.
Please Open the Port	Click the Connect button.
Hardware Not Responding or Hardware Not Ready	Turn on the XRF Analyzer. If you are using a serial cable, check that the cable is inserted snugly. If you are using a serial cable, select the other COM port. If you are using the wireless USB adapter, connect the serial port. See the "Installing and Using Bluetooth" manual for complete instructions on setting up the Bluetooth adapter to work with your analyzer. Check that the spare battery is fully charged.
The Serial Port connection failed: RFCOMM connection failed	Check that the battery is fully charged.
WARNING: 38400 baud rate not supported.	This indicates a potential problem. Test the serial port. If there is a problem connecting, switch baud rate on both the analyzer and the NDT software to 115200.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR1.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR2.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.

Table 4: Error Messages while Downloading

Incorrect Data in reading # XXX. Reading will be skipped. Error code: BOUNDARY_ERROR3.	This indicates a version mismatch between your instrument code and the NDT code running on your computer. Use a version of NDT that matches the ver?sion number of the software on your analyzer.
WARNING: 115200 baud rate not supported.	This indicates a potential problem. Test the serial port. If there is a problem connecting, switch baud rate on both the analyzer and the NDT software to 38400.
SH4 Successfully Communicating Result: SUCCESS	This indicates a normal connection.

Note When using the wireless USB adapter, if the serial port repeatedly disconnects, check that the battery is fully charged.

Live Download (Automatic Save)

If desired, your Niton XL2 analyzer has the capability to download and store each reading to the PC in real time to a file of your choice. To enable this feature, you must do the following:

• Your Niton analyzer must be turned on and connected to the PC. See Using Your Analyzer With Your PC.

• The NDTr program module must be running and connected to your analyzer. See Operating Your Analyzer Remotely.

• The Download icon in the NDTr toolbar must be selected.

Figure 66. Live Download Icon

The file created is in a format readable by the NDT program module, has an extension of .ndt, and looks identical to a file of manually downloaded readings - see Standard Download. It can also create a simultaneous .CSV file. Simultaneous Save as CSV File .

Please note the following:

1. When the instrument is unplugged, selecting the Download icon does nothing.

2. When you disconnect, then reconnect, your analyzer, Download appends future readings to same file.
3. Live Download does not overwrite any previous readings in the file. If you want to do this, you must first explicitly erase the file before initiating Live Download.
4. Live Download does not retroactively add any readings taken while your analyzer was disconnected.

Changing the Filename for Live Download

Once you have selected the Download icon, a dialog box appears as shown below:

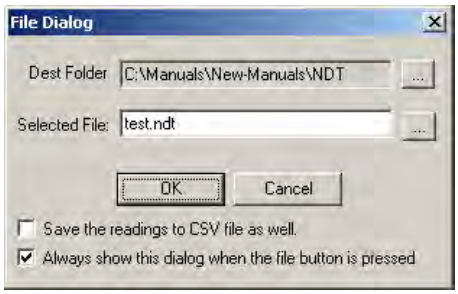


Figure 67. File Dialog Box

You can change the destination file or folder by clicking in the appropriate text box and typing in the new file name, or by clicking on the browse button (...) to the right of the text box and selecting a different pre-existing filename. To implement these changes, click the OK button.

Your instrument serial number is associated with the file. If a different instrument is connected and Live Download is started, a message will appear saying that the connected instrument and file instrument do not match, and Live Download will not start. Saving the session as a new file will alleviate this issue

Simultaneous Save as CSV File

By clicking on the checkbox box labeled "Save the readings to CSV file as well" you can enable simultaneously saving the data as a standard CSV (Comma Separated Value) file for use with other programs.

Controlling Your Analyzer From Your PC

The NDTr program allows you to completely control your Niton analyzer remotely, from your computer. It works over serial or USB connection over the supplied connector, or Bluetooth wireless communication. See Using Your Analyzer With Your PC for details on how to connect your Analyzer and PC.

The NDTr Toolbar

The NDTr Toolbar is a string of icons along the top of the NDTr window. It looks like this:

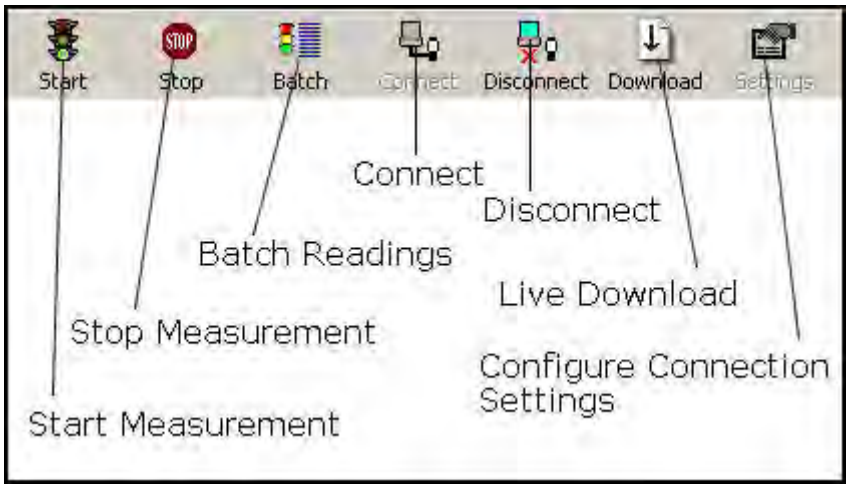


Figure 68. The NDTr Toolbar

Start Measurement

Clicking this icon will initiate a measurement in whatever mode the analyzer is in currently.

Stop Measurement

Clicking this icon will halt any ongoing measurement on the analyzer.

Configure Connection Settings

Clicking this icon will allow you to change your configuration settings.

Connect

Clicking this icon will attempt to establish a connection between your computer and your analyzer.

Disconnect

Clicking this icon will disconnect your computer from your analyzer.

Live Download

See Live Download from NDTr

Configure Connection Settings

Clicking on the Configure Connection Settings icon allows you to change the Com Port for connecting your computer to your analyzer. Once you click on the icon, a settings dialog box will appear.

Figure 69. Connection Settings Dialog Box

Selecting the Com Port

Selecting the down arrow in the "Connect Using" field will display the various Com Ports on the computer that the analyzer can connect through.

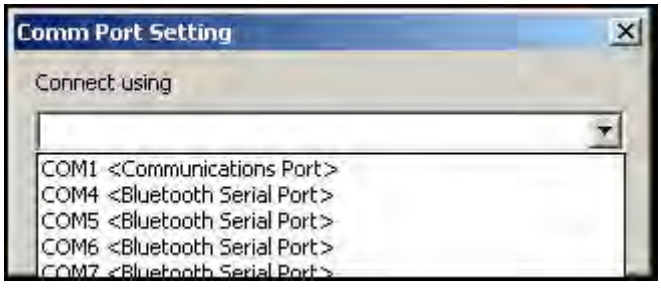


Figure 70. Selecting the Com Port

Select the proper com port from the list, then select the OK Button.

Operating Your Analyzer Remotely

NDTr version 7 and above will automatically select the proper virtual interface for you, whether you have a Thermo Scientific Niton XL2 or XL3. The virtual interface operates exactly as the analyzer would. Selecting the buttons, icons and menus with your mouse works exactly as if you were selecting them with your finger or stylus on the real analyzer.

Figure 71. Niton XL2 Virtual Interface

Live Download from NDTr

Once you have connected to your analyzer using NDTr, click on the Download icon on the NDTr toolbar. When you click the Download icon, a Download dialog box will appear.

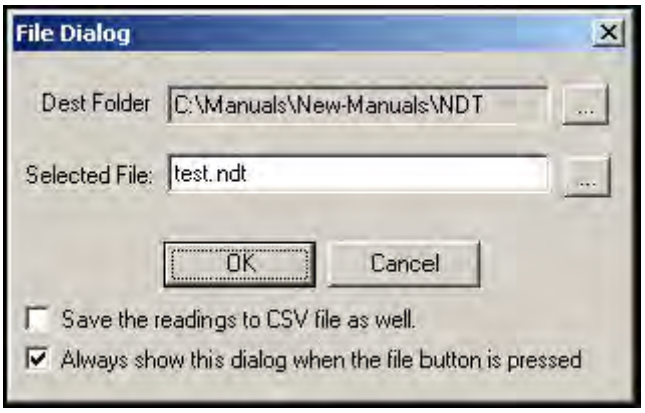


Figure 72. File Dialog Box

Dest Folder

This field shows the last used save folder, defaulting to the folder where NDT is installed.

... (Browse Folders) Button

Selecting this button enables you to select a different folder for saving the file. This will change the Dest Folder Field.

Selected File

This shows the filename the reading will be saved to unless you change it.

... (Browse Files) Button

Selecting this button enables you to select a different file name by browsing the file listing. The file extension ".ndt" will be appended to the name - i.e. File name "file" will be saved as "file.ndt" and the file will be in the NDT format.

Save the Reading to CSV File as Well Checkbox

Selecting this checkbox enables you to create a second autosave file with CSV format for importing into spreadsheets such as Excel. This file will have the same name as the NDT file above, but with the file extension ".csv" instead - i.e. "test.ndt" will be saved as "test.csv" as well. The checkbox is selected when there is a check in it, and deselected when it is empty.

Always Show this Dialog Box when the File button is Pressed Checkbox

Selecting this checkbox will enable you to change the filename whenever you want. Deselecting this checkbox will save the file under the same name in the same folder every time. The checkbox is selected when there is a check in it, and deselected when it is empty.

Learning More, Service, and Support

This section of the Resource guide is about getting the most out of your analyzer. We cover troubleshooting your analyzer by using the Specs screen. We also cover advanced topics like setting thresholds, using the Tools menu, correcting for light elements in the sample composition, setting up pass/fail analysis, changing safety and start/stop parameters, and many other special situations you may need. We have also included a number of documents for reference, so you can learn more about XRF analysis if you are so inclined.

Replacing the Measurement Window

WARNING Before you begin, remove the battery from your analyzer!

- Remove the two screws holding the Measurement Window Bracket to the nose of your analyzer.

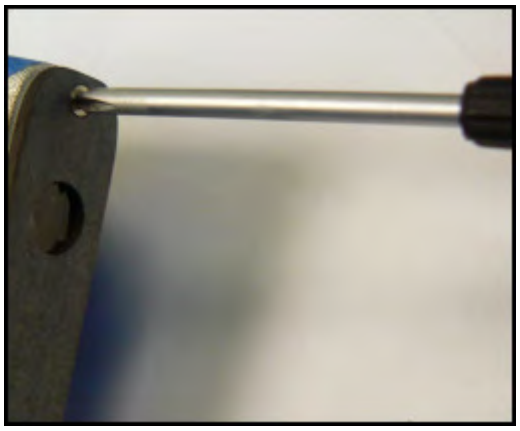


Figure 1. Removing the Window Bracket Screws

- Remove the Measurement Window Bracket from the analyzer, and turn it over, exposing the back with seal and Measurement Window.
- Remove the old Measurement Window from the bracket.
- Clean the Window area thoroughly, using a clean, guaranteed lint-free cloth and isopropyl alcohol.

Figure 2. Removing the Old Window

- Measurement Windows come in two types - Prolene (P.N 187-1454) for 900 Series analyzers, and Polypropylene (P/N 187-1555) for all other analyzers.

Figure 3. Polyethylene Window P/N 187-1555

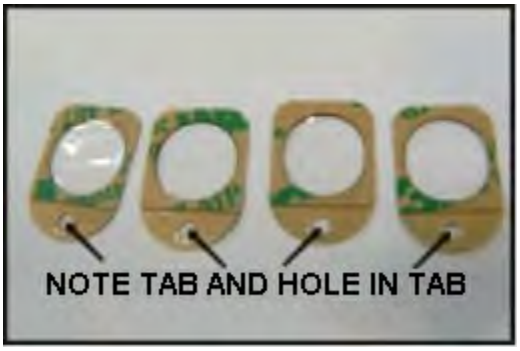


Figure 4. Prolene Window PN 187-1454

- When the bracket is clean, remove the backing from the Measurement Window. Place the window on the Bracket gently. Make sure the opaque portions of the window do not intrude over the large measurement hole in the Bracket.

Figure 5. Removing the Backing from Prolene Window (Left) and Applying Window to Bracket (Right)

CAUTION Do not use fingers to press window into place! Use a smooth, hard surface such as back of tweezers.

Figure 6. Measurement Window Replaced

- Replace Window Bracket on the front of your analyzer, then insert screws.

Tips and Troubleshooting

The Specs Screen

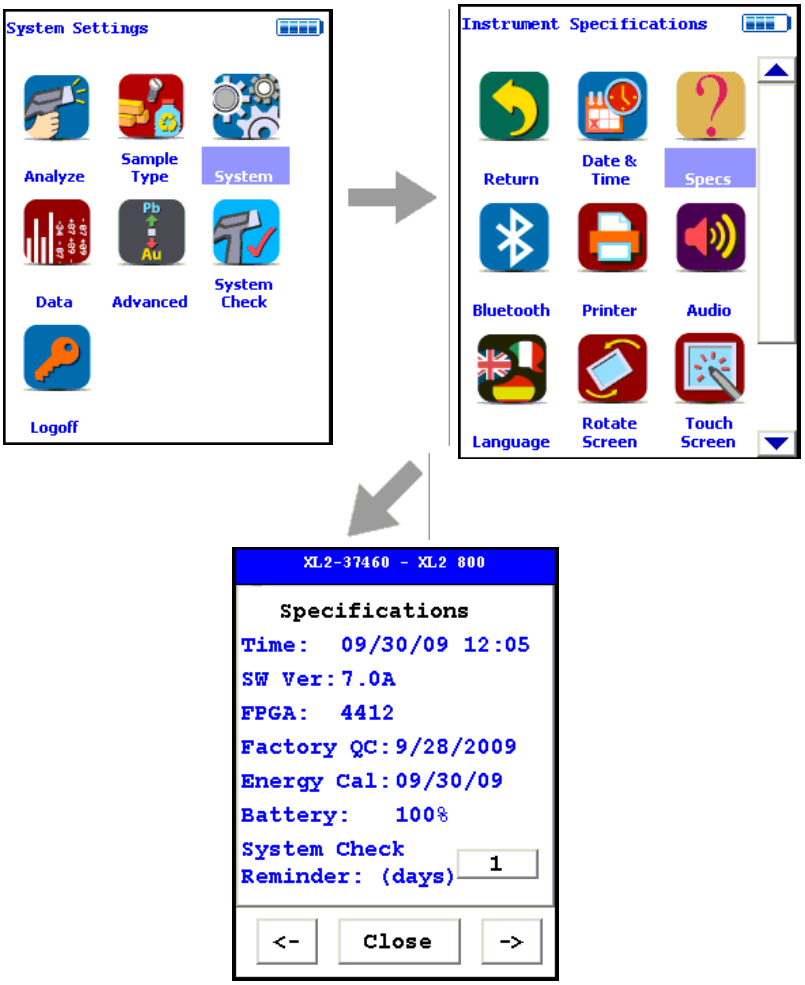


Figure 7. The Specs Screen Menu Path

Select the Specs icon from the System Menu to display the analyzer's specifications. These specifications include your analyzer's serial number, software and firmware versions, and other data. Press the Close Screen Button to return to the previous menu. Press the ">-" Screen Button to go to the Diagnostic Menu, and press the "<-" Screen Button to return to the Specifications Screen.

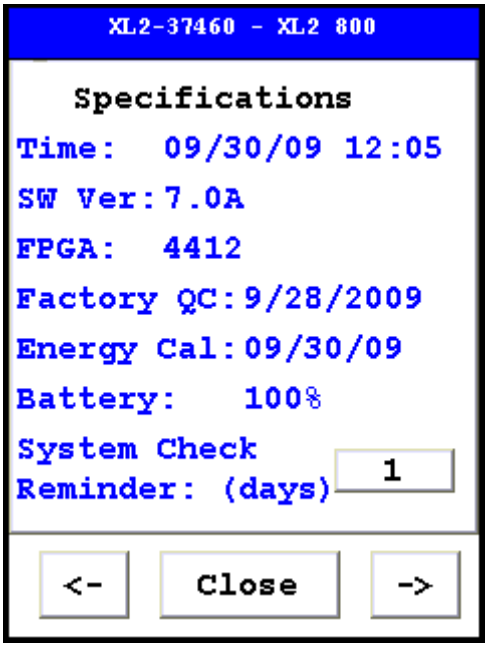


Figure 8. The Specifications Screen

On the Specs Screen, standard information on the state of your analyzer is shown for your reference. This information should be reported to Service if there is a problem.

Specs Information

The following is the information supplied on the Specs Screen:

Instrument Specific Serial Number

This is located in the left part of the blue band at the top of the screen.

Model Number

This is located in the right part of the blue band at the top of the screen.

Date And Time

This is the current Date and Time. This is particularly important for date stamping.

SW Version

This is the currently loaded software version, which should be reported to Service if there is any problem.

FPGA

This is the currently loaded FPGA software version, which should be reported to Service if there is any problem. FPGA versions are always a four digit number. Any other number of digits may be a sign of a problem in the FPGA code.

Factory QC

This is the date that the machine was QCed at the factory.

Energy Cal

This line notes the last time a System Check was performed.

Battery

This line gives the proportional charge remaining to the battery.

System Check Reminder

Select the OFF Screen Button after "System Check Reminder" to set a reminder to calibrate your analyzer. Selecting the button will open the Cal. Reminder Editor. Select the number of days you want between reminders with the numeric keys. Of the other screen buttons, C = Clear All, E = Enter, and OFF shuts off the Reminder Function. Selecting the E button will enter the current value as the reminder interval and return to the Specs Screen.

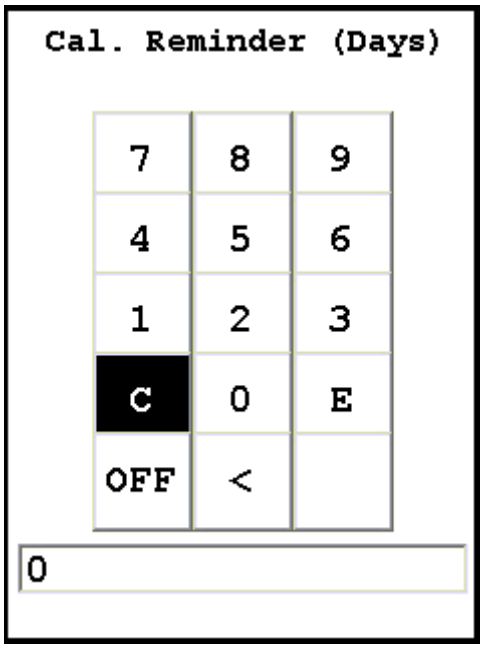


Figure 9. Cal Reminder Editor Screen

Diagnostics

Select the "->" Screen Button to load the Diagnostics Screen. The Diagnostics Screen shows Detector Temperature, Bias, Cooler Voltage, SubBias, Energy Scale, and Temperature in C and F scales.

The Diagnostics Screen can be of great utility in assuring proper operation of your analyzer.

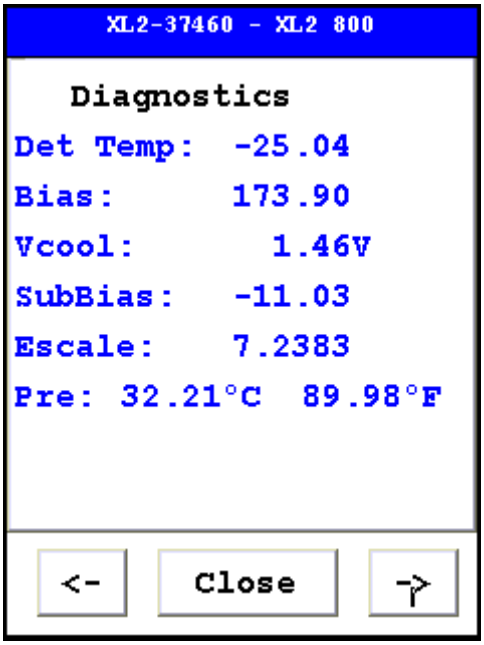


Figure 10. The Diagnostics Screen

The proper ranges of operational values on the Diagnostics Screen follow.

Det Temp:

Detector Temperature should be within this range:

- 25 + or - 5 degrees

Bias:

Bias should be within this range:

175 + or - 10

VCool:

VCool will vary with the ambient temperature.

SubBias:

SubBias should be within this range:

-11 + or - 3

Escale:

Escale should be within this range:

6.6 through 9.0

Preamp:

Preamp value should only be noted, and reported to Service if there is a problem.

Error Messages

The analyzer will auto-detect many perceived hardware problems and may display the following error messages:

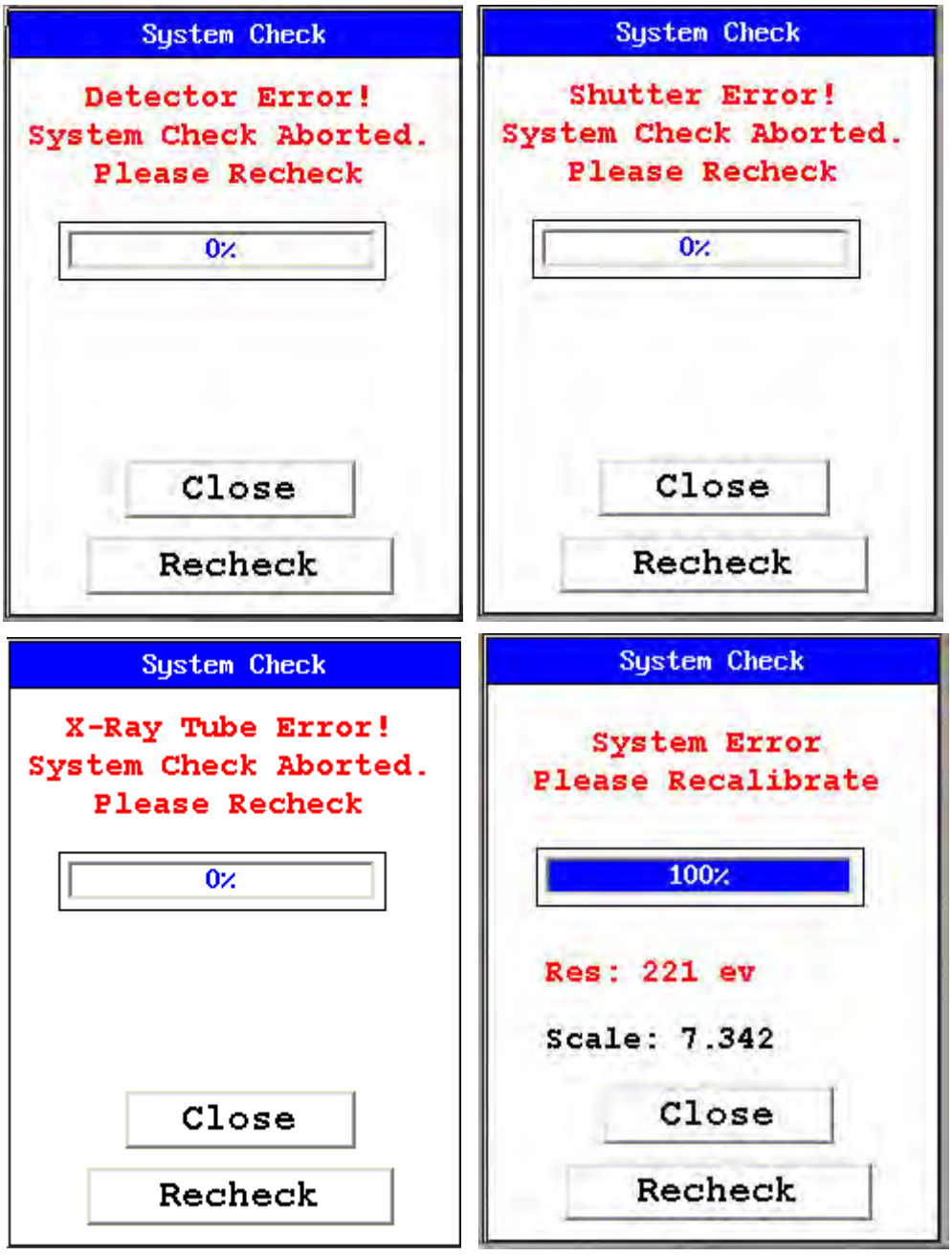


Figure 11. The Specs Screen Menu Path

Please re-run the System Check and if the problem persists, contact customer service.

Registration and Licensing FAQ

As a user of a Niton XL2 analyzer, you may be required to register or obtain a license with your local radiation control authority. In the US, if you intend to do work with your analyzer in states other than your own, you may be required to register there as well. Below is a list of commonly asked questions that come up when filling out registration forms.

FAQ

Q: What is the max mA, max kVp, and max power?

A: Maximum mA is 0.1 mA

Maximum kVp is 45 kVp

Maximum power: 2 watts

Q: What is the accelerator voltage or MeV?

A: This should be filled out as not applicable N/A as it does not apply to Niton XL2 analyzers.

Q: What is the radioisotope?

A: There are no radioactive isotopes in Niton XL2 analyzers.

Q: What category is the Niton XL2?

A: States differ greatly in their categories; the following is a list of common categories:

- X-Ray Fluorescence
- Analytical or Analytical XRF
- Open Beam or Open Beam Analytical
- Portable Gauge or Portable XRF
- Industrial Analytical or Non-Destructive Testing

When selecting the category make sure that you don't select medical or radiographic.

Q: How many tubes are in the Niton XL2?

A: One.

Q: What is the analyzer serial number?

A: The serial number is a 5 digit number located on the yellow sticker on the underside of your analyzer.

Q: What is the tube serial number?

A: The serial number of the tube can be found on the Calibration Certificate.

Q: What is the type of X-Ray Processing?

A: None. Niton XL2 analyzers do not use film.

Q: How often do I need to perform leak tests on the Niton XL2?

A: Never. Leak tests are only required for analyzers with radioactive isotopes. Niton XL2 analyzers do not have radioactive isotopes.

Storing and Transporting Your Niton XL2 Analyzer

All Niton Analyzers are transported in waterproof, drop-resistant, fully padded carrying cases with padlocks. In most countries, Niton XRF analyzers may be transported by car or plane or shipped as an *ordinary* package. For most courier services, no special labels are required on the outside of the Niton analyzer case or on additional packaging.

Figure 12. The Niton Carrying Case

All padlocks are shipped with a default combination of "0-0-0". If you change this combination, please inform Thermo of the new combination if you return the unit for service.

To change the combination:

1. Dial the default combination to open the lock, and pull out the shackle.
2. Rotate the shackle 180 degrees and push it down as far as it can go.
3. While holding the shackle down, rotate it 90 degrees back in either direction and release shackle.
4. Change the dial settings to the desired combination, record the combination, and without disturbing the dials, rotate the shackle back 90 degrees to the position it had in step 2.
5. Pull shackle out and rotate it 180 degrees and secure it. Your lock now has its own secret combination.

CAUTION Always transport the unit in its padded carrying case, and store the Niton Analyzer in its case whenever it is not being used.

CAUTION In most cases, no notification is required if transporting within state boundaries. This may not be the case when entering federal properties.

CAUTION Within the United States, always keep a copy of the US DOT compliance statement in your Niton analyzer case at all times. A copy is included with your analyzer.

CAUTION Always follow all pertinent local and national regulations and guidelines, wherever your analyzer is transported or used.

CAUTION Always obtain a Return Authorization (RA) number from Thermo Fisher Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your analyzer to the Service Department or to your local Authorized Niton Analyzers Service Center.

CAUTION If you return your Niton analyzer without the carrying case, you will void your warranty in its entirety. You will be billed for a replacement case plus any repairs resulting from improper shipping.

CAUTION CAUTION Always remove the battery pack when transporting or storing your analyzer.

Advanced Settings

Adjusting the Element Range

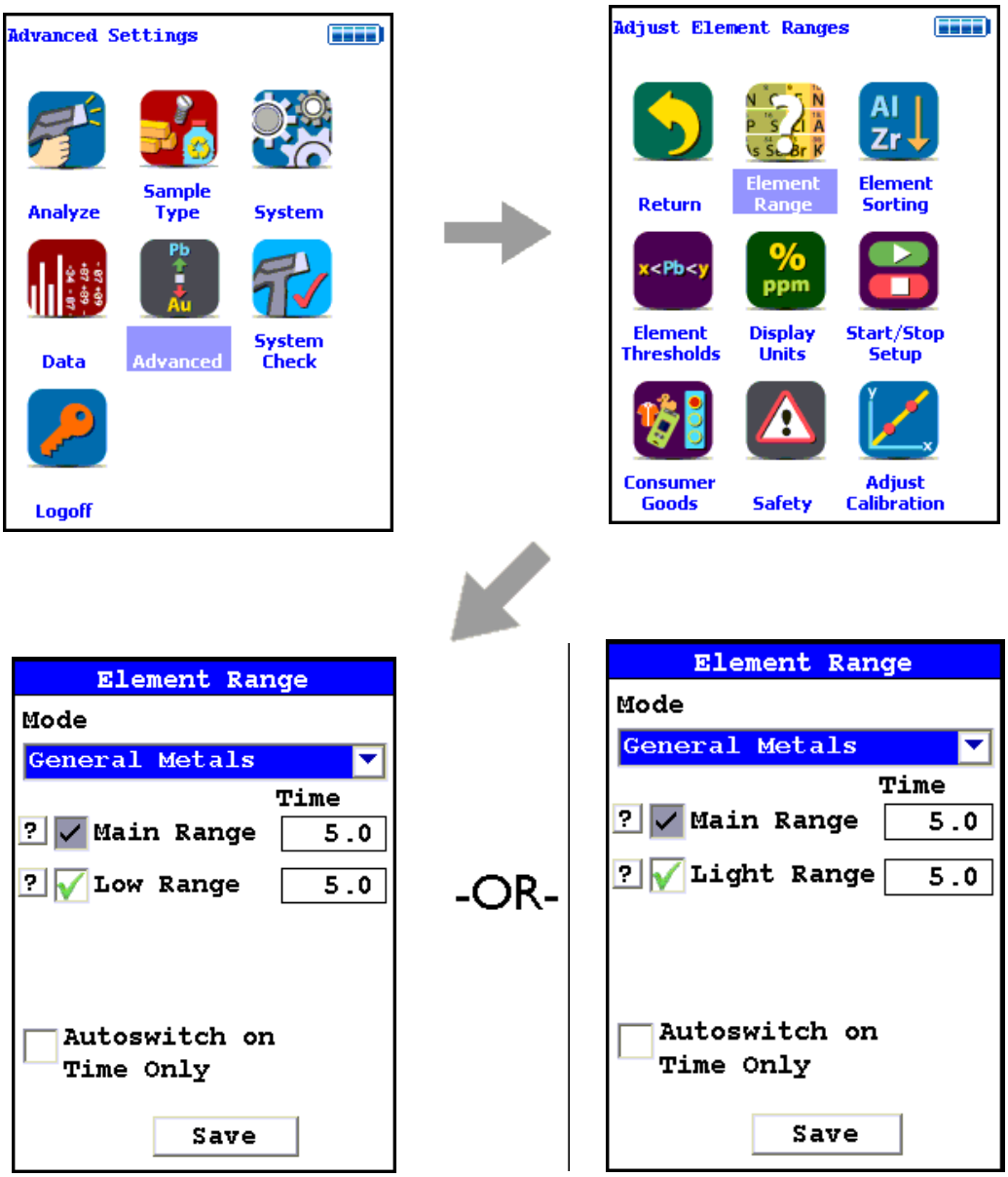


Figure 1. The Range Menu Path (Main)

Multi-Range tests are used to either preferentially excite specific elements for increased sensitivity, or to cover a wider element range than one Range alone can provide. Most modes, when enabled, will use two Ranges in sequence to produce a combined analysis result. In typical

alloy analysis applications, Main Range is used for the analysis of most elements, Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr, and Light Range is available only with 900 series GOLDD technology analyzers, and is typically used in light element analysis. Multi-Range switching can be set to activate off time alone, or, when time switching is disabled, off settings in the General Metals grade library. In most modes, Low and Light Range add the capability to analyze light elements which cannot be efficiently excited by Mid Range.

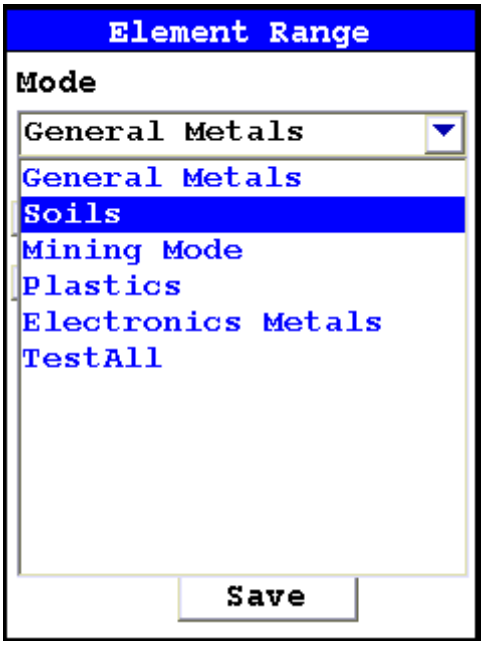


Figure 2. Selecting the Mode

Select the mode you wish to configure. You can set different configurations for different modes.

The Element Range Screen enables you to directly enable or disable any Range, or control the time that a Range alters the irradiation of the sample before auto-switching to another Range.

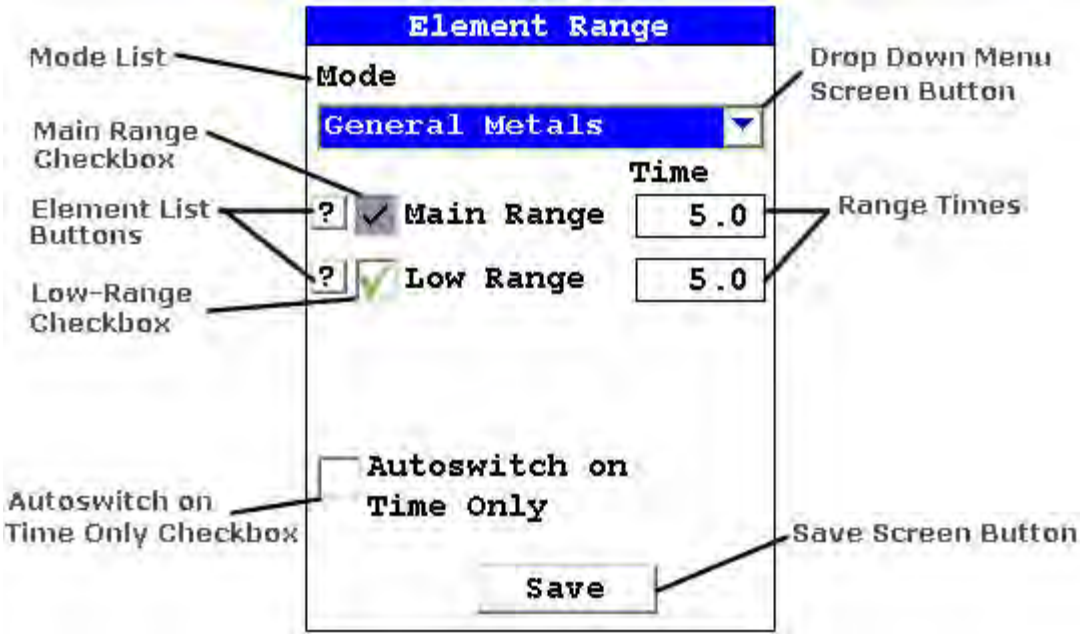


Figure 3. The Element Checkboxes

Select the checkbox next to the Range you want to use to determine exactly which of the Ranges contained in your Analyzer is used for sample testing. Selecting an empty checkbox will enable that range and place a check into the box as an indicator. Selecting a checked box will disable the Range and clear the box.

In typical alloy analysis applications, Main Range is used for the analysis of most elements. You cannot deselect the Main Range in alloy analysis

Low Range is utilized for the subsequent high sensitivity analysis of V, Ti, and Cr.

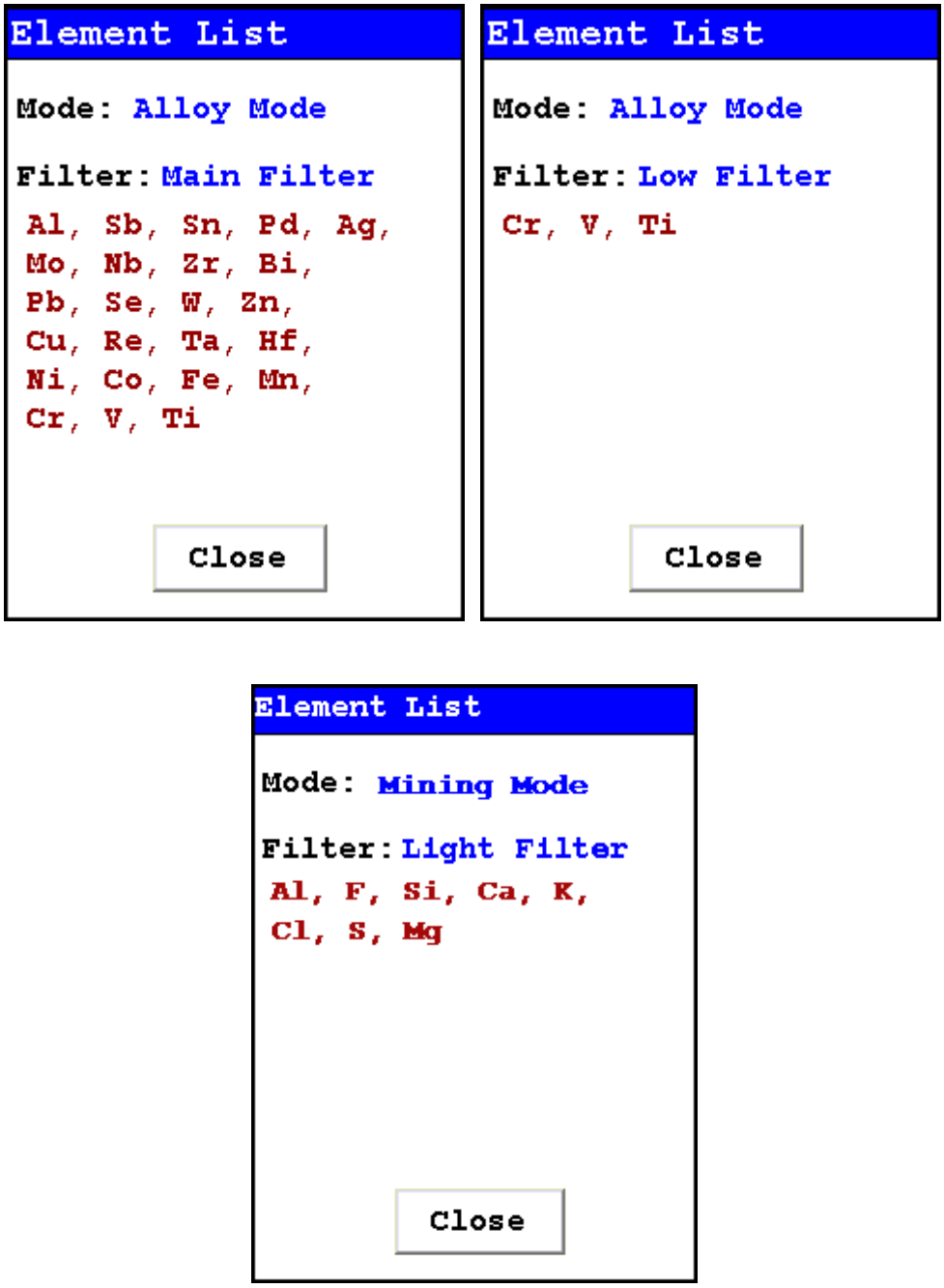


Figure 4. The Range Element Lists

Select the Element List Button to display the Element List for that Range. This list shows the elements that the Range is best designed to detect.

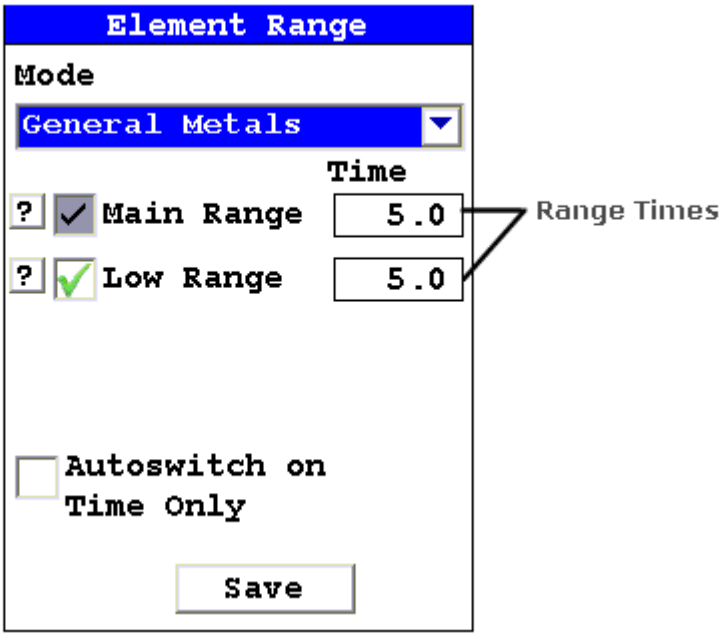


Figure 5. The Range Time Fields

Select the Range Time field for the intended range to change the switch time for that range. The Range Time Editor will appear. This enables you to set the number of seconds each enabled range is allotted before auto-switching will occur when needed during sample testing. Your analyzer will auto-switch from one range to another when the testing time for that range is greater than or equal to the time you have chosen, and the identified alloy is flagged as needing the switch in the Niton Alloy Library.

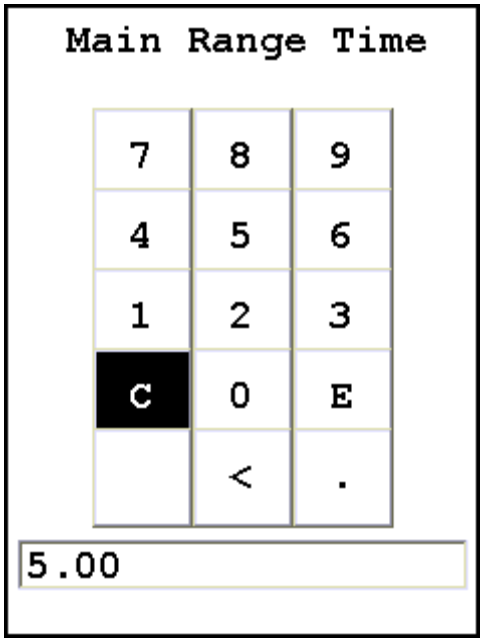


Figure 6. The Range Time Editor

Select the C button to clear the current time, then from the virtual numeric key pad, select each digit you want to input, then select the E button to enter.

Tools Menu Options

The following options can be performed from the Tools Menu. Options which are only on the main Tools Menu are listed as (Main). Those which can be found only on the alternate Tools Menu are listed as (Alt).

Avg Forward

Enables you to average different readings together from this analysis forward. Select the Avg Forward button to initiate future sample averaging. Avg Forward will set up an automatic personal averaging protocol to be followed until your analyzer is shut down, or this feature is disabled. To begin, select the number of readings you want to average from the virtual numeric keypad. Your analyzer will calculate an average reading after that number of tests, and continue this pattern until stopped. For example, if you select 3 on the virtual keypad, the analyzer will automatically calculate, average, and store a reading for every three tests you take, storing the individual readings along the way.

The range number is selected using a virtual numeric keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the E button to enter the number. The C button will clear all, and the "<" button will clear the last digit entered. The average will automatically be displayed.

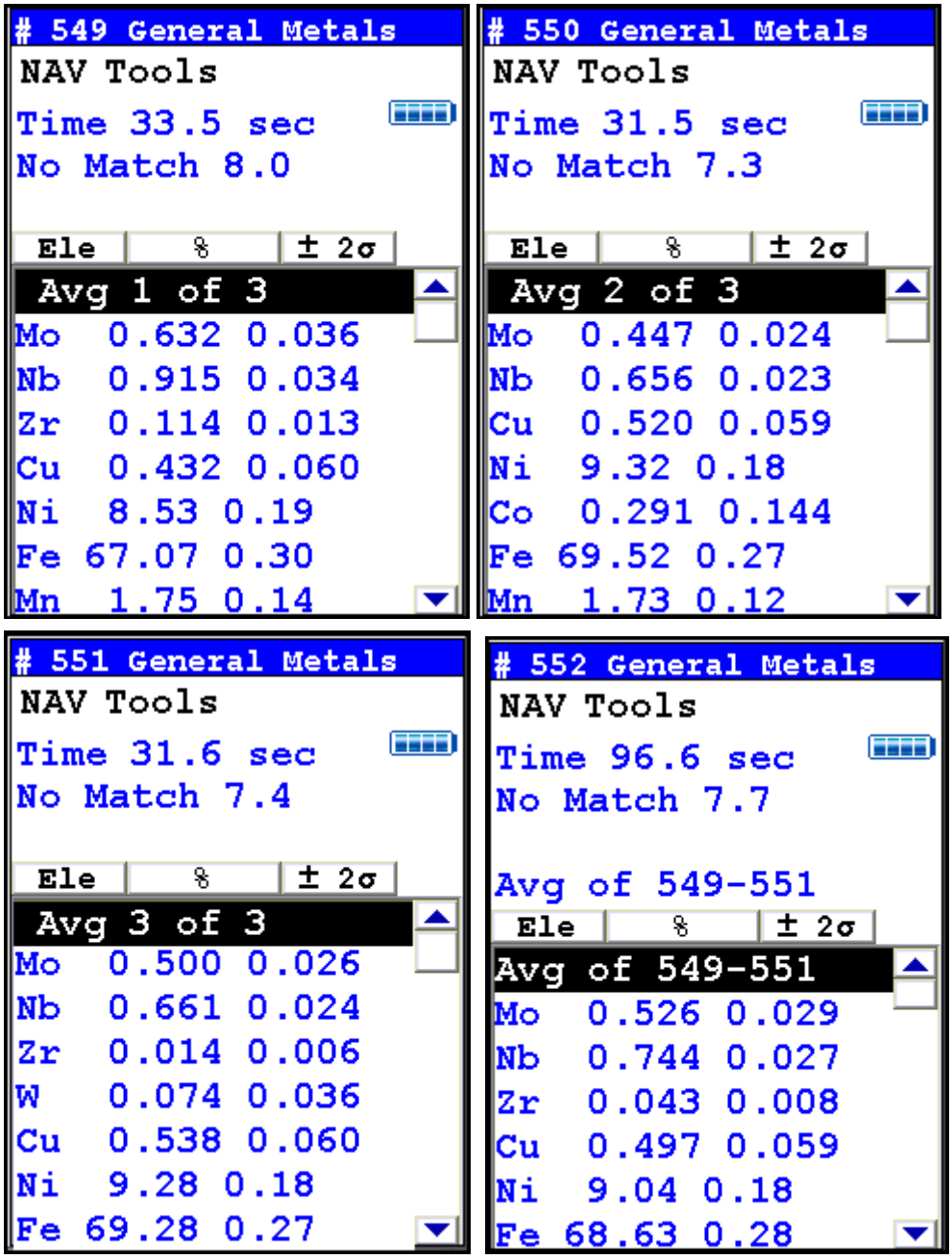


Figure 7. Example Averaging Screens

Avg Back (Alt)

Note The alternate Tools Menu is only available when viewing readings, and the menu is only accessible through the touch screen interface or NDTr.

Enables you to average different readings together from this analysis backward. Select the Avg Back option to initiate backwards sample averaging. Avg Back will take the number of readings you select and average their analytical results. The range is counted from the last reading backward by the number of readings selected. If your last reading was #15, selecting 3 would average readings #13, 14, and 15. The average is calculated, displayed, and stored into memory as the next sequential reading number, in this case, #16.

The range number is selected using a virtual numeric keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the E button to enter the number. The C button will clear all, and the "<" button will clear the last digit entered. The average will automatically be displayed.

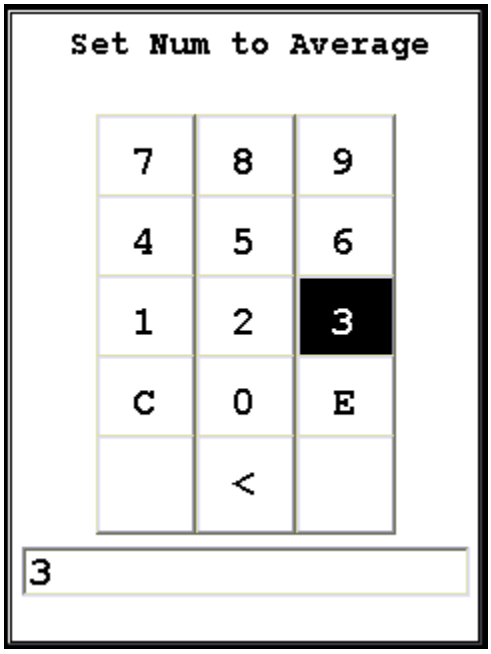


Figure 8. The Virtual Numeric Keypad

Note You cannot average readings taken in different modes. Doing this will generate an error.

Spectrum:On/Spectrum:Off

The Tools Menu contains a toggle option to display live spectra as sample analysis occurs.

Activating and Deactivating the Live Spectrum

From the Tools Menu, select the Spectra:On button to turn the Spectrum feed on. Once the spectrum is displayed, selecting Spectra:Off from the Tools Menu will stop the live spectrum display.

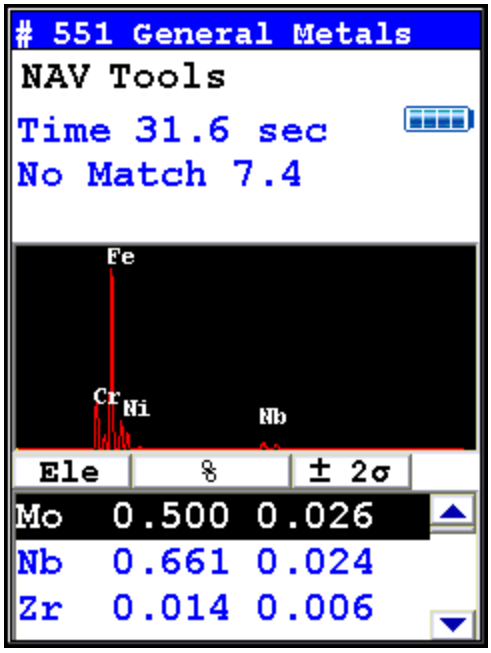


Figure 9. Example Analysis Screen Showing Live Spectrum

Print (Alt)

Select the Print option from the Tools Menu to print the current analysis screen to any attached portable printer. If you do not have a portable printer attached to your analyzer, nothing will happen.

Set Pass/Fail

You can set up your analyzer to sort on a pass/fail basis. Pass/Fail uses the chemistry of a user-generated list of alloys in the library as a basis for comparison. If the sample analysis is entirely within the specifications for one of these alloys, a PASS result is given, otherwise a FAIL result is returned. To turn on Pass/Fail, select the Tools Menu and select Set Pass/Fail from the menu. The Pass/Fail Setup Screen will come up.

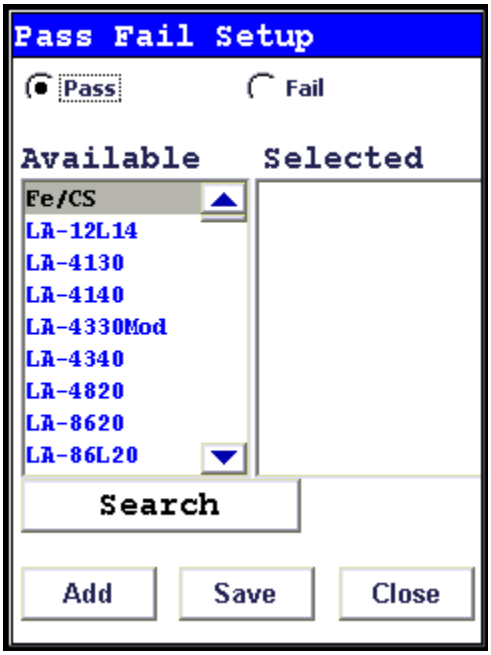


Figure 10. Set Pass/Fail Screen

Add/Remove (Toggle)

Select alloys from the Available list and then the Add Button to move the alloy to the Selected List. Select alloys from the Selected list and then the Remove Button to remove the alloys from the Selected List.

Save

Select the Save Button to save these criteria.

Close

Select the Close Button to exit without saving.

Pass

Select the Pass Single button to initiate Pass Mode. Use Pass Mode when you have a desirable match. If the alloy being analyzed matches one of the alloys in the selected list, the alloy will Pass the analysis.

Fail

Select the Fail button to initiate Fail Mode. Use Fail Single Mode when you have an undesirable match. If the alloy being analyzed matches one of the alloys in the selected list, the alloy will Fail the analysis.

Setting the Reference Alloys for Pass or Fail

Before you use Pass or Fail mode, you need to set the Reference Alloys. Select the alooy or alloys from the slide down menu on the Pass Fail Setup Screen, then select the Add button.

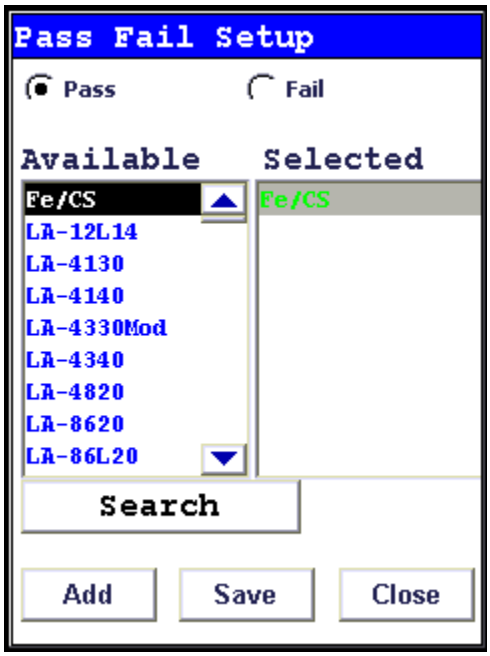


Figure 11. . The Pass Fail Setup Screen

Searching for Reference Alloys

Select the Search button to search the library for the alloy you want as your Reference Alloy.

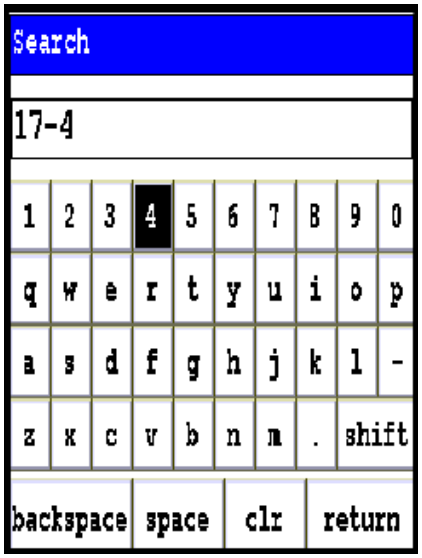


Figure 12. Using the Search Function

Type the name of your reference alloy into the Virtual Keyboard, and the left column will display any matches. Select the match you want and the Add button to make it your reference alloy.

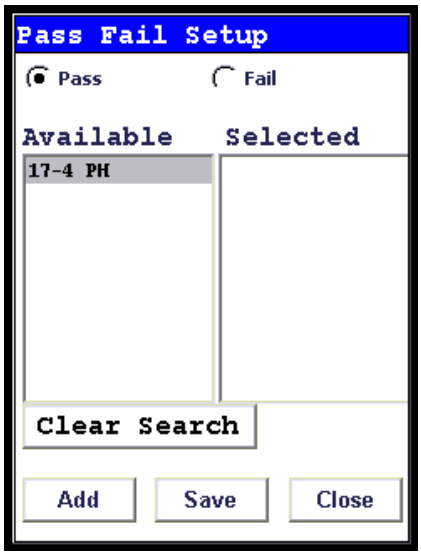


Figure 13. Search Results

UHow Pass/Fail Mode Works

Pass/Fail Mode compares the chemistry to that of the alloy(s) selected, using the cutoff you selected. When the sample analysis reaches a match with the chemistry of any one of the alloys on the Selected list, a PASS or FAIL notice is generated as appropriate.

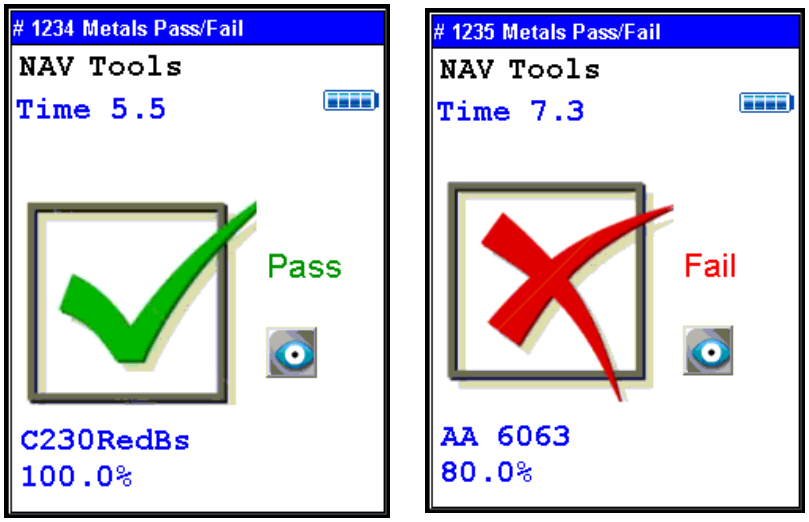


Figure 14. Metals Pass and Fail Screens

Switch Library (Main)

Selecting the Switch Library button from the Tools Menu will swap the currently loaded library with the other library on the analyzer. Selecting Switch Library again will switch them back.

Enable/Disable Al

Normally, the collective amount of unquantifiable light elements in alloy analysis - the "balance" - is assumed to be aluminum and labeled as such in the analysis. Selecting the Disable Al button from the Tools Menu will delete this "aluminum" from the analysis results, showing only the quantified elements. Selecting the Enable Al button, the default state, will label this "balance" as "aluminum".

Thickness Correction

Plastics, and polymers in general, unlike metals or soil, are very weak absorbers of X rays. This is because polymers are composed mainly of very light elements such as carbon and hydrogen. While just half a millimeter of steel will completely stop 23.1 keV energy X rays of cadmium, for example, it takes at least 10mm of plasticized PVC and as much as 100mm of polyethylene (PE) to do so. Fortunately, polymers that may contain cadmium (Cd), lead (Pb) and other restricted elements would also contain considerable quantity of elements such as antimony (Sb), bromine (Br), titanium (Ti), etc. Their presence results in much stronger absorption of X rays which means that, instead of 100mm, it takes only about 15mm of compounded PE to achieve saturation thickness for these X rays. If the thickness of analyzed polymer sample is less than 5mm for PVC or less than about 9mm for a “typical” PE, the measured intensity of X rays will be a function of both analyte concentration and sample thickness. This is why measurements performed on thin samples (less than saturation thickness) need to be corrected for thickness.

How to apply Thickness Correction.

In order for the instrument to apply thickness correction to the measured concentration results, the user must be using the Thickness Correction screen and enter the thickness of the analyzed plastic object expressed in [mm] before the measurement is initiated. The thickness may be entered with precision to the second decimal place, although in practice only one decimal place is sufficient for effective correction.

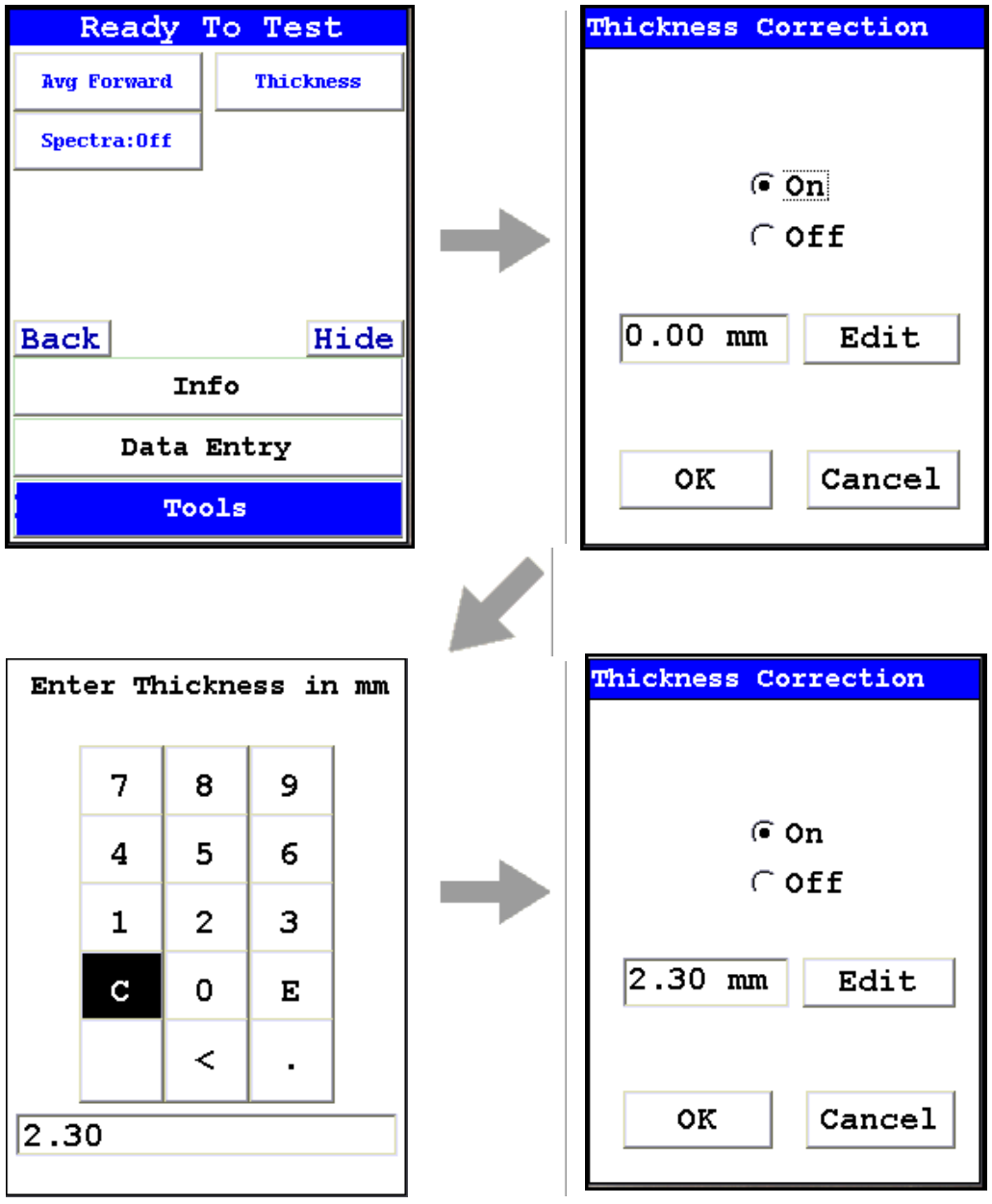


Figure 15. How to Enable and Adjust Thickness Correction for Plastics Analysis

When to use Thickness Correction

Thickness Correction should only be used during the analysis of plastic (polymer) objects. It has been experimentally verified that the correction algorithm will yield satisfactory results, for a 60 second minimum testing time, for samples as thin as 0.3mm. Nevertheless, the recommended range of use of the correction is from 1mm upwards. It is imperative that this correction is not used for thin films such as single foils and plastic membranes; analysis of thin films is performed using the Thin Sample Mode. (Contact Contact Thermo Scientific or your local Niton Analyzers representative for information on this testing mode.)

Whenever possible, one should analyze as thick a sample as available. For example, if the analyzed object is a piece of heatshrink tubing with wall thickness of 0.3mm, the best way to analyze it is to obtain several pieces of the tubing (four for example) and stack them like a flat sandwich, with the thickness correction set to 1.2mm. Doing so makes for faster and more precise analyses. While it would be possible to analyze just a single layer of the tubing with correction at 0.3 mm, by stacking several layers we reduce the relative error of measurement (by a factor approximately equal to the square root of the number of layers). Conversely, when analyzing thinner samples, we need to extend the measurement time fourfold (by the number of layers) in order to maintain the same relative error of measurement. We can see how quickly measurement time would escalate to impractical levels for thinner samples.

Examples: The most frequent instances in which thickness correction would be called for are analyses of plastic sheeting or plastic insulation on wires and/or cables and heat shrink tubing. Flat plastic sheeting or plastic enclosures pose no problems. We can either analyze an object “as is”, or stack several layers of it before analysis. Plastic insulation such as that on wiring or cables requires a little more sophisticated approach. First, the wire must be removed so that only insulation is analyzed. Then, the insulation should be flattened for analysis, and a thickness correction should be applied that is equal to double the wall thickness. Alternatively, if the insulation is stiff, it should be cut lengthwise into strands which are placed on the indtrument for analysis. The applied thickness correction should be equal to the wall thickness of the sleeve. Both operations are shown in Figure 37 and Figure 38.

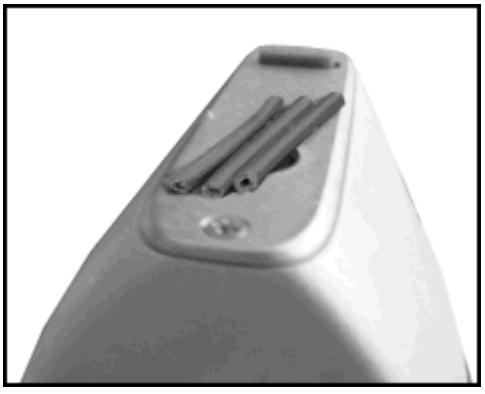


Figure 16. Wire Insulation Cut Into Strands

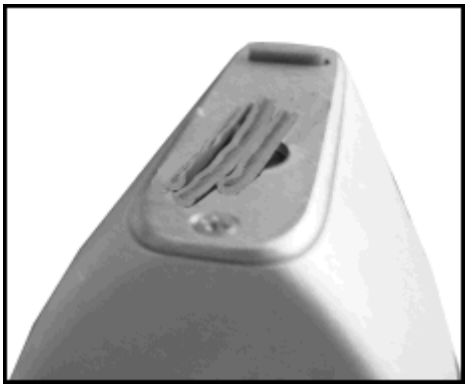


Figure 17. PVC Wire Insulation With Conductor Removed

A piece of large diameter heat shrink tubing presents an interesting case. It is tempting to analyze this object as is - see Figure 39. However, one needs to know that while lead or bromine or chromium X-rays from the upper wall of tubing will not contribute to the signal measured, X rays of such elements as cadmium, antimony, tin or barium in the upper wall will significantly contribute to overall signal. It is therefore imperative to either flatten the tubing for analysis or cut it in pieces and then analyze as shown in Figure 40.

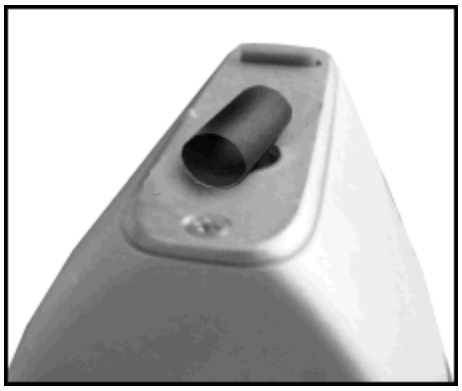


Figure 18. . Incorrect Way to Measure Heat Shrink Tubing

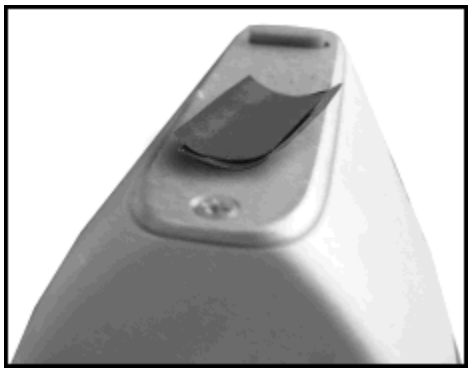


Figure 19. . Correct Way to Measure Heat Shrink Tubing

WARNING Thickness correction is only for use with plastic/polymer samples.

Enable/Disable Paint

Selecting the Enable Paint option from the Tools Menu will enable the Painted Products mode and toggle the option to Disable Paint. Selecting the Disable Paint option will disable Painted Products mode and toggle the option to Enable Paint.

Action Level

Selecting the Action Level option from the Tools Menu will anable you to change the action level used for qualitative testing.

Print Data

Selecting the Print Data option from the Tools Menu will print the currently displayed data to the optional attached printer. See Setting Up the Printer for details.

Coatings Method

Metals are sometimes coated with various materials. If you wish to analyze the coating, select the Coatings Method.

Passwords and User Privileges

1. Install the latest version of Niton software (NDT) on your PC, if possible. You may obtain the latest version of NDT by contacting service at 800-875-1578.

2. You can check the version number by opening NDT, selecting the Help menu, then selecting “About Niton Data Transfer”

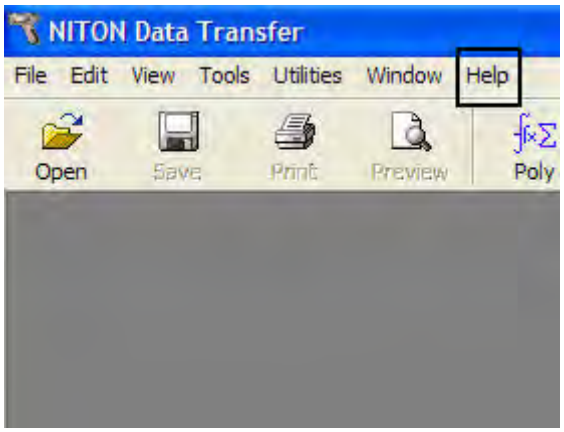


Figure 20. . Selecting Help

3. Select the File menu

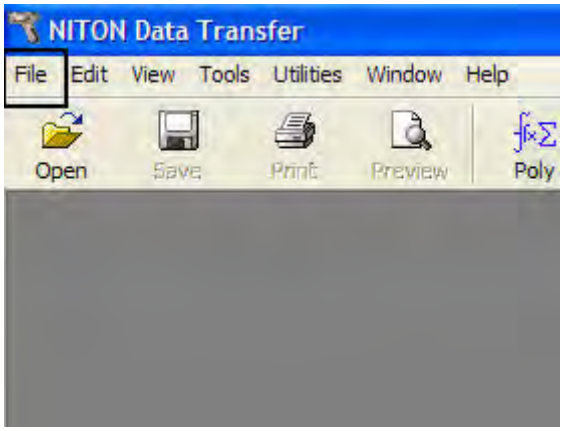


Figure 21. Selecting File

4. Select “New” then “New Password File”. Your screen should look like this:

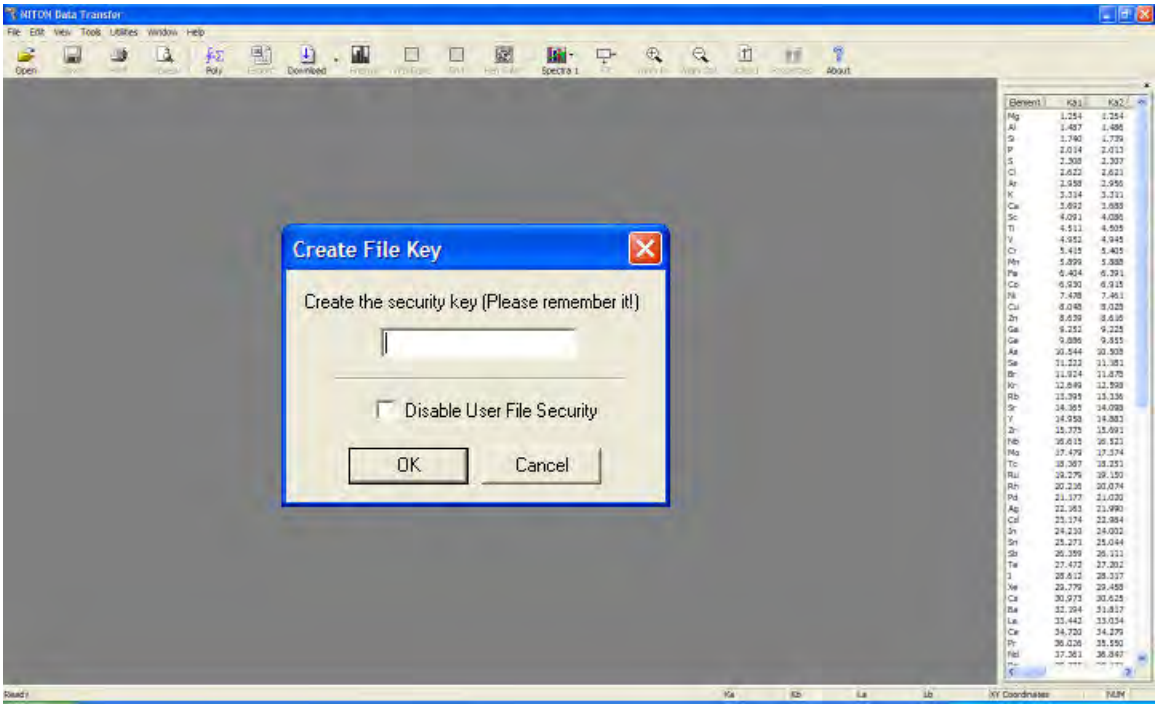


Figure 22. Creating the Security Key

5. Create a unique security key, then select the OK Button

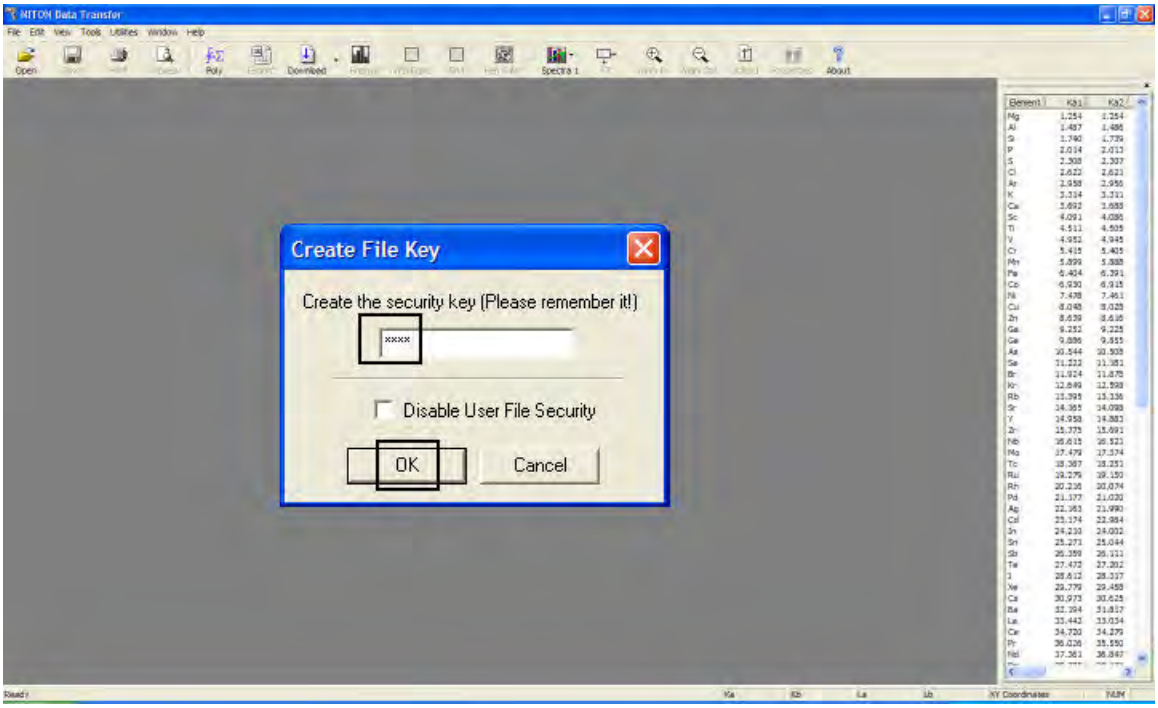


Figure 23. Security Key

6. Your screen should look like this:

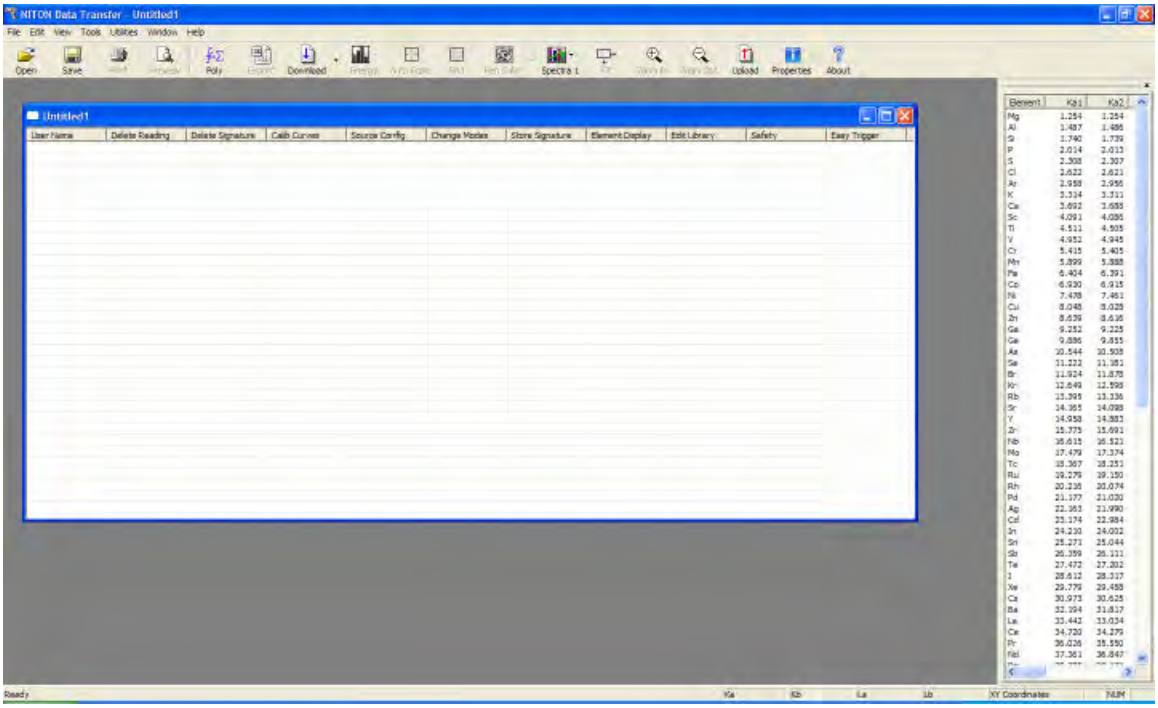


Figure 24. User Account Creation Screen

7. Right click, then select “New User”

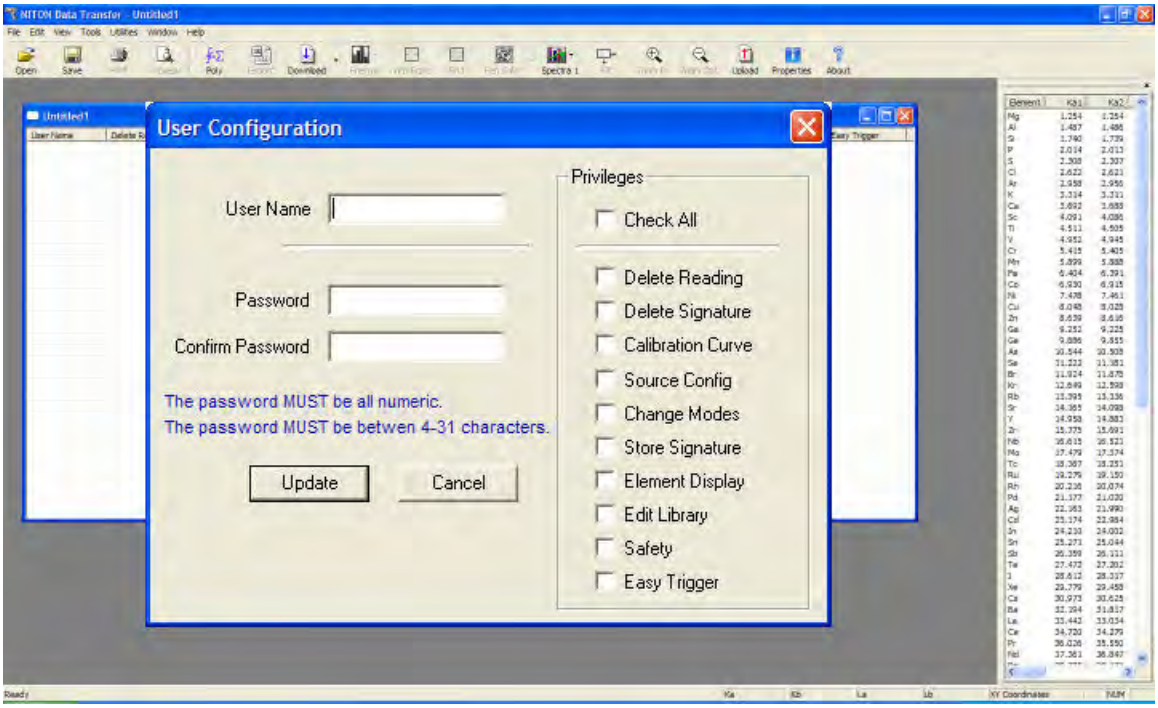


Figure 25. User Creation Dialog Box

8. Enter a user name and password, then select the privileges assigned to this user. Selecting the Check All check box will result in enabling all features.

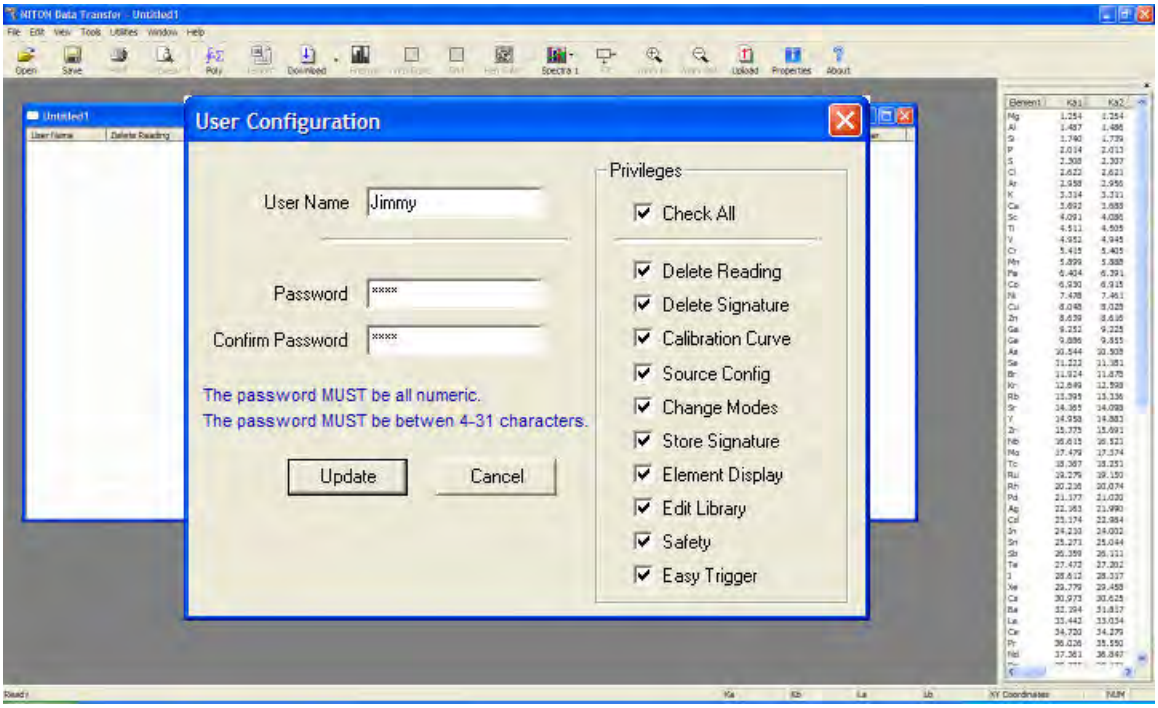


Figure 26. . Creating a User

WARNING it is recommended that only users at the highest level have access to the "Safety" feature. This should be unchecked for all other operators.

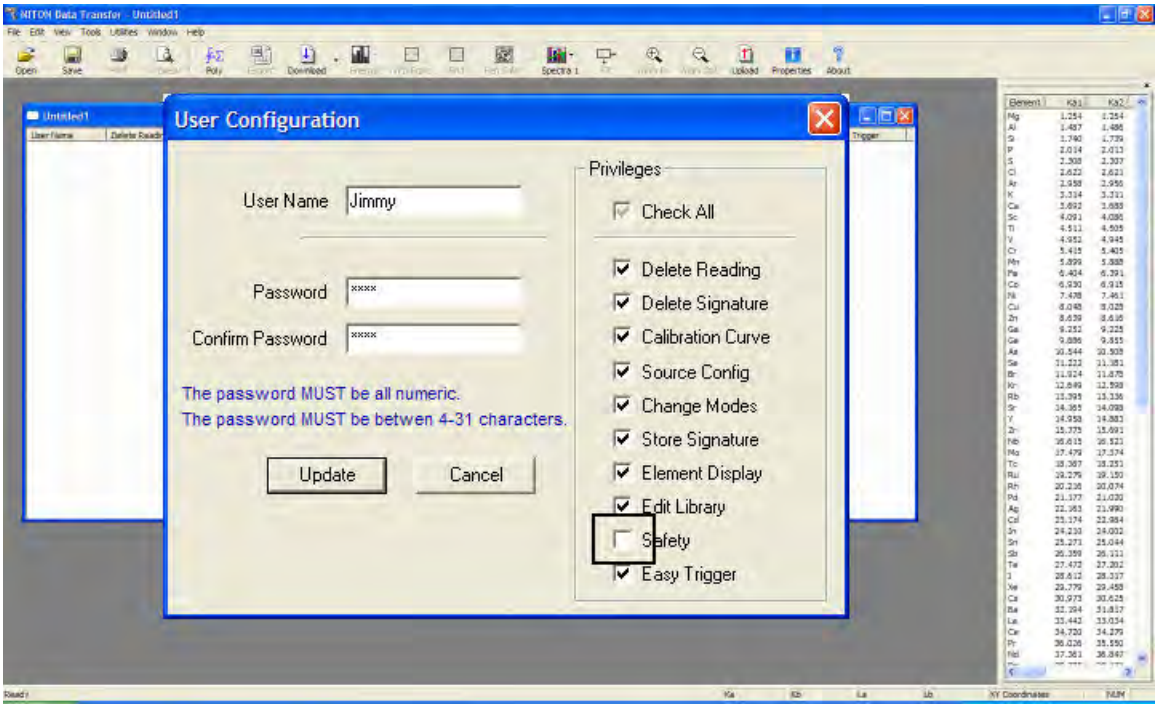


Figure 27. Unchecking Safety

9. Select the Update Button

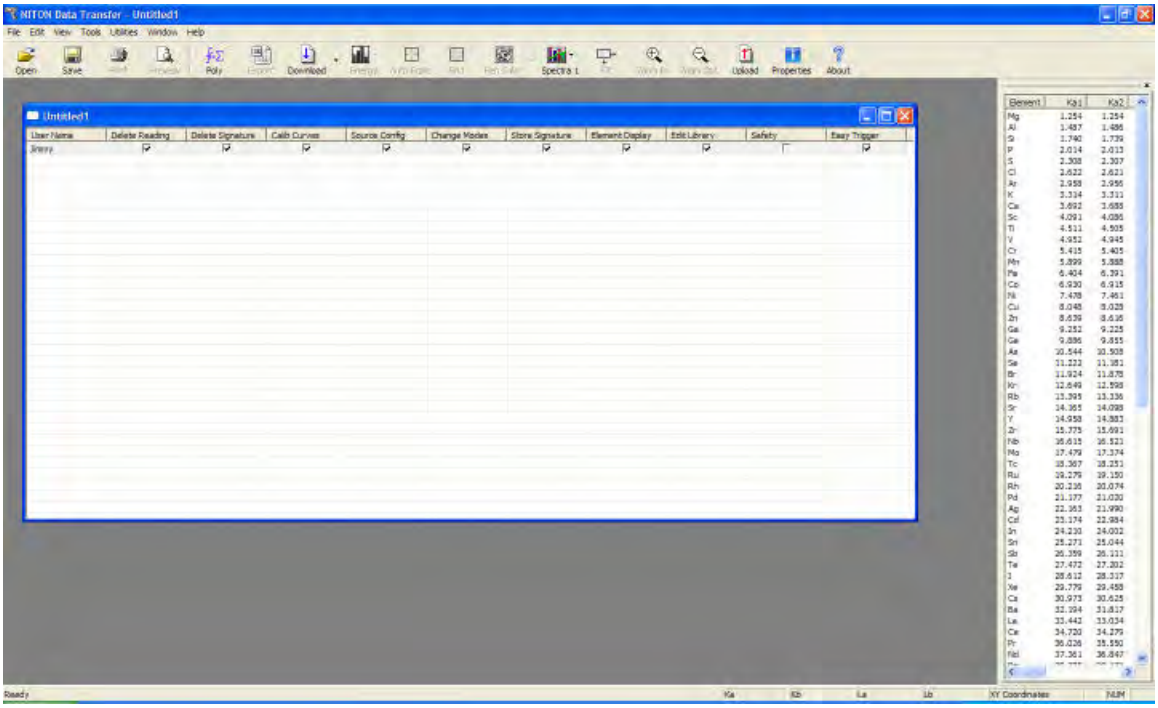


Figure 28. User is Created

a. You are now ready to upload your password file to the analyzer.

10. Be sure the analyzer is switched on; connect the analyzer using USB or serial connection.

11. Select the Upload icon.

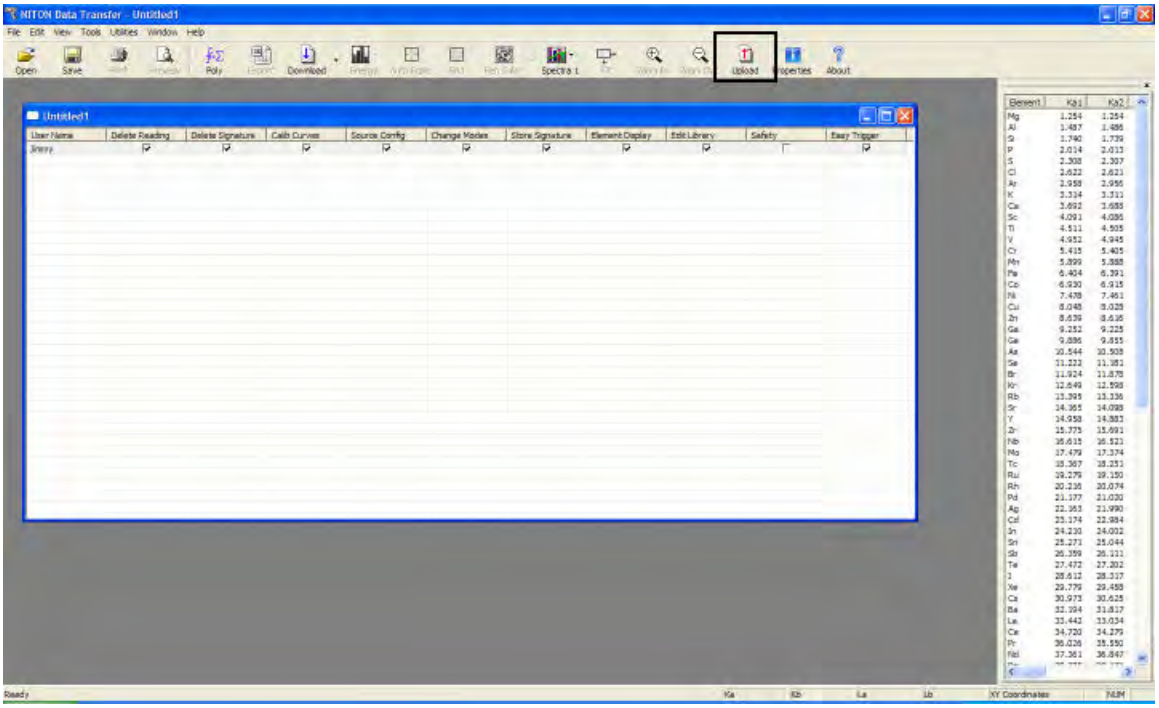


Figure 29. Selecting Upload

12. Your screen should look like this:

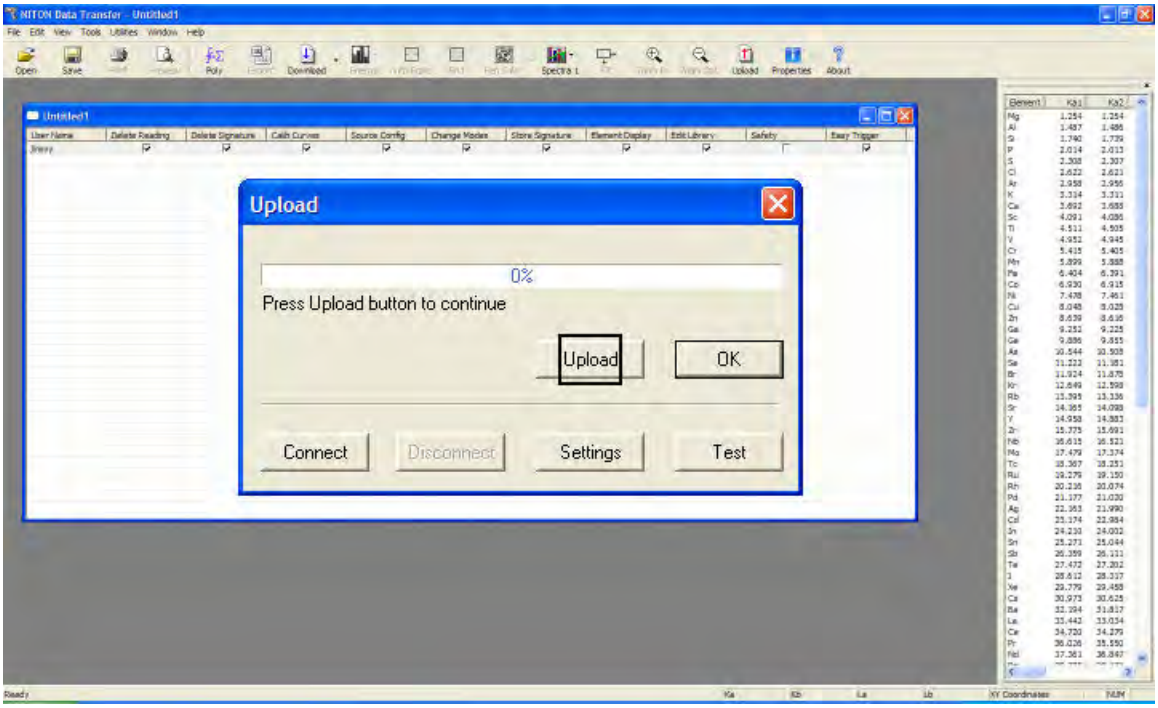


Figure 30. Selecting Upload

13. Select the Settings Button and choose the comm port that your analyzer is connect to.

14. Select the Connect Button, then the Upload Button.

15. Upon completion, you will receive a “File Upload Successfully Completed” message.

16. Click the OK Button; save your password file at this time by selecting the File icon then “Save As.

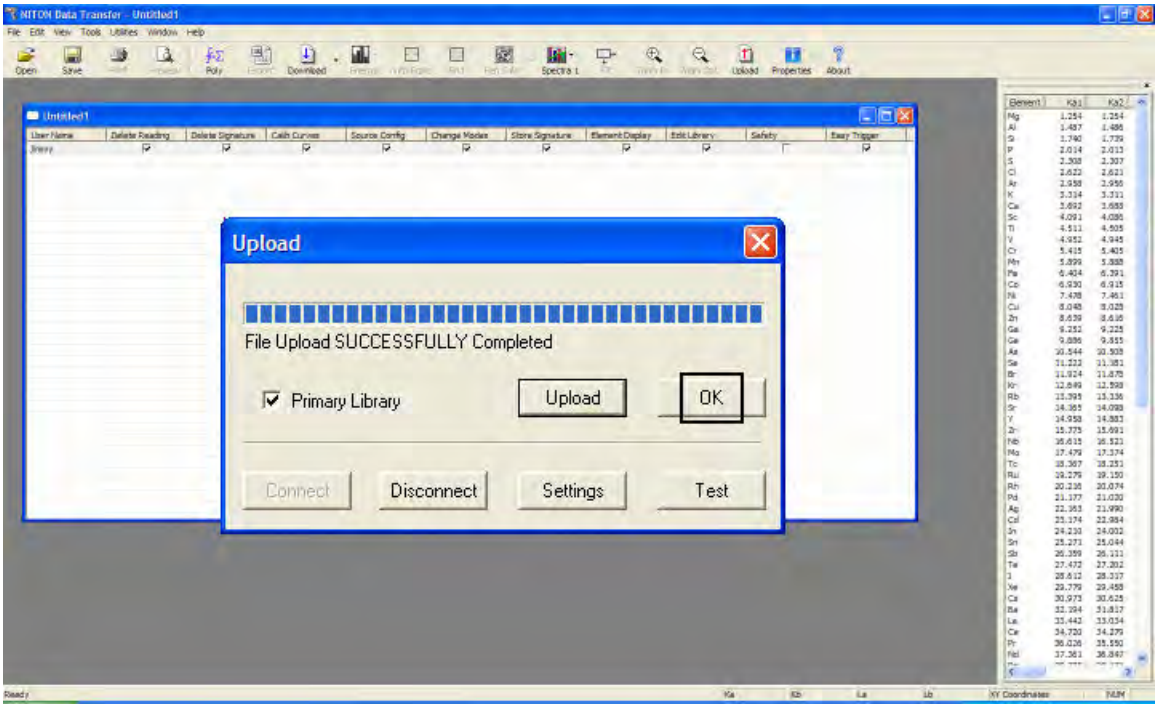


Figure 31. Successful Installation Message

17. Restart your analyzer; your password file should be successfully installed.

NDF Files: User Data Structuring

Creating New User-Defined Fields

You can create your own data entry fields for your Niton analyzer customized to your own needs and usage. These fields are saved in a special format called an NDF (Niton Data File) file. To create a new NDF file, select the File menu, then select New, then select New NDF File.

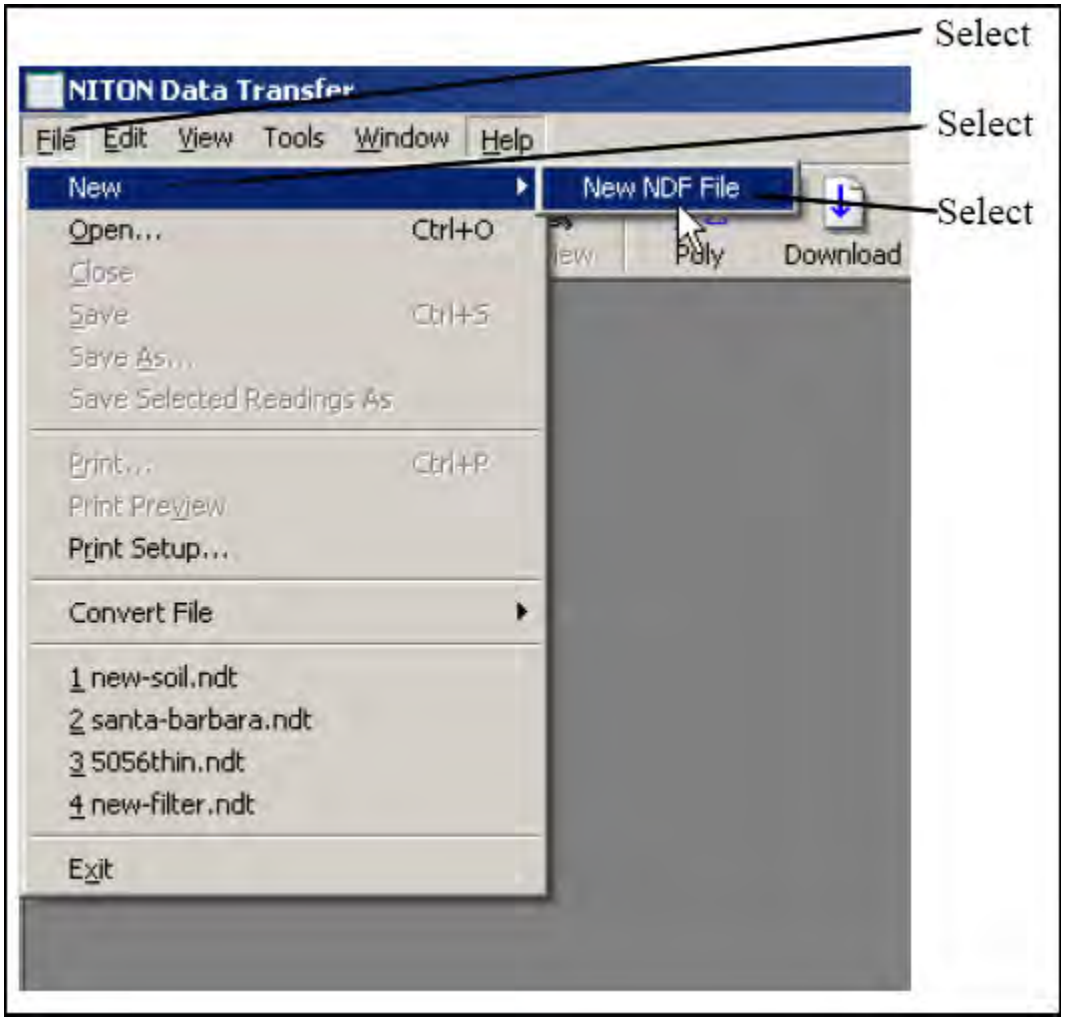


Figure 32. Creating a New NDF File

This will create a new window in which you can create your own fields, and specify their structure and parameters. The new window will appear with a single box, called "Untitled."

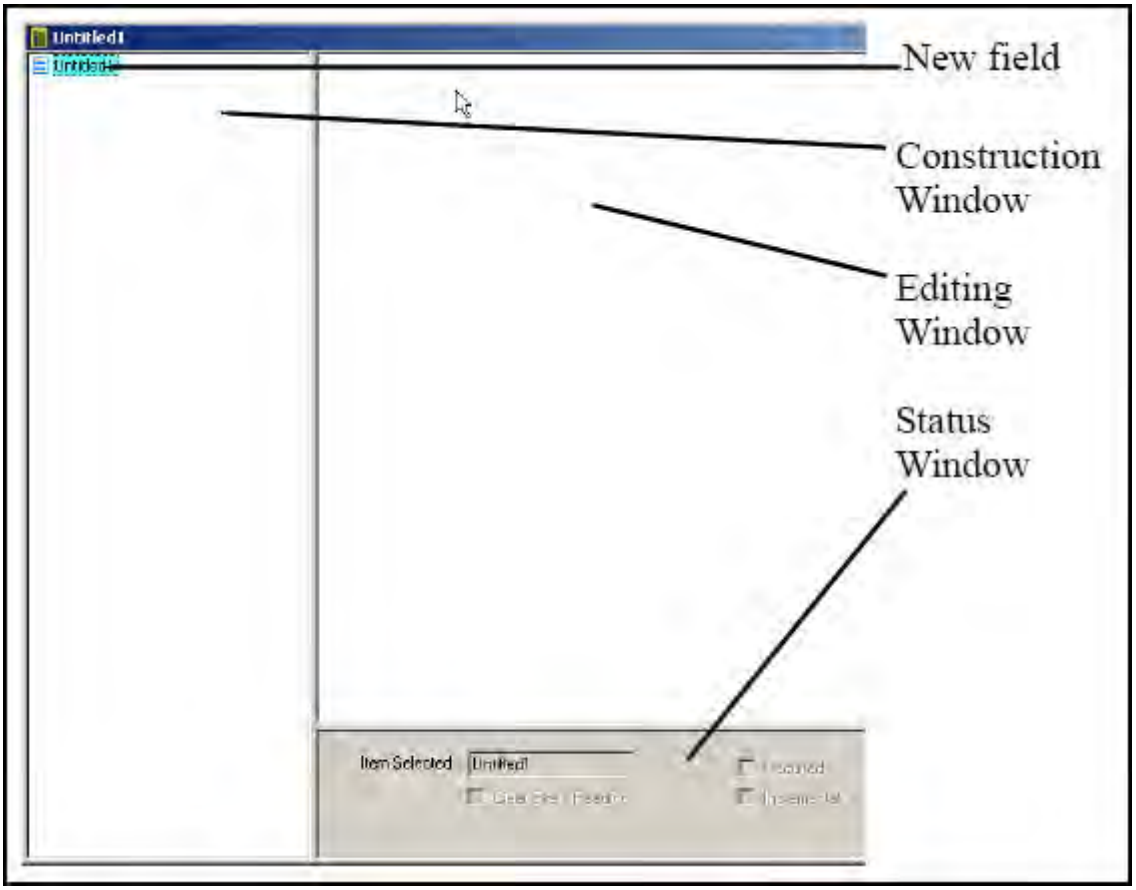


Figure 33. NDF File Work Area

By right-clicking on this box, you can access a pop-up menu allowing you to set the mode of the new data fields. Select New Mode to access the menu.

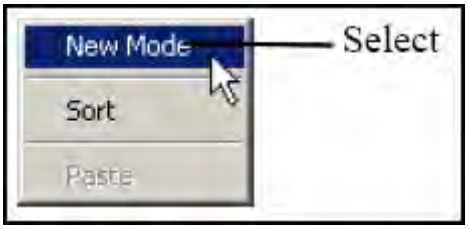


Figure 34. Selecting New Mode

The Mode you select will be the Mode within which the new data entry fields will appear. If you have multiple Modes enabled on your analyzer, the new fields will only be available from the Mode you select. Only the default fields will be available from the other Mode or Modes.

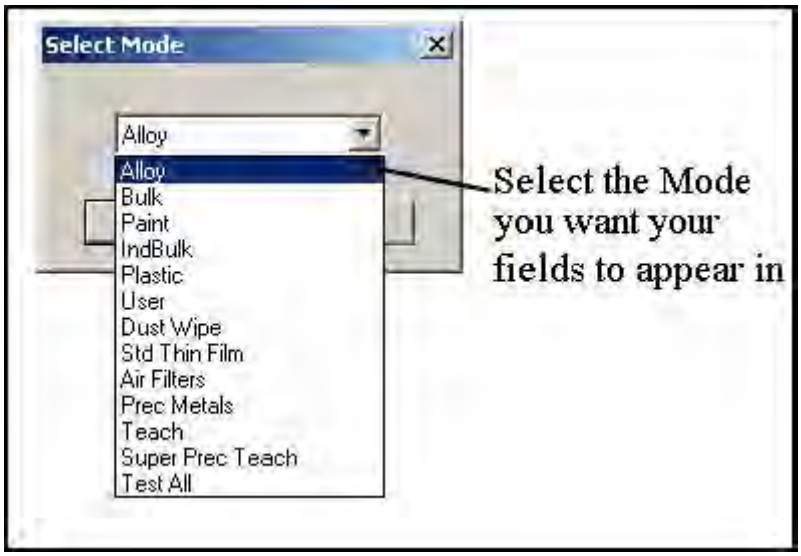


Figure 35. Selecting Mode

When you select the Mode for the new data fields, the Construction Window will change to look like this:

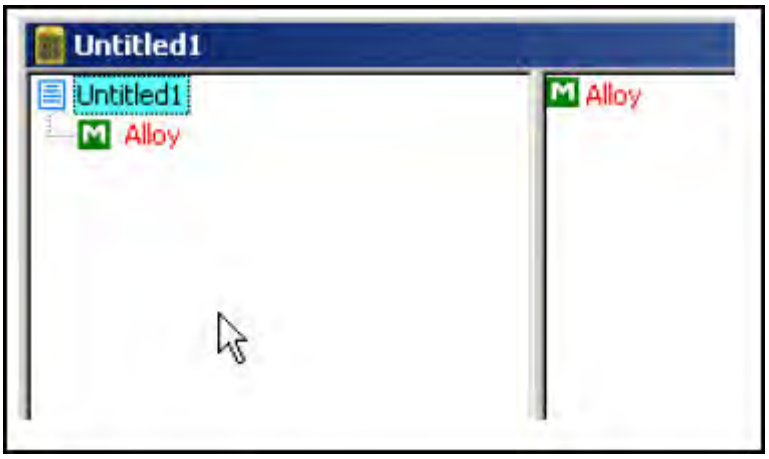


Figure 36. Working within a Mode

The "M" indicates the mode you have chosen - in this case Alloy Mode. Right click on the Mode name to access a pop-up menu.

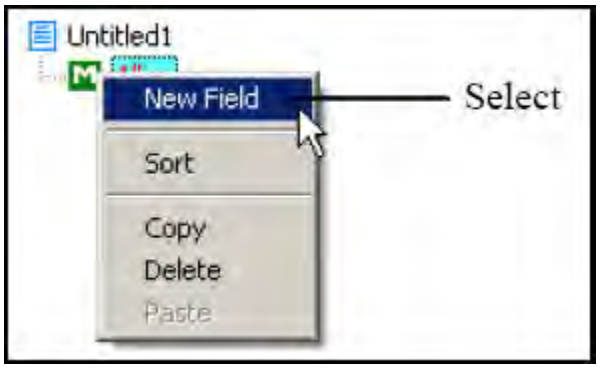


Figure 37. Mode Pop-Up Menu

Select New Field from the menu, and a blank new field will appear in the construction window.

Figure 38. Adding a New Field

Right clicking on the New Field box will bring up another pop-up menu. This menu gives you various options for using the field in your operations.

Selecting Required makes it mandatory that the new field be filled in prior to taking a measurement. This is very useful for necessary descriptors which vary from measurement to measurement, such as lot numbers, condi-tion descriptors, locations, etc.

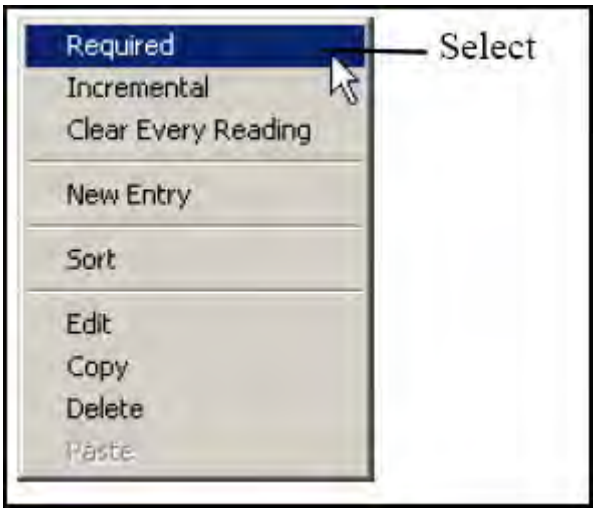


Figure 39. Making Fields Required

Selecting the Incremental option sets up a field which increments the field descriptor by one for each measurement taken. This option is handy for measuring several items with identical descriptors, such as samples within a single lot, or several instances of the same part number, because it appends the incremental number to the descriptor.

For example: P/N 455A2-1, P/N 455A2-2, P/N 455A2-3.

Another Example: Impeller-1, Impeller-2, Impeller-3.

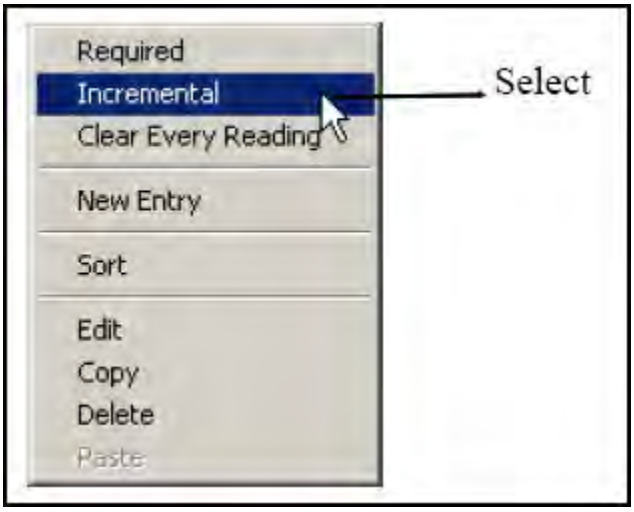


Figure 40. Making Fields Incremental

Selecting Clear Every Reading will toggle between two states. By default, the field will fill with the data which was input during the last reading. By selecting Clear Every Reading, you tell the instrument to clear the data from the field for each new reading, insuring that the person taking the reading must input new data each time. This is very useful for times when the data descriptor is expected to vary widely between readings.

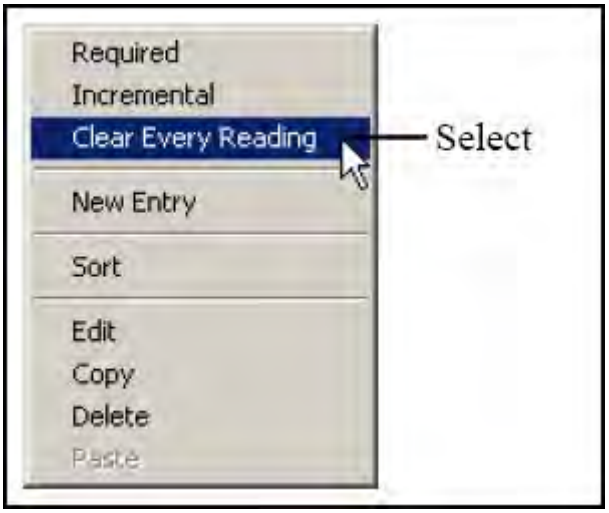


Figure 41. Clearing Data for New Readings

The state of each of these options can be seen in the Field Status Window at the bottom of the Construction Window. All options in effect for the field selected are checked.

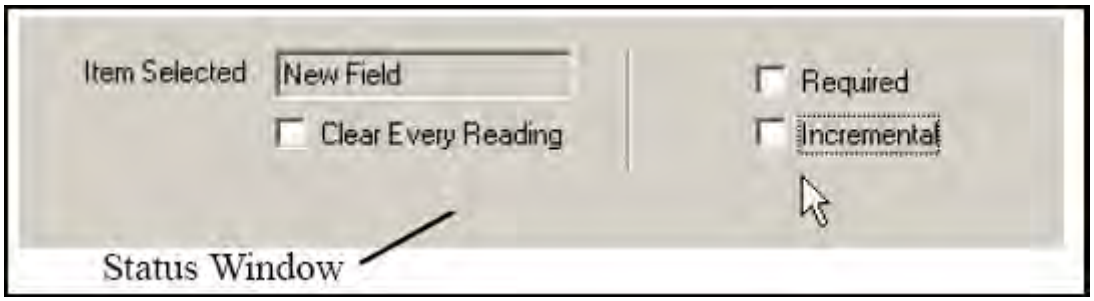


Figure 42. Field Status Window - Default

This shows a field with no options in effect, the default configuration. This is a field that will present the previous reading's data for this field - which may be changed by the user - without incrementing it, but does not require the user to input any data if there is none already there from a previous reading.

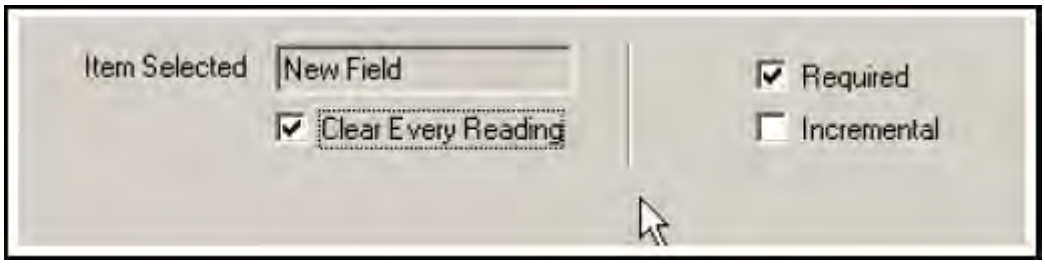


Figure 43. Field Status Window - Options Enabled

This shows a field with both Required and Clear Every Reading options in effect. This presents a field that is cleared for each reading, and must be filled in by the user before a reading is taken.

Selecting Edit from the pop-up menu allows you to edit the name of the field in the Editing Window to the right of the Construction Window.

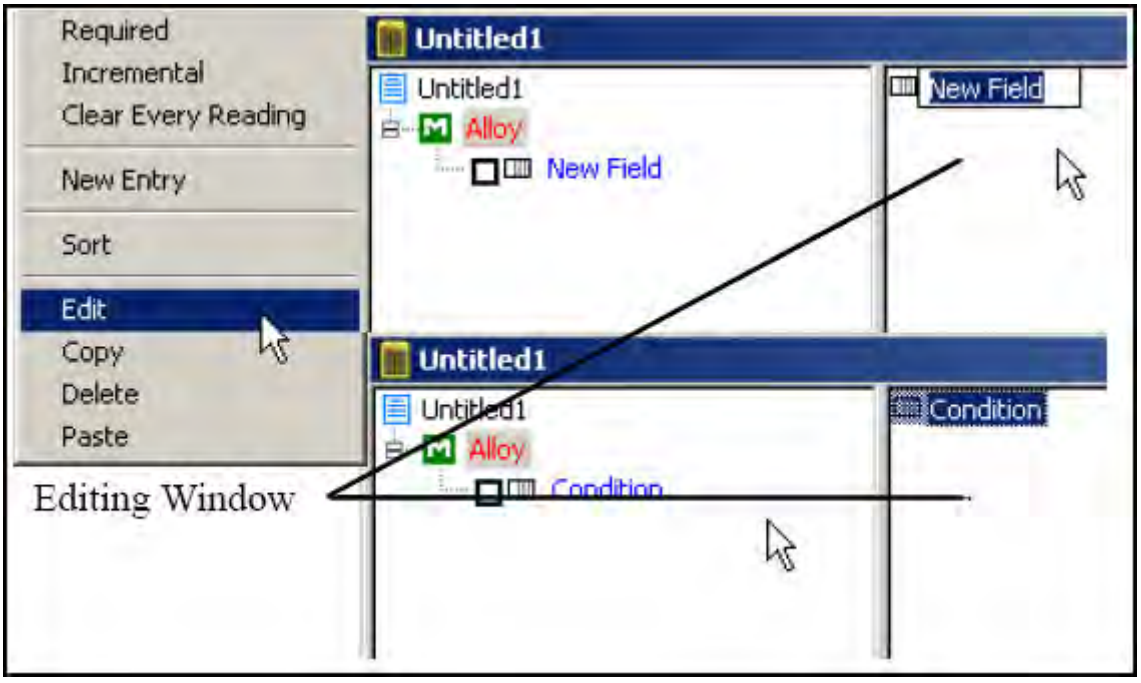


Figure 44. Editing the Field Name

Selecting the box to the left of the field toggles the Required option on or off.

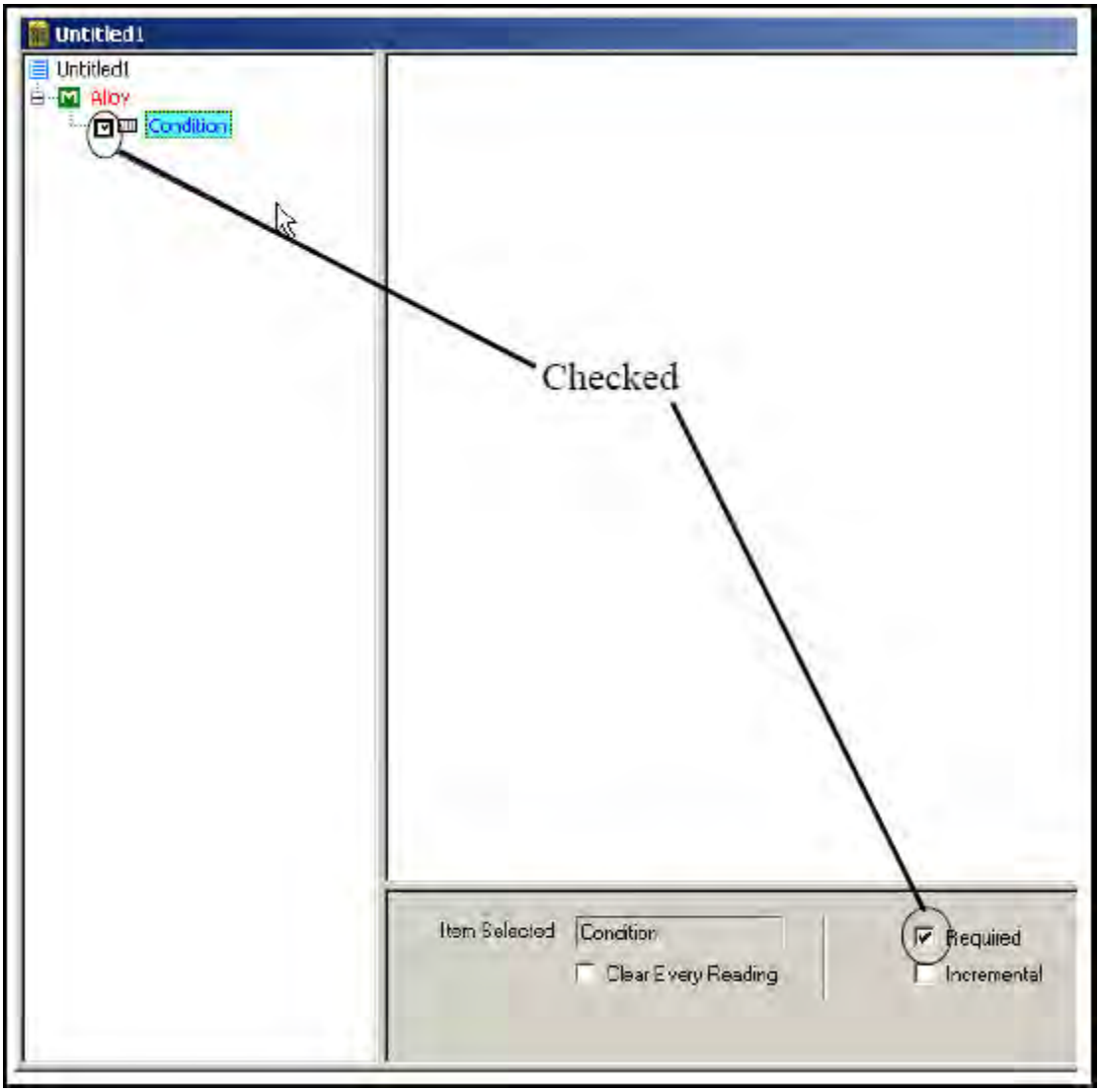


Figure 45. Toggling the Required Option

Selecting Copy from the pop-up window allows you to copy the currently selected field.

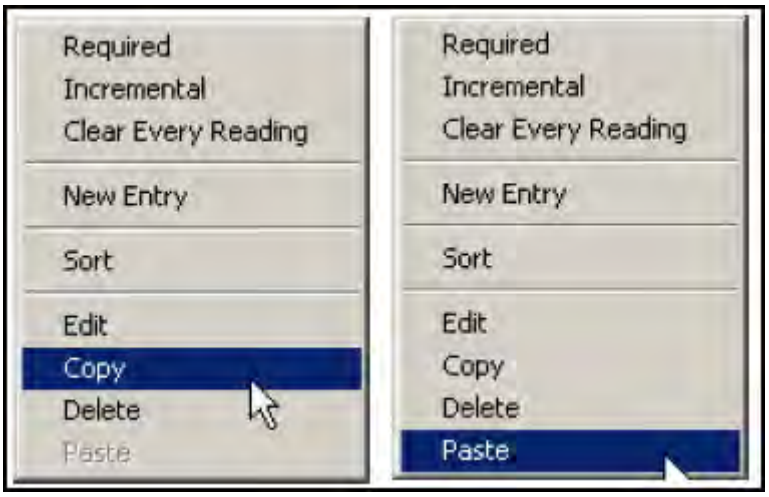


Figure 46. Copying the Current Field

Once you copy a field, the Paste option can be selected to paste the copied field into the Construction Window.

Figure 47. Pasting a Copied Field

Selecting the New Entry option from the pop-up menu allows you to define a choice for the user for this field.

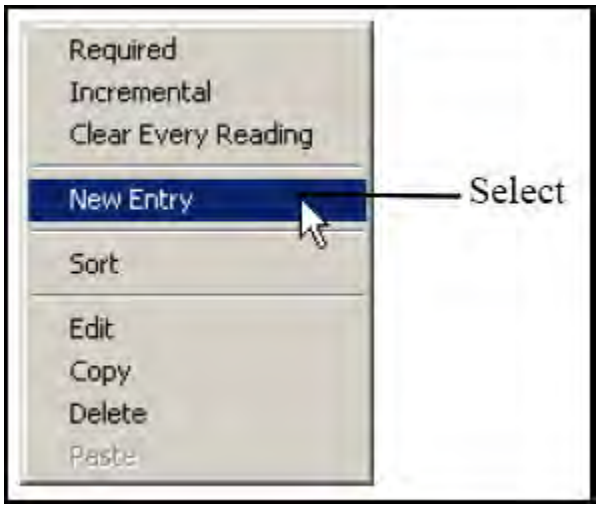


Figure 48. Creating a New Entry

This is a New Entry in the Construction Window.

Figure 49. New Entry in the Construction Window

The "E" is for "Entry." You can edit the entry once it is created, the same way as you edit the field name. Right click on the entry name, and choose Edit from the pop-up menu.

Figure 50. Editing the New Entry

You can sort your entries by name, alphanumerically, by right clicking on the field and selecting "Sort" from the pop-up menu.

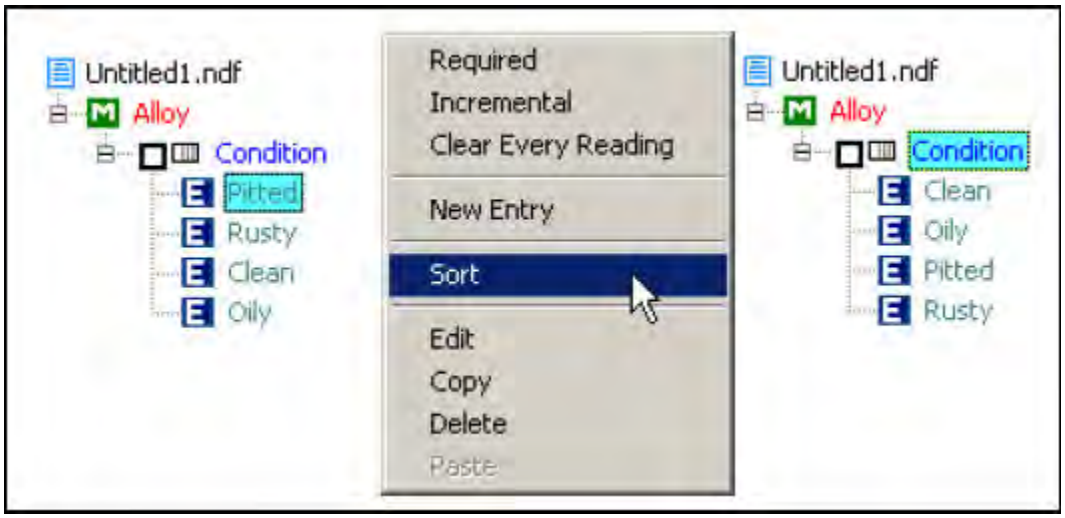


Figure 51. Sorting Entries

To delete a field or entry, just right click on the item you wish to delete, and select Delete From the pop-up menu.

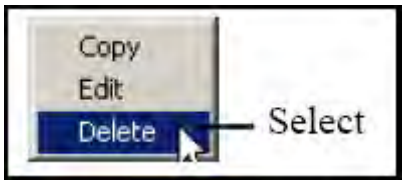


Figure 52. Deleting Fields and Entries

When you are finished creating your new NDF file, Upload it to your instrument using the Upload icon.

Figure 53. Uploading the NDF File

Make sure the instrument is connected to your computer by testing the con-nection first. Use the Test button on the Upload Window.

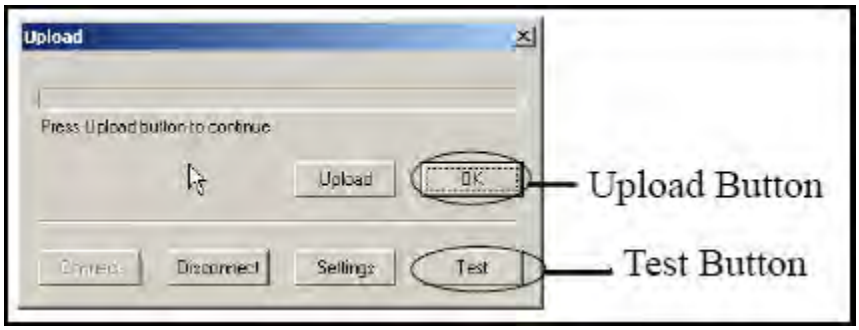


Figure 54. Testing the Upload Connection

Safety Settings

Access to the Safety Settings Screen is blocked unless the user logging in has explicitly been granted Safety access. The default login of 1234 does not have Safety access. See Passwords and User Privileges.

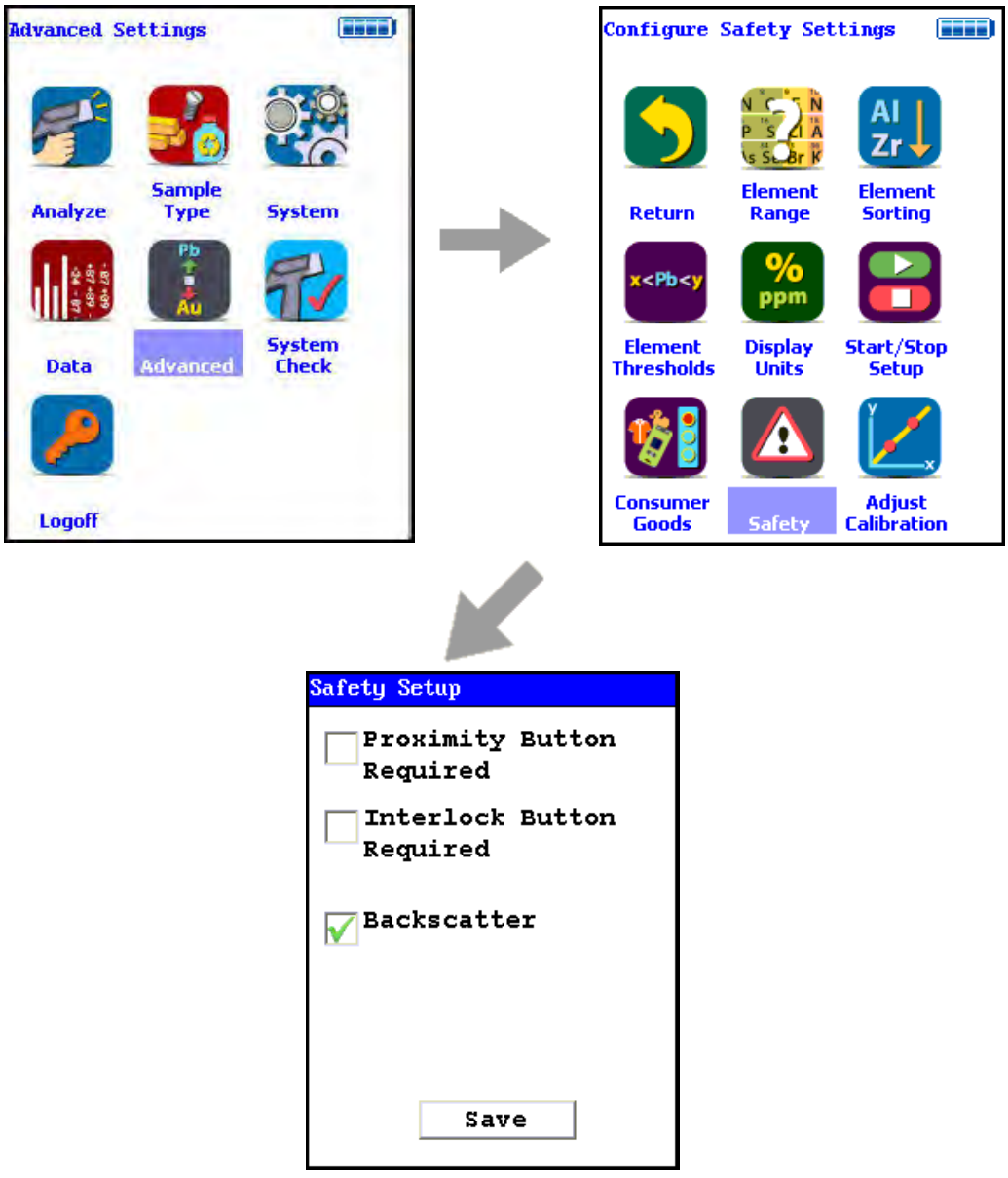


Figure 55. . Safety Settings Menu Path

The Safety Settings Screen enables you to change the Method of Operation for your analyzer. Each checkbox on the screen enables or disables the safety device named for purposes of the preconditions for operation. For example, checking the Proximity Button Required checkbox sets the engagement of the Proximity Sensor as a precondition for operation. Checking the Proximity Button Required checkbox and the Interlock Button Required checkbox sets the engagement of both the Proximity Button and the Interlock Button as preconditions for operation.

Safety settings always override start-stop settings. If your safety setting requires the use of the Proximity Button, you cannot set start-stop settings which ignore the Proximity Button. For example, the Easy Trigger start-stop setting must have the Backscatter safety setting enabled. While using Easy Trigger, you cannot disable Backscatter.

WARNING The backscatter sensor is enabled by default and acts as a recommended safety feature for most applications. Some sample types, however, cannot be analyzed when this feature is enabled. Samples that present very little mass to the analysis window, such as certain thin films, thin layers of plastic, and very thin wires, may not be of sufficient mass to allow the analysis to continue while backscatter is enabled. One should disable the backscatter feature only when necessary to analyze such low mass samples, and re-enable the feature when finished with these sample types. These samples also provide very little absorption of the primary x-ray beam so it is typically most appropriate to analyze these samples in a test stand when possible.

Start/Stop Setup

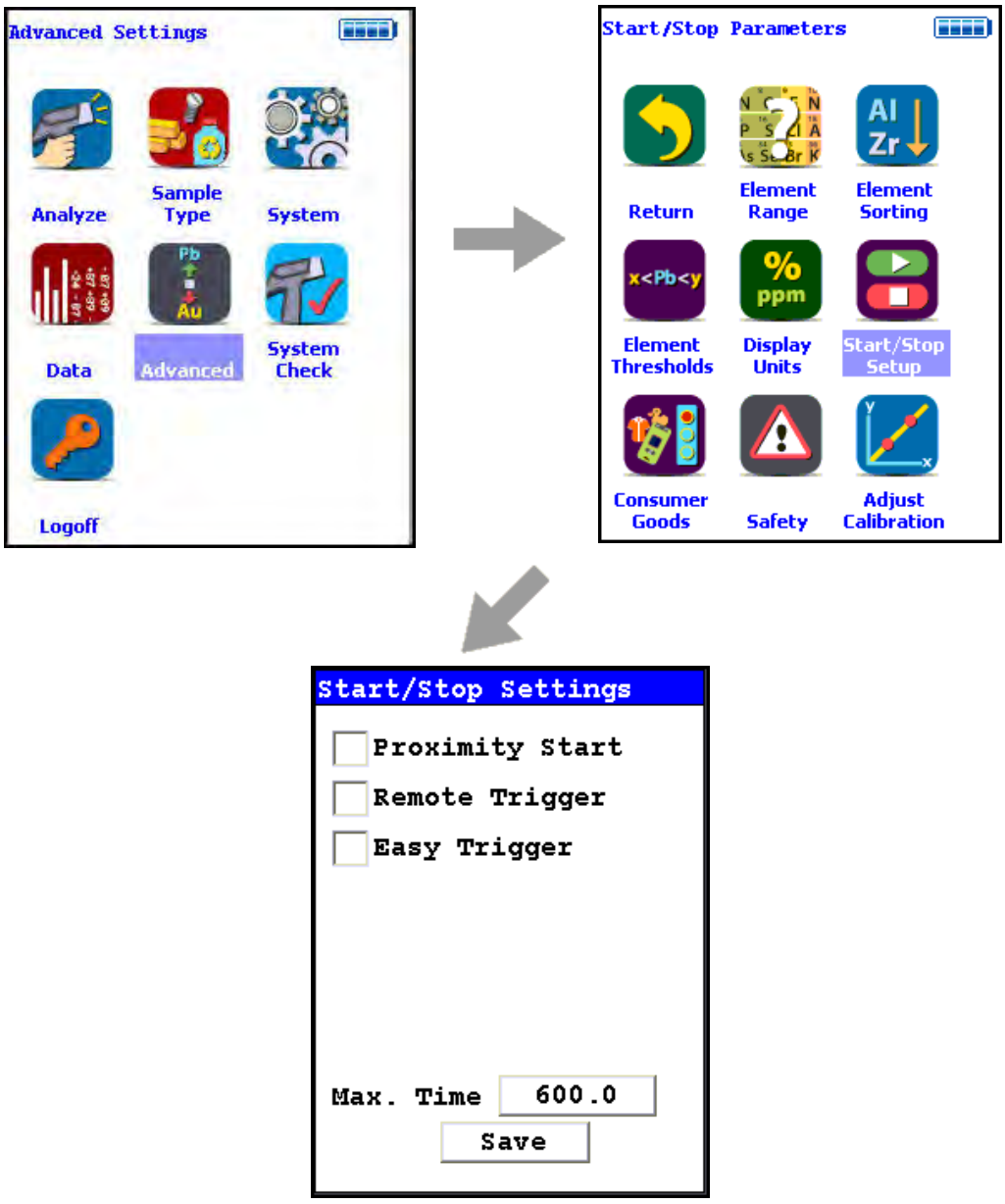


Figure 56. The Start/Stop Settings Menu Path

The Start/Stop Setting Screen enables you to change the preconditions for operation at a lower level than the Safety level. See Safety Settings for more information. Start/Stop settings cannot contradict Safety settings. If your safety setting requires the use of the Proximity Button, you cannot set start-stop settings which ignore the Proximity Button. For example, the Easy Trigger start-stop setting must have the Backscatter safety setting enabled. While using Easy Trigger, you cannot disable Backscatter.

The Start/Stop parameter options are Proximity Start, Remote Trigger, and Easy Trigger. There is also a field to set the maximum time for sample analysis before the analysis stops.

Proximity Start

Select the Proximity Start checkbox to use the Proximity Start parameters. Using Proximity Start, once the reading has been started, release of the Proximity Button will immediately stop the analysis. You cannot use Proximity Start with Easy Trigger.

Remote Trigger

Select the Remote Trigger checkbox to use the Remote Trigger parameters. Remote Tigger is used with the Extend-a-Pole accessory to control the analysis. With the Extend-a-Pole's input cable connected to the analyzer's Remote Trigger port, you can initiate and stop analysis remotely from the Extend-a-Pole's handle trigger. You can use Remote Trigger with either Proximity Start or with Easy Trigger.

Easy Trigger

Select the Easy Trigger checkbox to use the Easy Trigger parameters. Easy Trigger uses a single press and release of the trigger to initiate analysis, and a second press to stop analysis. Selecting Easy Trigger will immediately disable Proximity Start.

Max Time Field

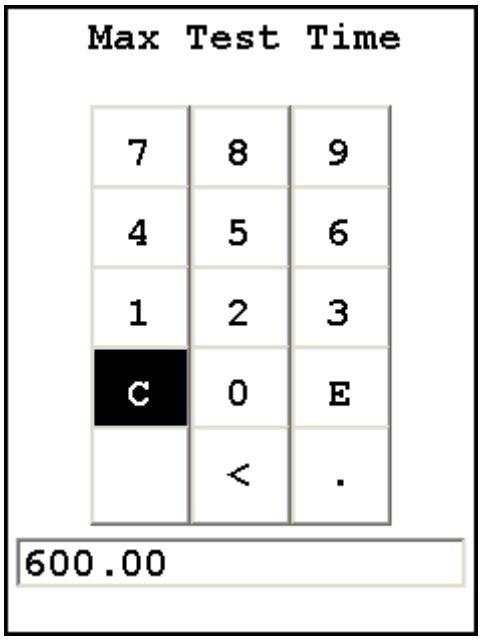


Figure 57. The Max Test Time Editor

Select the Max Time field to change the maximum analysis time parameter. Selecting the Max Time Field brings up a Virtual Numeric Keypad. To input the maximum number of seconds before automatic shutoff, select the C button to clear the current time, then from the Virtual Numeric Keypad, select each digit you want to input, then select the E button to enter. Of the non-numeric screen buttons, C = Clear All, E = Enter, and ">" will backspace over the current value. Selecting the E button will enter the current value as the Max Time, and return to the Start/Stop Settings Screen.

Save Button

Selecting the Save Button will save your current settings.

Methods of Operation

CAUTION After being powered on, your Niton Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your analyzer warm up for ten minutes after start up, before testing is begun.

There are six different methods of operation for taking a sample measurement, and your analyzer will be configured to use one of those methods for alloy samples, depending on the regulatory requirements of your locality. These methods are:

- Trigger-Only method. With the Trigger-Only method, you only need to place the measurement window flush with the sample to be analyzed and pull the trigger for sample analysis to be initiated.
- Trigger-and-Proximity-Sensor method. With the Trigger-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the analyzer, then pull the trigger for sample analysis to be initiated.
- Momentary-Trigger-Touch-and-Proximity-Sensor method. With the Momentary-Trigger-Touch-and-Proximity-Sensor method, you must place the measurement window against the surface to be analyzed to engage the proximity sensor on the front of the analyzer, then pull the trigger. The trigger may be released and the reading will continue until you release the proximity button, or other criteria (such as Max Time) are reached.
- Trigger-and-Interlock method. With the Trigger-and-Interlock method, you need to place the measurement window close to the sample to be analyzed, press and keep pressing the interlock button at the rear of the analyzer with your free hand, then pull the trigger for sample analysis to be initiated.
- Trigger-Interlock-and-Proximity-Sensor method. With the Trigger-Interlock-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the analyzer, press and keep pressing the interlock button at the rear of the analyzer with your free hand, then pull the trigger for sample analysis to be initiated.
- Easy Trigger method. With the Easy trigger method, you need only place the measurement window against the sample area and pull the trigger once to initiate a sample analysis. Your analyzer will continuously sample the backscatter, using a complex internal algorithm, to determine if the measurement window is against a sample or pointing to the empty air. If it finds that there is no sample directly against the measurement window, the analyzer will stop directing radiation through the window as soon as this determination is made.

The analyzer is constantly checking the backscatter characteristics to determine if a sample is against the measurement window, whether or not the Easy Trigger method is being used, and will shut off any radiation directed through the window if it determines that there is no sample present.

With any of these methods, analysis will stop if any one of the preconditions are violated. For example, with the Trigger-Interlock-and-Proximity-Sensor method, if the trigger or the Proximity Sensor or the Interlock is released, the reading will stop immediately, and the X-ray tube will shut down.

After your analyzer is calibrated, initiate a sample reading using the appropriate method. If you attempt to initiate a sample reading using a different method, the analyzer will inform you that one or more of the preconditions need to be met in order for sample analysis to begin.

Note The LED lights will blink whenever the x-ray tube is on.

WARNING The nose should not be touched during sample testing and calibration. If an ESD event occurs during measurement, the instrument may terminate the testing in progress and automatically reset to LogOn screen. Any test data collected prior to reset will be lost and the testing may have to be repeated.

WARNING The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, the x-ray tube will turn off, the calibration shutter will close, and the measurement will end. The LED lights will stop blinking when the measurement is ended. The flashing of the LED lights is not synchronized to minimize power consumption.

To end the test, simply release the trigger mechanism, or any other applicable preconditions.

Camera

The Camera feature is only usable with properly configured analyzers, and the Small Spot feature is only available on Small Spot analyzers.

If your analyzer is equipped with an internal video camera, you can turn that camera on and off, and turn the saving of images with the readings on and off through an interface. When the camera is on, the image will show in the Ready to Test screen, as in Figure 123. If the camera is off, saving of images will also be off. If the camera is on and the image saving function is also on, the images will automatically be saved with the reading. Saving images will curtail the maximum number of readings stored.

How to Use the Camera

When a Camera equipped analyzer is in the Ready to Test screen, the video feed appears live on the analyzer's touch screen. This is the image that can be saved with the sample analysis. When you take a measurement, if you choose to do so, the bitmap image will be saved on the analyzer along with the analysis results. The interface is accessible through the System menu, as in Figure 120.

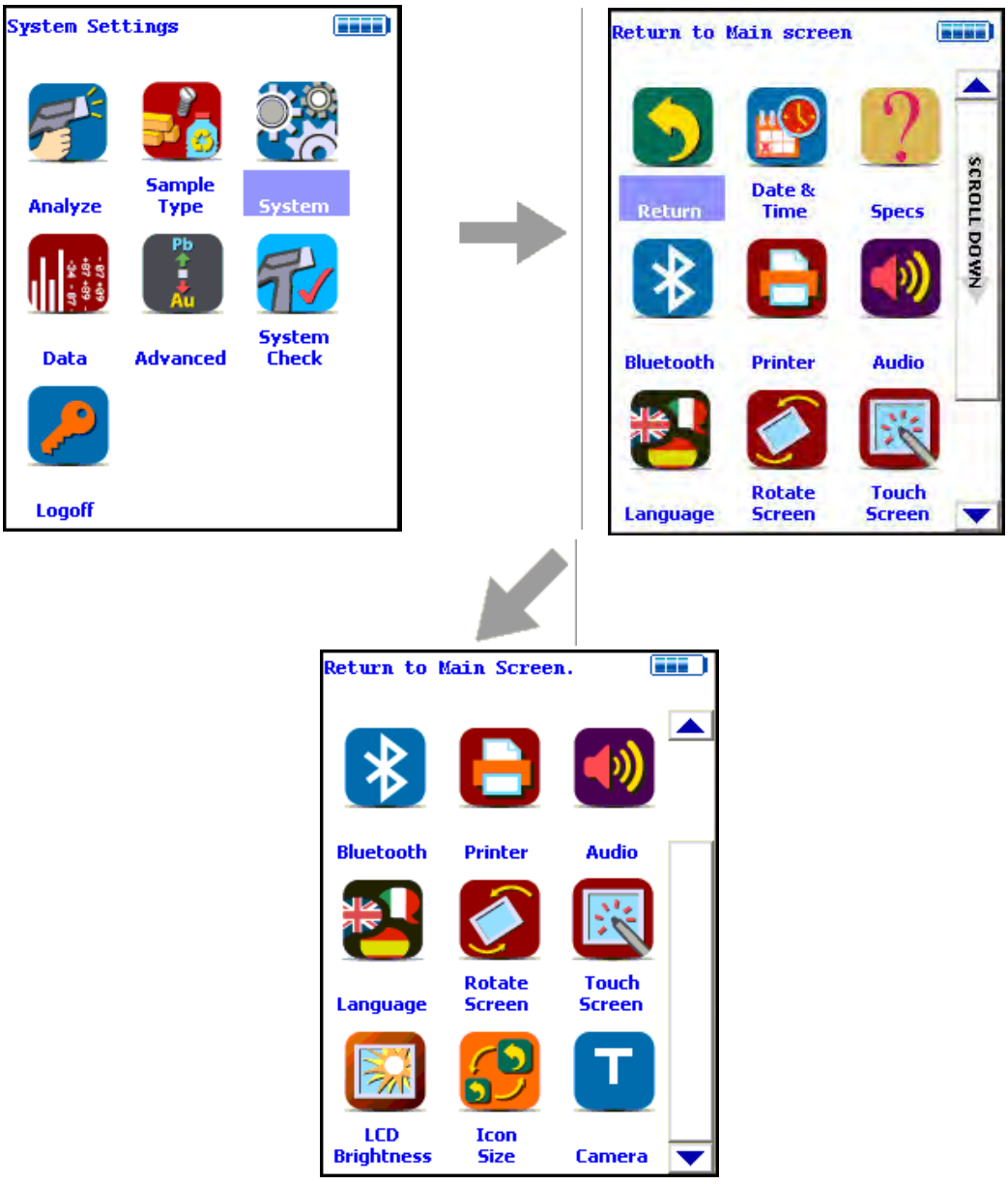


Figure 58. The Camera Menu Path

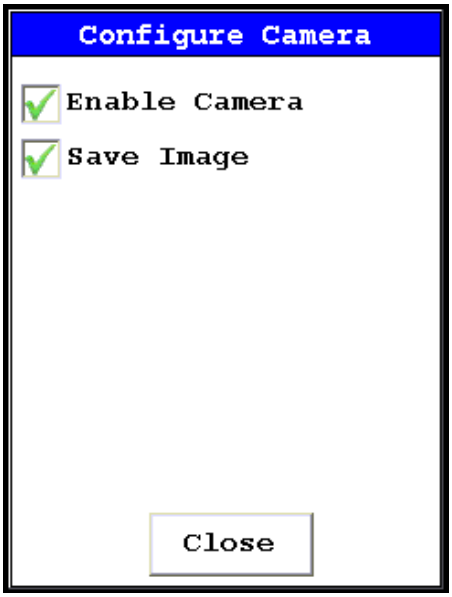


Figure 59. Setting Up the Camera View and Image Saving

Selecting the empty checkbox next to Enable Camera will turn the internal camera on, displaying the camera view in the Ready to Test screen. Selecting the checkbox again turns the camera off. Enable Camera is enabled by default.

Selecting the empty checkbox next to Save Image will enable image saving with the analysis. Selecting the checkbox again will disable automatic saving of image data. Save Image is enabled by default.

Stored camera images from previous measurements can be viewed on the analyzer.

Service

See "Contact Us" on page 1 to find your nearest Service facility.

Warranty

Warranty statement

Seller warrants that the Products will operate or perform substantially in conformance with Seller's published specifications and be free from defects in material and workmanship, when subjected to normal, proper and intended usage by properly trained personnel, for the period of time set forth in the product documentation, published specifications or package inserts. If a period of time is not specified in Seller's product documentation, published specifications or package inserts, the warranty period shall be one (1) year (unless otherwise agreed upon at time of purchase) from the date of shipment to Buyer in the country of purchase. Any part replaced on an instrument, covered by the original factory warranty, will be warranted for the remainder of the instrument's factory warranty. Seller agrees during the Warranty Period, to repair or replace, at Seller's option, defective Products so as to cause the same to operate in substantial conformance with said published specifications; provided that Buyer shall (a) promptly notify Seller in writing upon the discovery of any defect, which notice shall include the product model and serial number (if applicable) and details of the warranty claim; and (b) after Seller's review, Seller will provide Buyer with service data and/or a Return Material Authorization ("RMA"), which may include biohazard or other Radiation safety decontamination procedures and other product-specific handling instructions, then, if applicable, Buyer may return the defective Products to Seller with all costs prepaid by Buyer. Replacement parts may be new or refurbished, at the election of Seller, the warranty of these parts expire with the instrument warranty. All replaced parts shall become the property of Seller. Shipment to Buyer of repaired or replacement Products shall be made in accordance with the Delivery provisions of the Seller's Terms and Conditions of Sale. Accessories and Consumables are expressly excluded from this warranty (see list A for details).

Notwithstanding the foregoing, Products supplied by Seller that are obtained by Seller from an original manufacturer or third party supplier are not warranted by Seller, but Seller agrees to assign to Buyer any warranty rights in such Product that Seller may have from the original manufacturer or third party supplier, to the extent such assignment is allowed by such original manufacturer or third party supplier.

In no event shall Seller have any obligation to make repairs, replacements or corrections required, in whole or in part, as the result of (i) normal wear and tear, (ii) accident, disaster or event of force majeure, (iii) misuse, fault or negligence of or by Buyer, (iv) use of the Products in a manner for which they were not designed, (v) causes external to the Products such as, but not limited to, power failure or electrical power surges, (vi) improper storage and handling of the Products, (vii) use of the Products in combination with equipment or software not supplied by Seller, (viii) Moderately heavy or excessive impact against any object, including but not limited to floors, walls, furniture, sample or other objects. A shock sensor is fitted inside of the instrumentation; warranty is void if this shock sensor is activated, (ix) Excessive water, moisture or condensing humidity that breaches the instrument seals, (X) Excessive or extreme ambient or direct temperature or (vi) Heavy vibrations directly to the instrument for extended periods of time. If Seller determines that Products for which Buyer has requested warranty services are not covered by the warranty hereunder, Buyer shall pay or reimburse Seller for all costs of investigating and responding to such request at Seller's then prevailing time and materials rates. If Seller provides repair services or replacement parts that are not covered by this warranty, Buyer shall pay Seller therefore at Seller's then prevailing time and materials rates.

ANY INSTALLATION, MAINTENANCE, REPAIR, SERVICE, RELOCATION OR ALTERATION TO OR OF, OR OTHER TAMPERING WITH, THE PRODUCTS PERFORMED BY ANY PERSON OR ENTITY OTHER THAN SELLER WITHOUT SELLER'S PRIOR WRITTEN APPROVAL, OR ANY USE OF REPLACEMENT PARTS NOT SUPPLIED BY SELLER, SHALL IMMEDIATELY VOID AND CANCEL ALL WARRANTIES WITH RESPECT TO THE AFFECTED PRODUCTS.

THE OBLIGATIONS CREATED BY THIS WARRANTY STATEMENT TO REPAIR OR REPLACE A DEFECTIVE PRODUCT SHALL BE THE SOLE REMEDY OF BUYER IN THE EVENT OF A DEFECTIVE PRODUCT. EXCEPT AS EXPRESSLY PROVIDED IN THIS WARRANTY STATEMENT, SELLER DISCLAIMS ALL OTHER WARRANTIES, WHETHER EXPRESS OR IMPLIED, ORAL OR WRITTEN, WITH RESPECT TO THE PRODUCTS AND INCLUDING WITHOUT LIMITATION ALL IMPLIED WARRANTIES OF MERCHANTABILITY OR FITNESS FOR ANY PARTICULAR PURPOSE. SELLER DOES NOT WARRANT THAT THE PRODUCTS ARE ERROR-FREE OR WILL ACCOMPLISH ANY PARTICULAR RESULT.

Accessories, Spares and Consumables - exclusions

(List A)

Specific warranties of some common accessories:

- Battery Charger and batteries - 12 months
- Instrument accessories - 12 months
- Consumable - no warranty

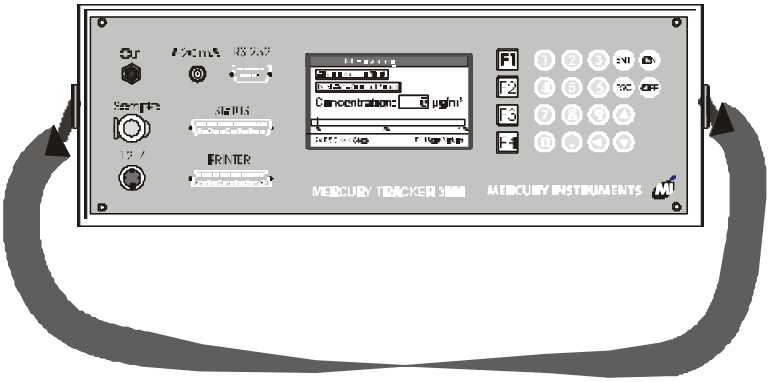
- Soil Grinder - no warranty
- Single-stage or two stage helium tank regulator - 12 months
- Test stands, extend-a-poles and docking stations – 12 months
- Parts or spares sold, installed or supplied outside of the product warranty period and not listed above – 12 months

ATTACHMENT E-3

OPERATING MANUAL MERCURY TRACKER 3000

Operating Manual

Mercury Tracker 3000



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Analytical Technologies

General information for safe operation

While operating the TRACKER-3000, parts of its interior are under high voltage and UV-beams are produced inside. If safety regulations are ignored physical and/or material damages could occur. Only qualified personal should be allowed to operate the TRACKER-3000. Following conditions for correct function of the TRACKER-3000 are to be held: careful and correct storage, proficient operation and maintenance.

- Do not operate instrument if it is damaged.
- When connecting the TRACKER-3000 to a power source please note the related safety regulations.
- The TRACKER-3000 should be operated from a type of power source delivering max. 13.5 V_{DC}.
- Make sure that plugs and power cords are not damaged.
- Regulations for prevention of accidents are to be followed.
- Before opening the TRACKER-3000 disconnect it from the external power supply.
- Repairs and maintenance on the opened and powered instrument should only be carried out by trained personnel.
- Operate the TRACKER-3000 on a stable and dry surface. The interior of the TRACKER-3000 should never get moist or wet. In case it happens, consult an expert.
- The TRACKER-3000 is dedicated for the measurement of mercury concentration in gases. Do regard the related dangers especially while operating with toxic gases. Make sure that these parts of the instrument guiding the gas are not damaged and that the gas is guided back to the source or into an absorber. If explosive gases are measured or if measurements are made at places where explosion is possible, please follow the specific safety instructions.

Contents

1. General description

1.1 Fields of application

The TRACKER-3000 serves for continuous measurement of the mercury concentration in gases like air, nitrogen, hydrogen.

Following applications are examples for the versatility of the TRACKER-3000:

- Tracing of mercury spillages in rooms.
- Survey of mercury in contaminated areas like abandoned plants which used mercury in production processes.
- Control of sanitation/decontamination work
- Measurement of mercury in laboratory room air of high schools and universities. Mercury was used very often in the past for thermometers and barometers. Therefore such rooms where practical exercises have been performed by students often show increased mercury concentrations.
- Measurement of mercury at working places where mercury is (or was) used. Such working places may be dentists laboratories, thermometer manufacturers, fluorescence lamp manufacturers, chlor-alkali production plants, battery manufacturers.
- Measurement of mercury in recycling plants for mercury containing material like lamps, batteries, filters and other components from natural gas industry.
- Monitoring of mercury in laboratories where mercury is used for example in porosimeters or diffusion pumps.
- Measurement of mercury in gases for quality control (hydrogen, nitrogen, calibration gases)
- As a highly sensitive mercury detector for research work
- Geochemical applications

figure: tracing mercury spillage with the TRACKER-3000

1.2 Measuring principle

Basis for determination of the mercury concentration is the resonance absorption of the Hg-atoms at a wavelength of 253.7 nm. The sample gas is drawn through a 1 micron PTFE filter into the optical cell by a membrane pump. The optical cell is entirely made of synthetic quarz glass. Radiation of a mercury lamp passes through the cell and is measured by a solid state detector. The attenuation of the UV light reaching the detector depends on the number of mercury atoms in the optical cell. The internal computer performs the quantitative evaluation of the mercury concentration in the sample in real-time. In order to get an extremly stable baseline, the UV-light source is controlled by a reference beam and reference detector device. In addition to this, the UV detectors of the TRACKER-3000 are thermostatically controlled.

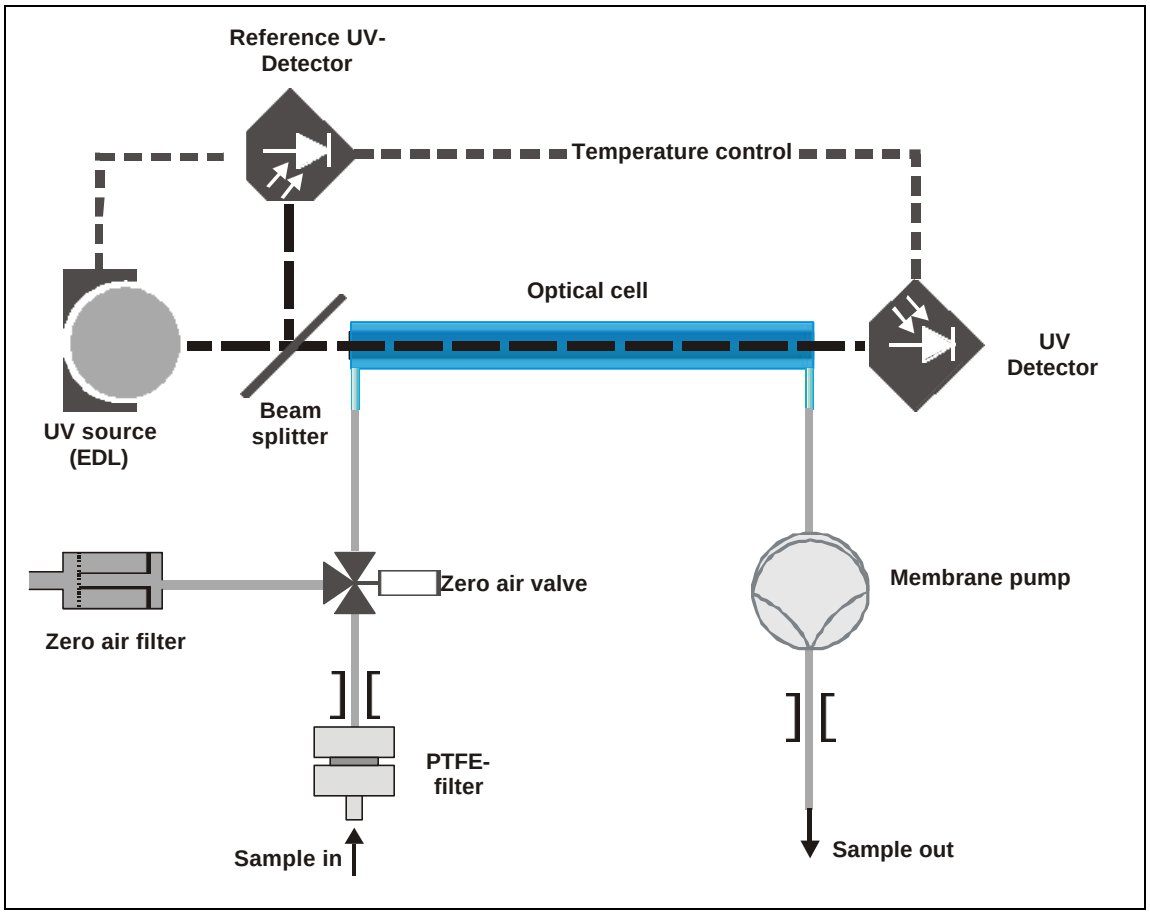


Figure: Schematic diagram of the TRACKER-3000

2. Preparation for operation

The TRACKER-3000 is unpacked and placed on a flat surface (e.g. table). The enclosed shoulder strap is engaged to the small triangles on both sides of the Tracker.

3. Installation

3.1 Electrical power supply

The Tracker-3000 can be operated with power from the built-in batteries as well as with the external 12 V power supply delivered with the instrument. Any other 12 V DC power source can also be used for powering the Tracker-3000. Please note that the power source has to deliver a minimum of 3 Amps. The voltage of the power source has to be max. 13.5 V. The 12 V power cord is connected to the 12 V receptable on the front of the TRACKER-3000. For operation turn on the TRACKER-3000 by pushing the ON button on the front panel.
If „LOW BATTERY“ is indicated on the display the TRACKER-3000 can still operate a few minutes in order to give the operator some time to finish measuring and recharge the batteries.

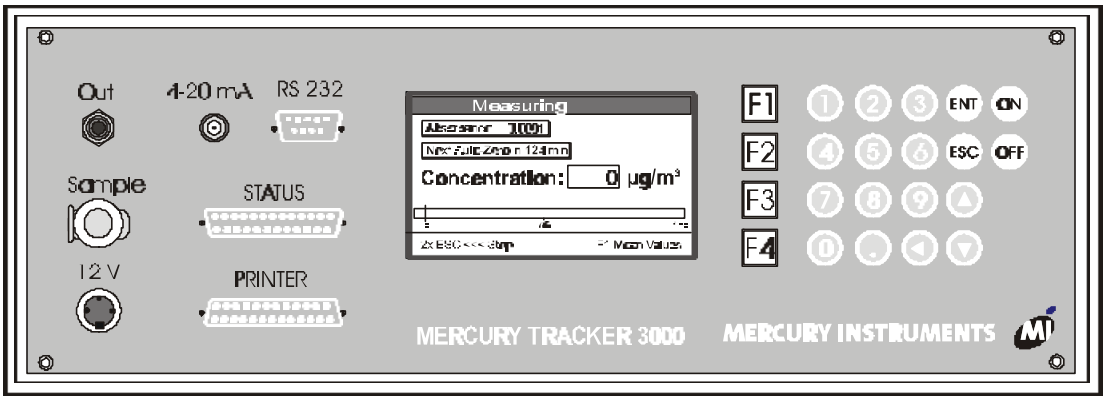
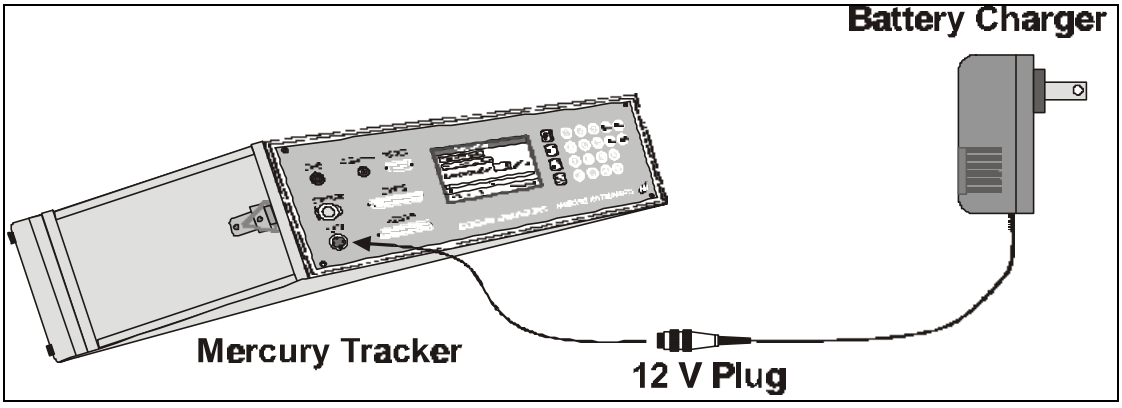


Figure: Front panel of TRACKER-3000

3.2 Operation on mains power with external power supply

The TRACKER-3000 can be operated with mains using the included power supply.

Figure: Charging of the Mercury Tracker 3000



3.2.1 Battery charger

The included charger is designed to charge the nickel-cadmium battery pack of the TRACKER-3000. Complete charging of empty battery packs takes approximately 10 hours.
Note that new battery packs or packs that have not been used for a longer period, in the beginning only use part of their capacity.

Operation :

➲ Switch off the Mercury Vapor Monitor.

➲ Disconnect Mercury Vapor Monitor from mains power supply by pulling the power plug.

➲ Plug battery charger into wall socket.

➲ Connect the round 3-pin plug of charger secondary cable into 12 V socket of Mercury Vapor Monitor

➲ Charging of the NiCd powerpacks will start automatically once being properly connected.

➲ Red LED will flash for about 10 seconds, battery contact detection - test phase.

Note: If the red LED keeps flashing after the test phase, the battery pack may be broken.

➲ The test phase is followed by the charging procedure (red LED on). After termination of the charging time the device will automatically switch over to impulse-trickle-charge. This is indicated by a green LED, red LED off.

Features:

- microcontroller controlled charging
- battery test phase at the beginningof the charging in order to detect and indicate defect battery packs
- short circuit detection
- electronic protection against reversed battery
- battery condition at the beginning of the charging is of no importance for the battery packs
- supervision of the charging condition by a microcontroller during the whole charging time
- safety stages like voltage gradient supervision and -delta U switch off as well as a safety timer are integrated
- automatic switching over to trickle charge
- button for discharging of the battery packs; after that automatically switching over to charging

To avoid memory effect (loss of capacity due to frequent partly discharging), you have to discharge the battery pack every now and then. After the test phase is finished, press the „PRESS" button aproximately 2 seconds (red LED flashing). After discharging, which can in individual cases last for several hours, the charger automatically switches over to charging.

LEDs:

Red LED flashing: battery contact detection (test phase, ca. 10 seconds)
battery pack reversed
battery pack broken or unsuitable amount of cells
discharging after pressing the PRESS button
Red LED on: Charging
Green LED on: Battey fully charged, trickle charge

Charging times:

Capacity: 5000 mAh

Charging time: approx. 10h

Safety:

Keep your charger in a dry place (indoor use only). Do not plug in the charger when housing or power plug is damaged. Do not charge dry batteries. Danger of explosion!

Only charge rechargable NiCd/NiMH battery packs.
Do not open the charger. Repairs may only be done by the manufacturer.
For indoor use only.

Environment:

Batteries are small chemical waste. Throw away broken or used up batteries in a special container or hand them in at a recycling centre.

Technical specifications:

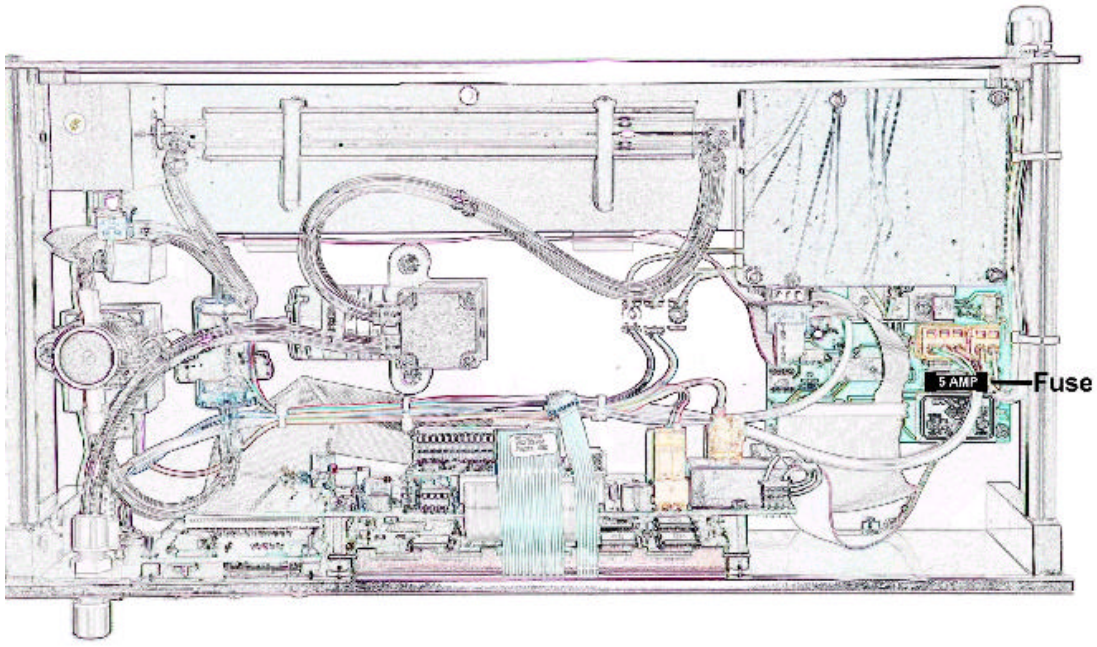
Operation on mains: 230 V~ (+-10%), 50Hz (Euro-version)
120 V~ (+-10%), 60Hz (US-version)
Charging tension: approx. 30V dc at no load, 4-18 Vdc at charge-/discharge function
Charging current: 700 mA +-10%
Discharge current: 200 - 500 mA

3.3 Fuses

The fuse is located on the TRACKER-3000 power board inside the instrument.

C

AUTION: Disconnect instrument from external power



supply before opening the fuse compartment !

Figure: Position of fuse

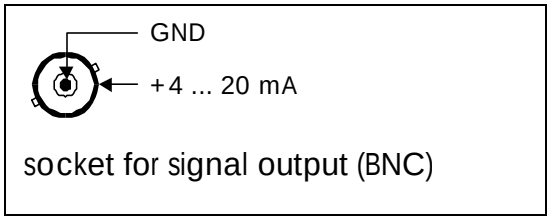
Fuse type: 5 Amps medium slow

3.4 Operation and display components

The TRACKER-3000 features a waterproof IP 65 keypad and a graphic LCD with background illumination for comfortable communication with the operator.

3.5 Connection of the electrical outputs

3.5.1 Analogue signal 4-20 mA



The socket for the analogue output signal is located on thefront panel of the TRACKER-3000. To transmit the signal the included cable

is connected with BNC-plug to the socket marked with "SIGNAL". Maximum load is 400 Ohm.

3.5.2 Serial data output (RS 232)

To transfer the data to a PC a 9-pin DSUB-socket is installed on the rear panel of the TRACKER-3000. The serial input of the PC has to be connected with an appropriate cord (part No. 202-07). All data are transferred as ASCII characters. They can be read by the PC with a terminal program like WIN-Terminal or Hyper Terminal.

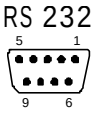
The parameters for data transfer are:

9600 baud / 8 data bit / no parity and Xon / Xoff**.**

The measurement actualizes every second, and every 30 seconds status informations are transferred as following characters:

N= NORMAL**;** **M=** ERROR**; A=** ALARM**; D=** DILUTION ACTIVE

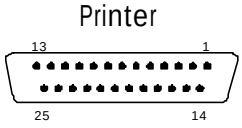
Figure: Serial Data output socket



Pin Nr.	5	3	2
	GND	RxD	TxD

3.5.3 Connection of a printer

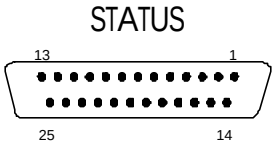
To connect a printer (EPSON-compatible) a 25-pin DSUB-socket is installed on the front panel. If the printer mode is acivated, a periodical printout of mercury concentration and according data like date and time is performed. The time intervals between two printouts can be set in the printer menue.



Pin Nr.	1	2	3	4	5	6	7	8	9	11	18
	PSTROBE	D0	D1	D2	D3	D4	D5	D6	D7	BUSY	DGND

3.5.4 Status output

Operational status and alarm messages are available through potential free relay contacts. The maximum load of the contacts is 80 V / 0.4 A. The contacts are open when active.



Pin Nr.	Status
2+14	Error
3+15	Zeroing
4+16	Alarm

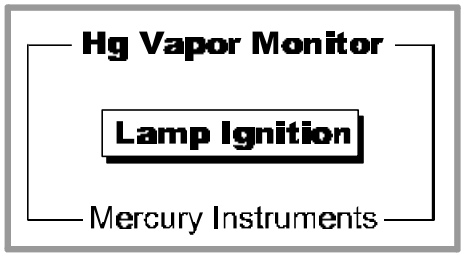
3.5.5 Sample gas connection

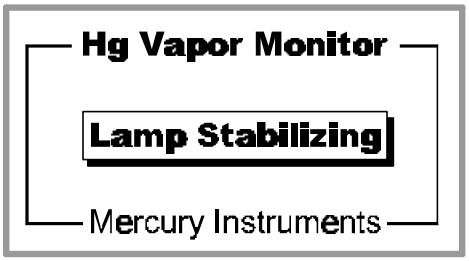
The sample inlet (quick fit) is located at the front panel of the TRACKER-3000. It is possible to analyze ambient air by directly taking it into the sample inlet. The sample gas can also be drawn from distance to the VM 3000 through a tubing which has to be connected to the sample inlet. For the TRACKER-3000 a wand is available which can be also connected to the sample inlet.

4. Operating instructions

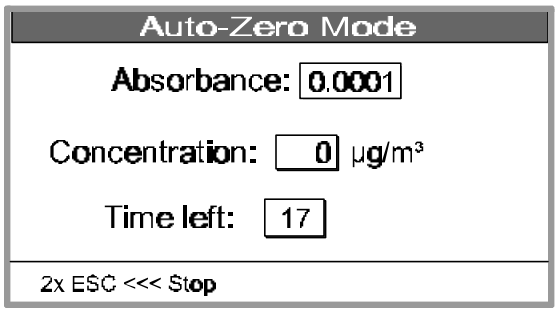
4.1 Start-up measuring

After turning the TRACKER-3000 on the mercury lamp is ignited and warmed up to operation temperature. Following indications appear on the display first:

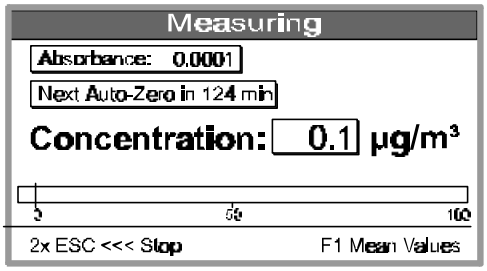




After the instrument has stabilized, which typically takes 3-15 minutes (depending on the ambient temperature), an Auto-Zero is performed:

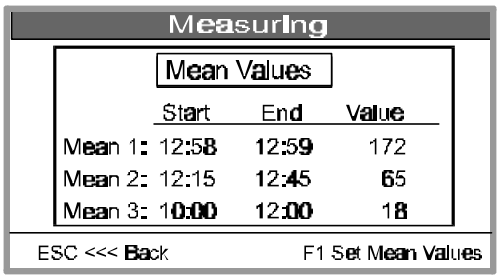


After the auto zero is finished, the VM 3000 automatically starts measuring.



Now on the display are shown:

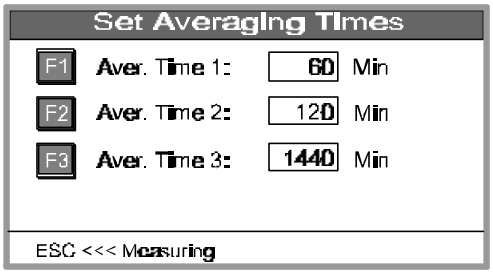
- actual absorption,
- concentration in µg/m³ or in ppb (whatever has been selected),
- time left until the next zeropoint adjustment,
- a bargraph visualizing concentration of mercury:



The measuring mode will stay active until it is interrupted by double pressing the ESC-key.

4.1.1 Display of mean values

If the **F1** key is pressed while measuring, the mean values over preset time intervals are shown. Following display appears:.

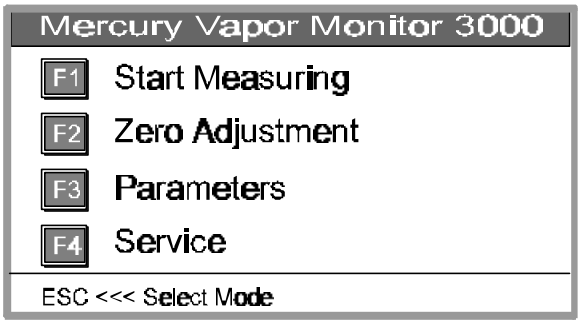


By pressing the **ESC** key the TRACKER-3000 returns to the measuring mode whereas after pressing the F1 key the averaging times can be edited:

After editing you can return to the measuring mode by pressing the ESC key.

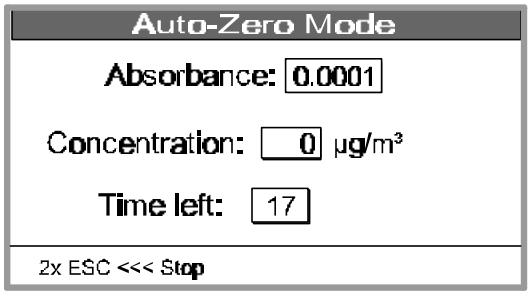
4.2 Selecting the Main Menue

The main menue appears if the ESC-key is double pressed during measurement.



4.3 Manual zeropoint adjustment

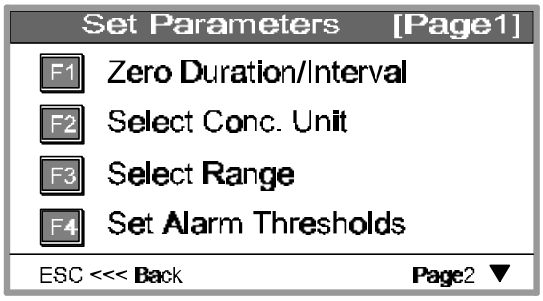
If **F2** is pressed in the main menue (see chapter 4.2) the zeropoint adjustment is started. During zeropoint adjustment an internal pinch valve cuts off the sample flow and filtered air is drawn through the optical cell.



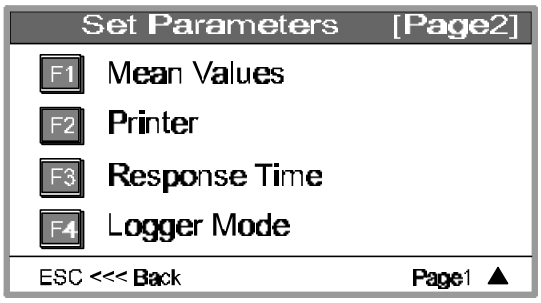
<u>CAUTION:</u> Performing zeropoint adjustment, the mean of measurements during the last second will be set as new baseline. If the zero adjustment procedure is interrupted (double press of ESC) but there is still sample gas in the optical cell the zeropoint adjustment may be incorrect.

4.4 Setting of parameters

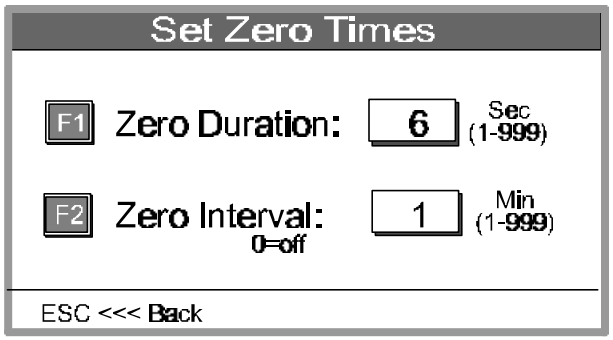
To get to the "Set Parameters" menue, **F2** has to be pressed in the main menue (see chapter 4.2). Following display will appear:



By pressing the arrow- symbol ▼ you can turn to page two of the parameter menue:



4.4.1 Setting of duration and repeat-intervall of the automatic zeropoint adjustment Press **F1** „Zero Duration/Intervall“ on page 1 of the parameter menue:



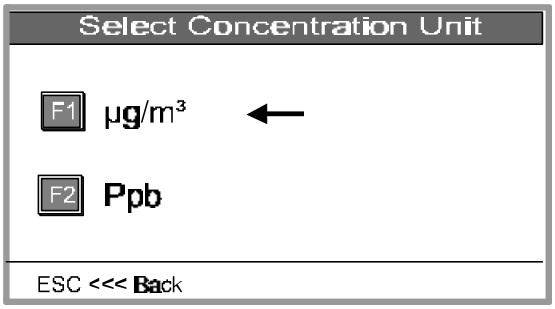
F1 is for setting the duration

F2 is for setting the time interval between two automatic zeropoint adjustments.

☞ **A typical setting for portable use at low measuring range is shown in above figure.**

The automatic zeropoint adjustment is off if "Zero Interval" is set to "0".

4.4.2 Selection of concentration units (µg/m³ / ppb)
Press **F2-** “Select conc. Unit“ on page 1 of the parameter menue:



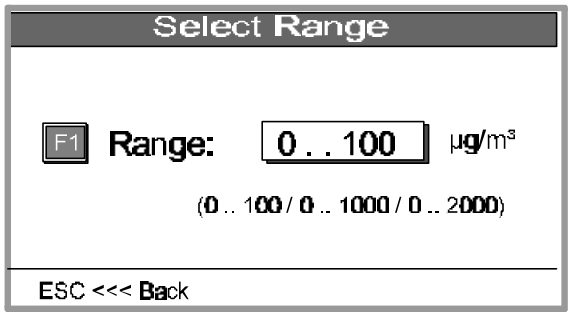
F1: selection of µg/m³

F2: selection of ppb

4.4.3 Selection of measuring range

Press **F3** -"Select Range" on page 1 of the parameter menue.

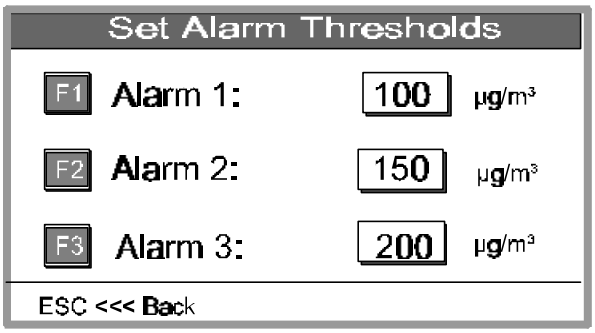
One of three measuring ranges can be selected by pressing **F1**.



4.4.4 Setting of alarm thresholds

Press **F4 Set Alarm Thresholds** on page 1 of the parameter menue:

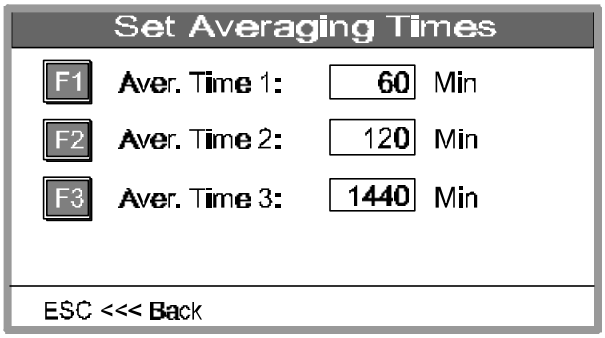
By pressing **F1**/ **F2**/ **F3** up to three alarm thresholds can be set.



4.4.5 Setting of averaging times for mean values calculation

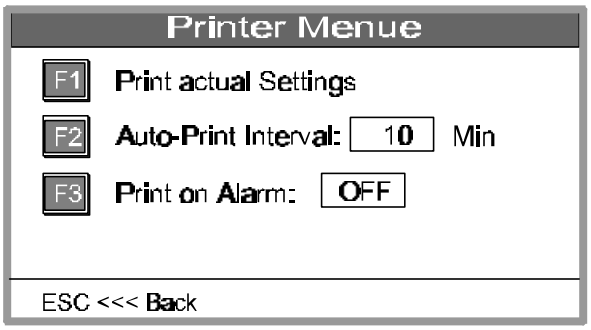
Press **F1 Mean Values** on page 2 of the "Set parameters" menue. While measuring the TRACKER-3000
determines the mean values for the preset time intervals. These can be retrieved during measuring and also printed out by a connected printer.

By pressing **F1/F2/F3** up to three time intervalls can be entered for determination of the mean values.



4.4.6 Printing the actual parameters

The printer menue will appear, if **F2 Enter** on page 2 of the parameter menue is pressed.



With a printer connected, the actual parameters will be printed by pressing the **F1** key.

```
 M-3000   Mercury Vapor Monitor
Parameters
Date :  11.08.97       Time :  11:45:09
Software-Version:  1.0
Zero Duration :    0 min
Zero Interval :   60 sec
Conc. Unit :  ug/m3
Range :  0..100 ug/m3
Alarm 1 :   100 ug/m3
Alarm 2 :     0 ug/m3
Alarm 3 :     0 ug/m3
Aver. Time 1 :     0 min
Aver. Time 2 :     0 min
Aver. Time 3 :     0 min
Auto-Print Interval :   0 min
Print on Alarm :  ON
Response Time :  0 sec
Cal.Factor :  16.07.97     12:53     1.00
```

Figure: Printout of the actual parameters.

4.4.7 Changing printer settings

Select printer menue as described in 4.4.6. The following printer settings will only be completed if a printer is on-line:

- Setting of auto-print interval: Press **F2 Auto-Print Interval**.

The automatic printout of the measurements will be completed periodically in the set time interval. If zero is entered , no print will succeed (printer= OFF).

- Print only in case of alarm: Press **F3 Print on Alarm**. If „ON“ is displayed printing of the measurements will succeed in one-minute-intervals as long as the alarm threshold is exceeded.

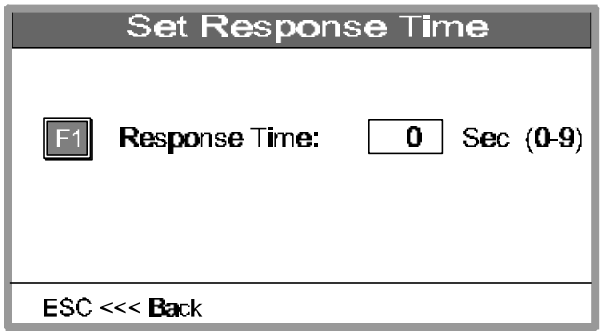
```
VM-3000   Mercury Vapor Monitor
11.08.97,11:56:38,   0,[ug/m3],  - ,[  2],  - ,[ 10],  - ,[  20]
11.08.97,11:57:38,   0,[ug/m3],   0,[  2],  - ,[ 10],  - ,[  20]
11.08.97,11:58:38,   0,[ug/m3],   0,[  2],  - ,[ 10],  - ,[  20]
11.08.97,11:59:38,   0,[ug/m3],   0,[  2],  - ,[ 10],  - ,[  20]
11.08.97,12:00:38,   0,[ug/m3],   0,[  2],  - ,[ 10],  - ,[  20]
11.08.97,12:01:38,   0,[ug/m3],   0,[  2],  - ,[ 10],  - ,[  20]
11.08.97,12:02:38,   2,[ug/m3],   0,[  2],  - ,[ 10],  - ,[  20]
11.08.97,12:03:38,   2,[ug/m3],   1,[  2],  - ,[ 10],  - ,[  20]
11.08.97,12:04:38,   3,[ug/m3],   1,[  2],  - ,[ 10],  - ,[  20]
11.08.97,12:05:38,   2,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:06:38,   2,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:07:38,   2,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:08:38,   2,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:09:38,   2,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:10:38,   1,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:11:38,   1,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:12:38,   1,[ug/m3],   2,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:13:38,   2,[ug/m3],   1,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:14:38,   2,[ug/m3],   1,[  2],   0,[ 10],  - ,[  20]
11.08.97,12:15:38,   2,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:16:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:17:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:18:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:19:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:20:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:21:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:22:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:23:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:24:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:25:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:26:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:27:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:28:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:29:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:30:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:31:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:32:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:33:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
11.08.97,12:34:38,   1,[ug/m3],   1,[  2],   1,[ 10],   1,[  20]
```

Figure: Printout showing date, time, mercury concentration, 2-minutes mean, 10-minutes mean, 20-minutes mean

4.4.8 Setting of response time

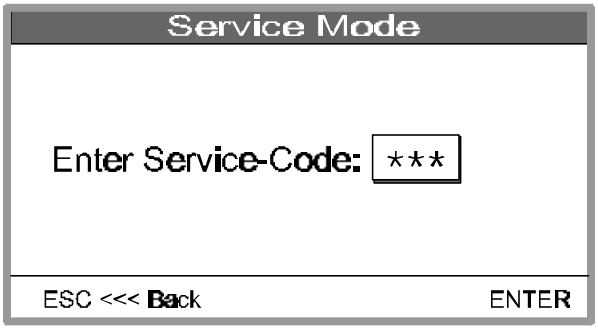
Press **F3**- "Response Time" on page 2 of the parameter menue.

By pressing **F1**- "Response Time" a time constant between 0 and 9 seconds can be entered. The measuring signal will be smoothened by this time factor.

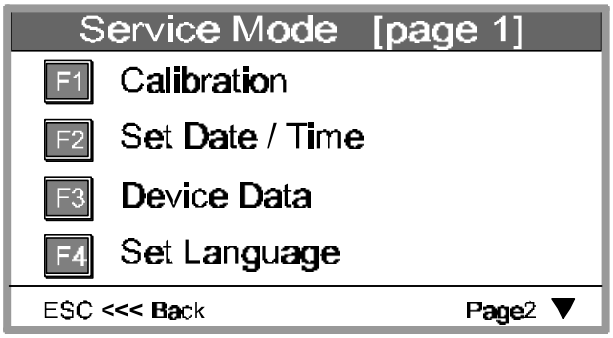


4.5 Service menue

Press **F4**- "Service" in the main menue (see chapter 4.2) and the program jumps into the"Service mode".



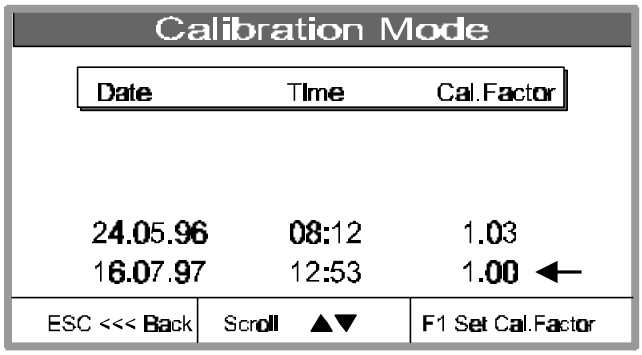
After entering the code **321** [ENT], you have access to the service mode:



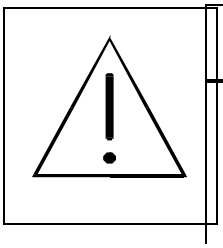
4.5.1 Display of calibration factors list

The actual calibration factor and all previously entered calibration factors can be recalled in form of a list on the display.
After pressing **F1**- "Calibration" in the service mode, following page will appear:

The actual calibration factor is marked by an arrow.



4.5.2 Entering a new calibration factor



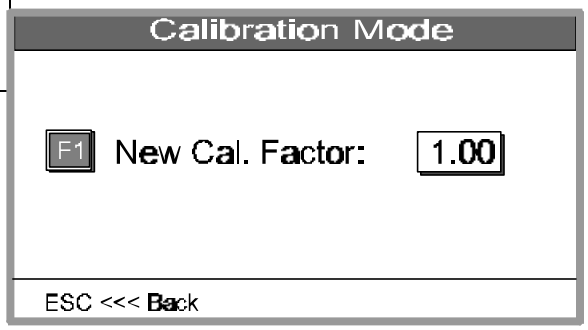
CAUTION:

Every alteration of the calibration factor influences the measurements. Therefore the calibration factor should only be changed after previous determination with a suitable calibration gas source (e.g. Mercury Calibrator MC-3000 by Mercury Instruments).

Each TRACKER-3000 has been calibrated before delivery. Every two years a professional calibration check is recommended. According to regulations applied on a special application more frequent recalibration may be necessary.

ATTENTION: If parts of the TRACKER-3000 in contact with sample gas are contaminated with active substances like sulphur, an immediate calibration check is recommended.

After pressing **F1**- "Calibration" and pressing **F1** again, a new calibration factor can be entered.

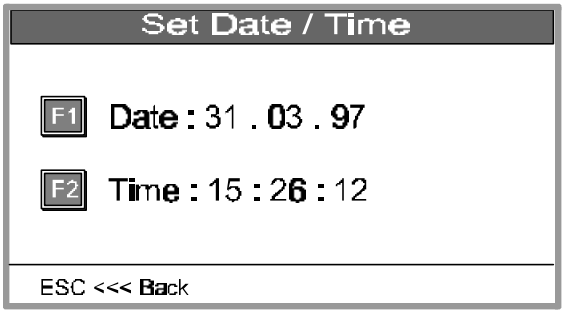


4.5.3 Setting date and time

Press **F2**- "Set Date/Time" in the service menue.

Press **F1** to edit date

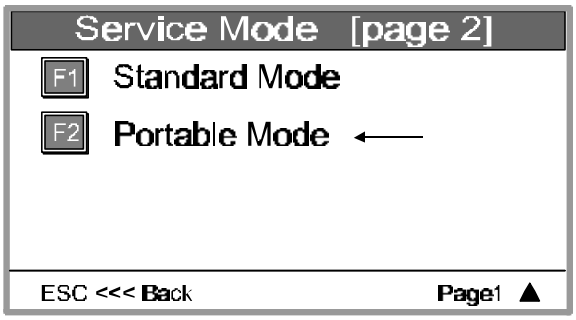
Press **F2** to edit time



4.5.4 Portable Operation

For some applications like indoor screening of mercury contaminations it is useful to select ***Portable Mode***.

- Select ***Service Mode*** (see chapter 4.5)
- Enter Service Code ***3 2 1***
- Go to page 2
- Press ***F2- Portable Mode***
- Press ***ESC*** to leave the Service Mode



If ***Portable Mode*** has been activated, the last measurement is frozen during Auto Zero. The performance of an Auto Zero is indicated on the screen.

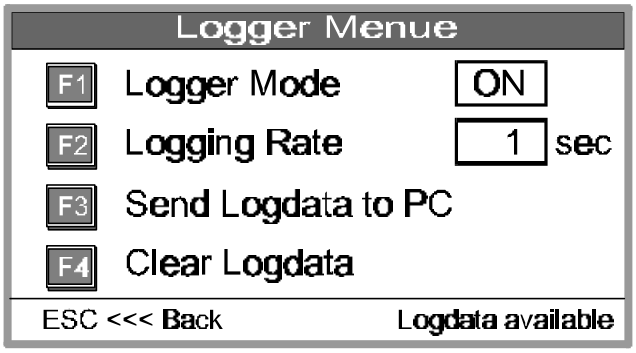
☞ Recommendation: If the instrument is used in Portable Mode, the ***Auto Zero Interval*** should be set to ca. 0.6-1.0 minutes. Thus zero drift which may be caused by temperature changes is compensated.

5. Data Logger Function

The TRACKER-3000 is available with a data logger function (as an option). Up to 15000 measurements can be stored in the RAM of the TRACKER-3000. This enables the user to register up to 4 hours of measuring time if a logging rate of 1 second is set (respectively 40 hours of measuring if a logging rate of 10 seconds is set). The logging function has to be activated after the instrument has been switched on (see 5.2).

5.1. Setting the logging rate

- Jump to the Set Parameters menue (cf. 4.4 of manual).
- Go to page 2 of Set Parameters menue by pressing the▼-key
- Press F4- Logger Mode. The Logger Menue appears.
- Press F2- Logging Rate (1-15 sec)
- Enter the desired logging rate. Values from 1 sec to 15 sec are allowed.



All previously stored data are cleared if the logging rate is changed !

5.2. Activating the data logger

After the TRACKER-3000 has been turned on the instrument automatically performs an Auto Zero and jumps into the measuring mode. For logging of data the data logger has to be activated.

- Go to the Logger Mode menue as described under 5.1.
- Press F1- Logger on/off to activate/deactivate the logger.

> ***The data logger is automatically deactivated after power has been turned off. It is also deactivated if 15000 measurements are stored in the RAM.***

5.3. Start logging of measurement data

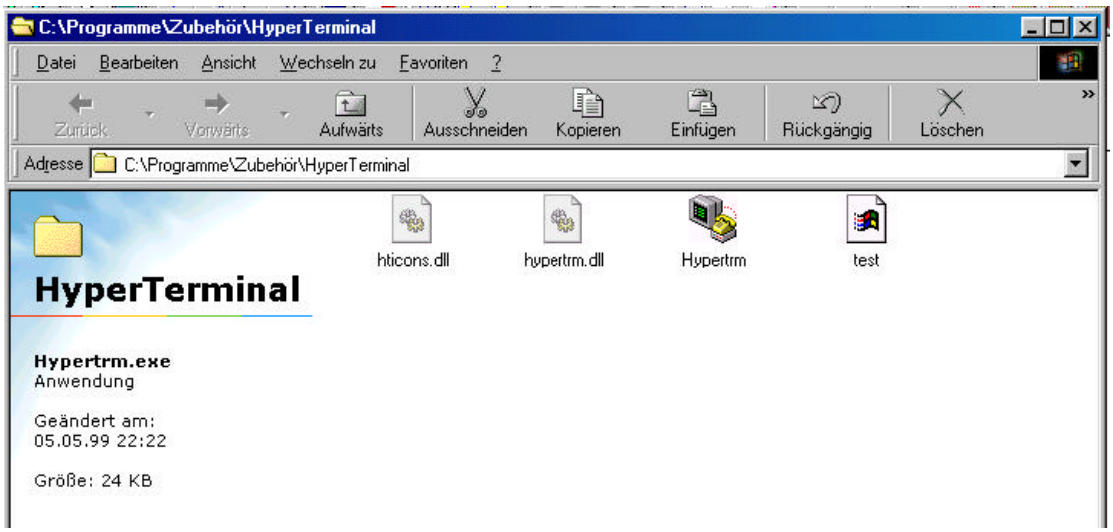
After the data logger has been activated the measurement data are automatically logged whenever the measurement mode is active. The status of the logger is indicated on the display (ON/OFF).

5.4. Sending logged data to a PC

For transferring the stored data to a PC the RS 232 output of the TRACKER-3000 (see 3.4.2) has to be connected with a serial interface (COM1 or COM 2) of the PC.

Select the Logger Mode menue as described under 5.1.
Then following steps have to be performed on the PC (in WINDOWS):

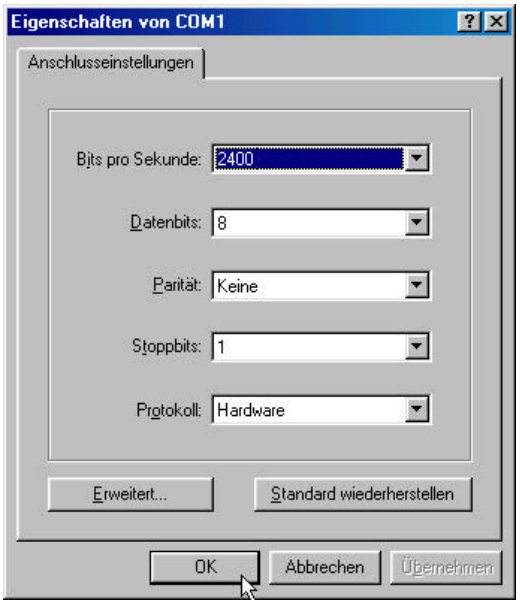
- Select Start ➟ Programs ➟ Accessories ➟ Communication ➟ Hyperterminal.
- Start the program hypertrm.exe



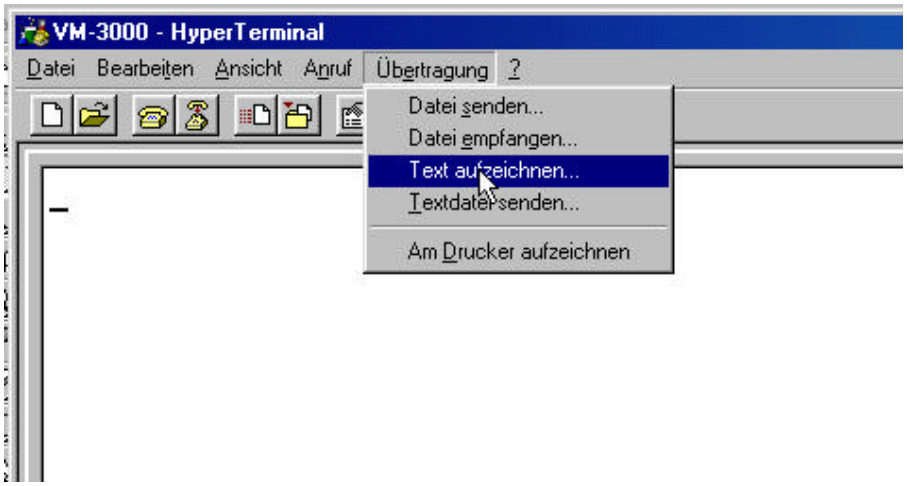
- Enter a name for the connection (for example TRACKER-3000), select a symbol and press ok.

- Select Direct connection COM1 (or CO
- M2 if used) and press ok.

- Settings: 9600 bits per second / 8 data bits / no parity / 1 stopbit / protocol hardware. Press the ok-bar.



- Select recording of text on the transmission pull down menue. Enter file name and press Start.



-
-

- The logged data are now transferred from the TRACKER-3000 to the PC. Before measurement data the starting date, starting time and logging rate used for measurements are transferred. Data are separated by a carriage return.

5.5. Delete logged data

Select the Logger Mode as described under 5.1.

- Press F4- clear Logdata.

> *Now all measurement data stored in the RAM are deleted.*

5.6. Import of data into EXCEL

The data once stored with the HYPERTERMINAL program can easily be imported into an EXCEL data sheet by selecting the data import function in EXCEL.

6. Maintenance

6.1 Particle filter of the wand

Under normal operation conditions, the sample gas particle filter has to be inspected regularly. If a deposit of particulate matter is visible, the filter membrane has to be replaced. Expected time interval for a filter replacement is 1 ... 6 months if normal room air is measured. For filter replacement carefully screw out the front part of the filter holder. Remove sealing ring and filter membrane. Insert spare filter membrane that the slick side faces towards the wand and away from the instrument. Place sealing ring and screw in front part of the filter holder. Make sure everything is gas tight.

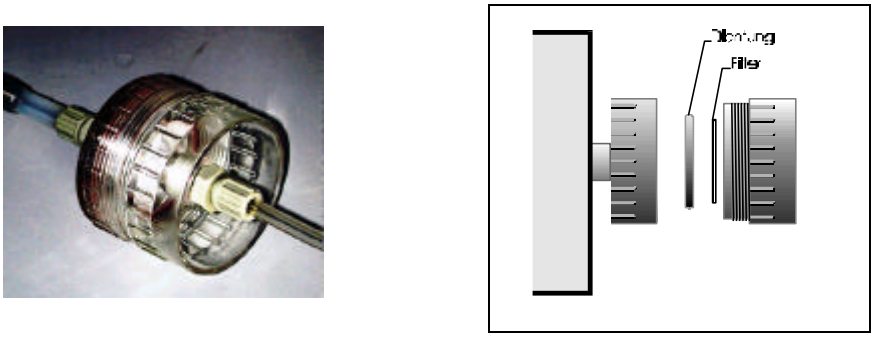


Figure: Particle filter replacement

6.2 Replacement of the carbon cartridge for zero air

The internal carbon cartridge of the TRACKER-3000 should be replaced annually.

Replacement of carbon cartridge:

Place the instrument on a flat surface downside up. Then loosen the four screws on the bottom side of the TRACKER-3000 and open it by carefully pushing the front panel into the housing. The carbon cartridge is now visible and can be removed from its holding clamps. To replace the carbon cartridge the hose has to be pulled off.

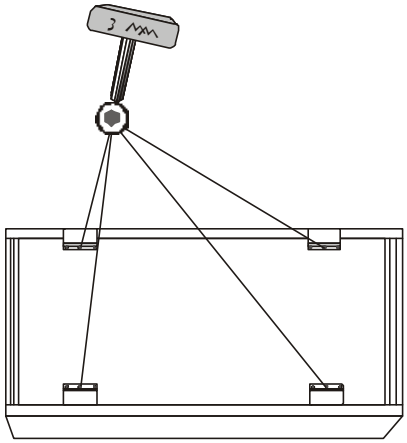


Figure: Opening of the instrument by unscrewing the 4 bottom screws.

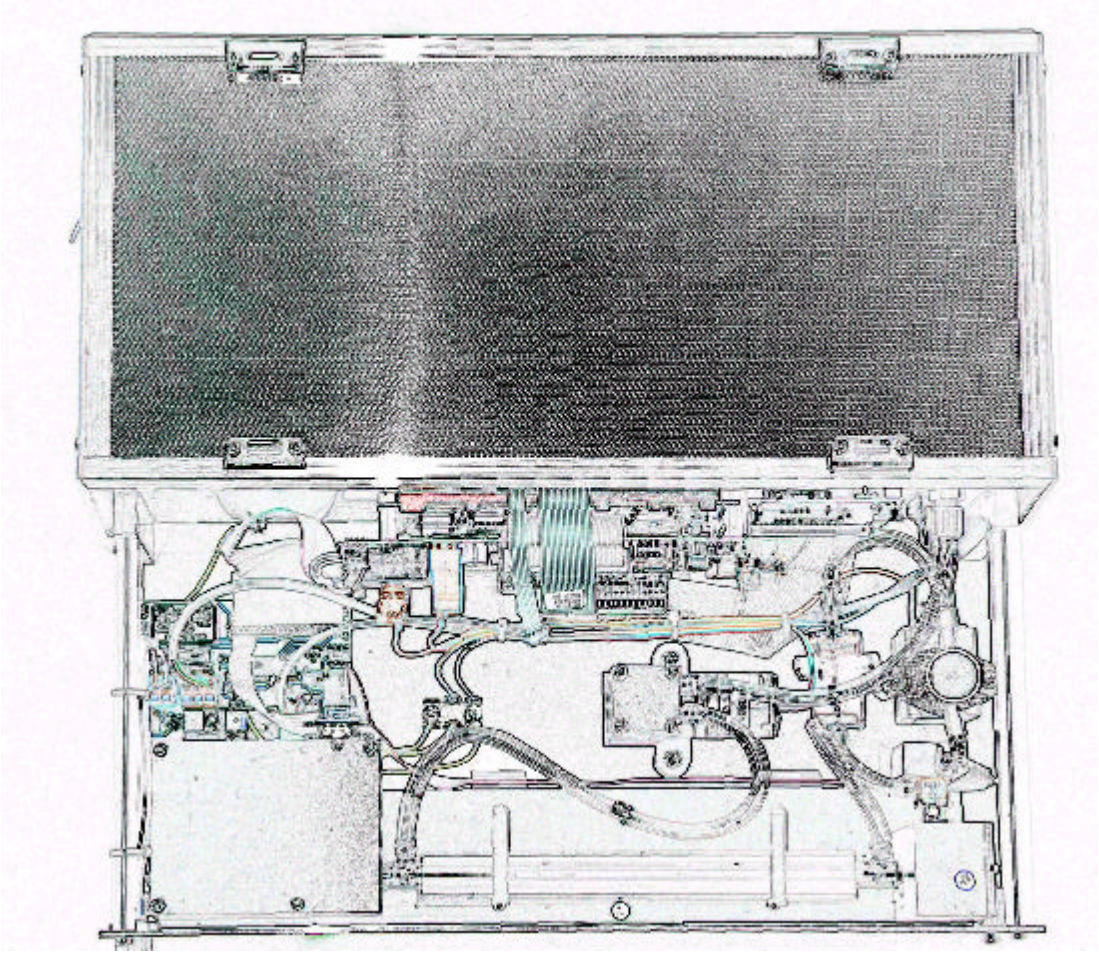


Figure: Opening the instrument

6.3 Care and maintenance

- The TRACKER-3000 should be maintained regularly once a year. Following service work has to be done: Replacement of the pinch valve tubing (part No. 203-12), check of the optical cell and of the ínternal tubing for visible deposits.
- If the TRACKER-3000 is in permanent operation the membrane pump should be replaced every two years.
- Calibration check: Depending on quality control requirements the calibration of the TRACKER-3000 may be checked regularly with a reference screen. Due to the high stability of the photometer anually is recommended. For the performance of the calibration check see chapter 6.4.

6.4 Calibration check with reference screen

This test is for check of the instruments calibration stability. Instead of a calibration gas source a simple reference screen with known absorbance is brought into the photometric light path.

CAUTION:

for this test the instruments top cover has to be opened during the instrument is powered. There is danger of electric shock and physical damage if parts under high voltage are touched. This test may be performed by personnel only who is trained and qualified for work at opened electric equipment.

Following steps have to be followed:

a) bring instrument into a dry room and place it on a dry and stable surface (table).

a) Switch on instrument and allow to warm up for min. 30 minutes.

c) Perform zero adjustment.

d) Open top cover (see chapter 6.2) of the instrument and place test screen into the gap between between UV detector and optical cell. Touch screen only on the labeled side, take care that the screen surface does not get dirty.

e) put the top cover onto the instrument to prevent lightfrom entering into the instruments interior.

f) take reading of the absorbance value on the instruments display on the front panel.

g) switch off power and disconnect from mains.

h) remove test screen and mount top cover again.

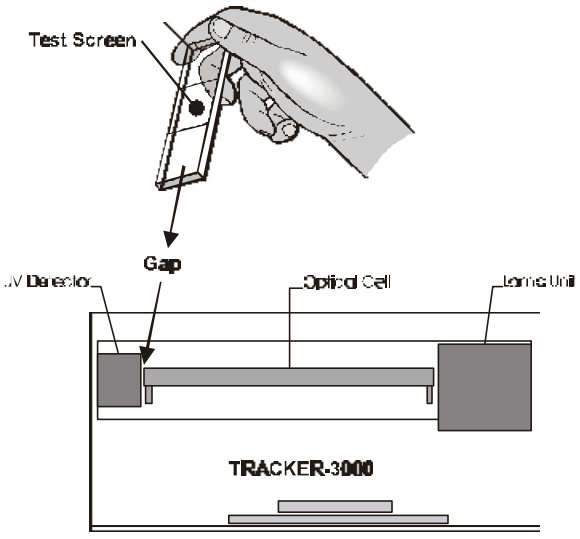


Figure: positioning of screen into gap

The reading of the absorbance value during the screen is positioned in the light path of the photometer should be in the range specified on the label of the test screen. If the reading is out of this range, contact the service for check.

7. Technical information

7.1 Technical specifications

Instrument type	MERCURY TRACKER-3000
Manufacturer	Mercury Instruments Analytical Technologies
Measuring component	Mercury vapor Hg°
Measurement principle	Atom absorption
Wavelength	253,7 nm
UV-source	electrodeless mercury discharge lamp
Method of stabilizing	optically with reference beam; thermally
Optical cell	entirely of fused silica Suprasil, l= 230 mm
Cell temperature	ca. 70 °C, heated
Measuring ranges	0.1 - 100; 0.1 - 1000; 0.1 - 2000 [µg/m³] 0 - 10; 0 - 100; 0 - 200 ppb
Sensitivity	ca. 0.1 [µg/m³]
Sample gas flow	ca. 70 - 90 l/h
Analogue output	Hg concentration in real time; 4 - 20 mA
Binary outputs	error; zeroing; alarm; dilution active
Digital outputs	Serial (RS 232) for PC connection, parallel (Centronics) for printer connection
Electrical power consumption	max. ca. 40 W
Power supply	230 VAC / 50 Hz ; 115 VAC / 60 Hz (option) ; 12 V DC (option)
Dimensions	42.5 cm x 35 cm x 14 cm
Weight	ca. 9 kg

7.2 Influencing effects

Operating temperature	0 °C ... 40 °C
Sample temperature max.	65 °C
Operating humidity	ca. 90 % max. R.H.
Sample gas humidity, max.	no condensing conditions
CE approval	according to 89/336/EEC and 73/23/EEC

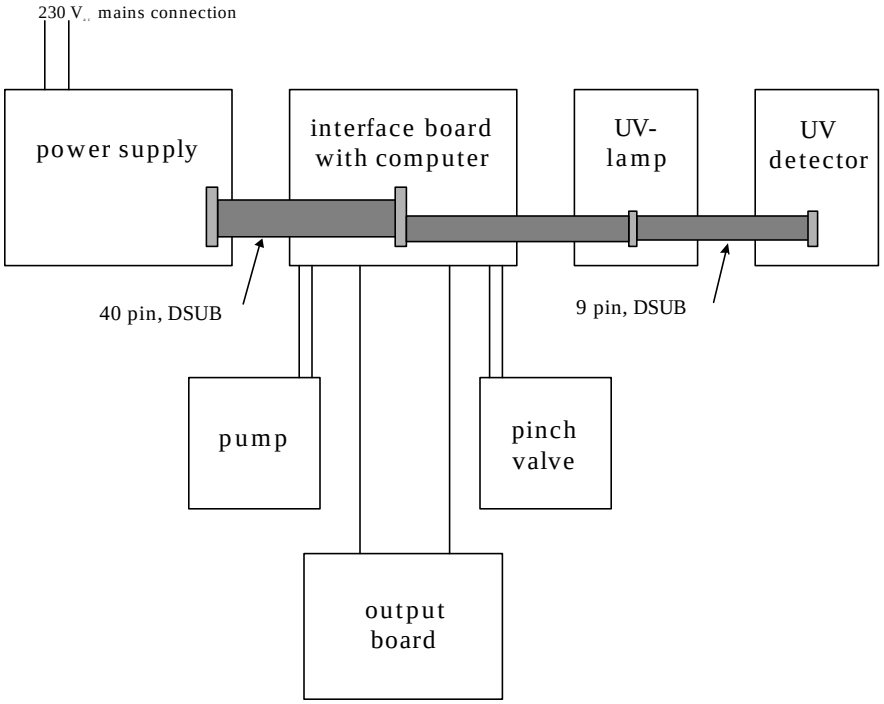
7.3 Storage and transport

The interior of the VM- 3000 should be kept save from moisture and hard shocks. Vibrations caused by transport in a car do not harm the TRACKER-3000.

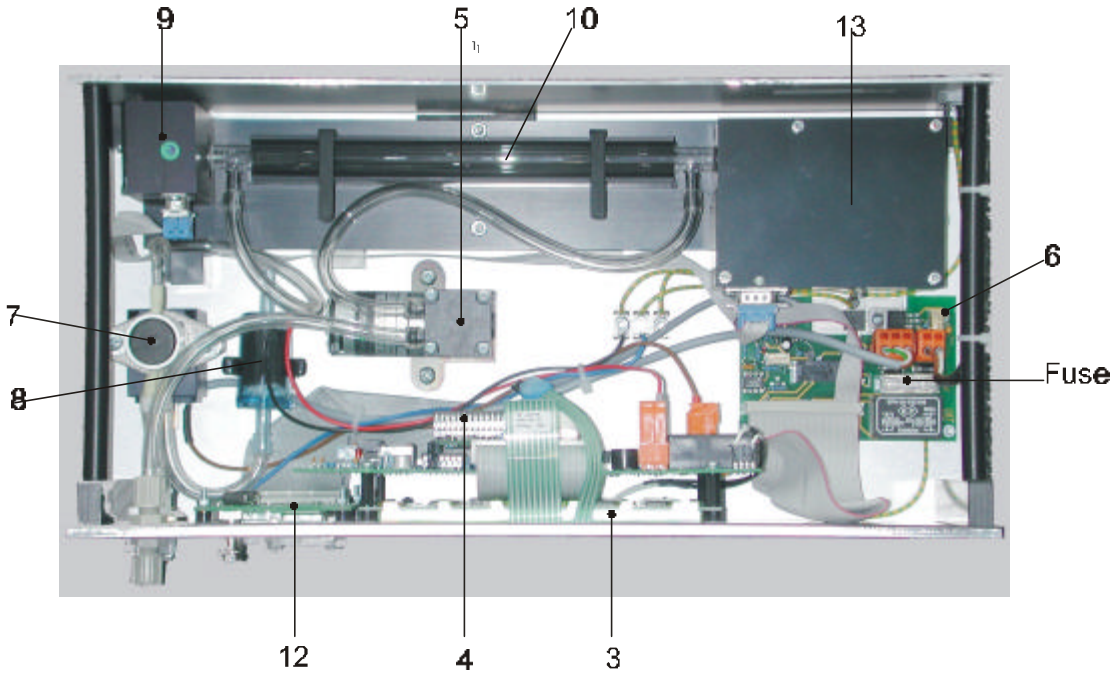
7.4 Spare parts and accessories

Ref.No.	Description
320-01T	Wand for field testing, with mounted filter holder and 3 m tubing
330-01 T	Quick-Fit connector, for connection of sample tubing or wand to sample gas inlet of TRACKER
330-02 T	Sample in Quick-Fit connector, mounted on front panel of TRACKER
201-03	Sample tubing 4 mm (i.d.) x 6 mm (o.d.); material Tygon
202-05	Filter membrane, PTFE, for use with 320-01T (wand)
201-04	Carbon filter, filled with sulphurized carbon, Zero filter
202-08	Printer connection cable
202-07	PC connection cable
202-09	Plug for status outputs
300-01	Rechargeable battery pack, 12 V, for ca. 6 h operation
202-11	charger for batteries with IC-controlled charge/discharge function,

7.5 Block diagram of electrical parts



7.6 Diagram of components



No. in figure	MI Part #	Description
3	203-51T	LC display board
4	203-52T	Microprocessor board with EPROM
5	203-01	Membrane pump
6	203-53T	Power supply board
7	203-03	Pinch valve
8	201-04	Activated carbon cartridge for zero air
9	203-54	UV detector
10	203-02	Optical cell
13	203-57	UV lamp unit with electrodeless discharge lamp and reference detector

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ATTACHMENT E-4

NITON XLP 3000 SERIES ANALYZER USER'S GUIDE

NITON XLp 300 SERIES
ANALYZER

User's Guide

Version 5.2.1 P/N 500-926

Thermo
SCIENTIFIC

Contents

Chapter 0 About This User's Guide

WARNING! Do not attempt to use this analyzer without first reading and understanding the entire User's Guide! ♦

Unpacking and Assembling Your Niton XLp XRF Analyzer

- Inspect the shipping carton for signs of damage such as crushed or water damaged packaging. Immediately notify the shipping company and Thermo Scientific, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, if there is any visible damage to the shipping container or any of its contents.
- Open the packing carton. If your analyzer is not packed in its carrying case, please call Thermo Scientific immediately, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460.
- Verify the contents of the shipping container against the enclosed packing list. If there are any discrepancies between the actual contents of the shipping container and the enclosed packing list, please notify Thermo Scientific immediately, in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460.
- Open the carrying case and visually inspect the analyzer for damage before removing it from the case. Call the shipper and Thermo Scientific if you find any damage to the case or its contents.
- Save the shipping carton and all packing materials. Store them in a safe, dry area for reuse the next time that you ship the analyzer.

The Niton XLp XRF Analyzer Overview

The Niton XLp Analyzer is a single unit, hand held, high performance portable x-ray fluorescence (XRF) elemental analyzer.

Figure 0-1. Analyzer Overview

The Control Panel

The control panel is located on the analyzer's top housing, directly below the LCD touch screen (see Figure 0-1). The control panel consists of a 4 way touch pad and two control buttons, one on each side. Using either the control panel or the touch screen (with or without the Niton standard touch screen stylus accessory that clips in the XLp battery pack) you may navigate through all of the analyzer's screens and menus. You can control the movement of the screen cursor by pressing the four way control pad in one of four directions to highlight each of the menu options. The enter button on the right side of the four way touch pad is used to select highlighted menu options. The on/off/escape button both controls the power to the analyzer and serves as an "escape" button. When the on/off/escape button is pushed and immediately released, it functions as an "escape", and brings you back to the Main Menu from the current screen in the menu system.

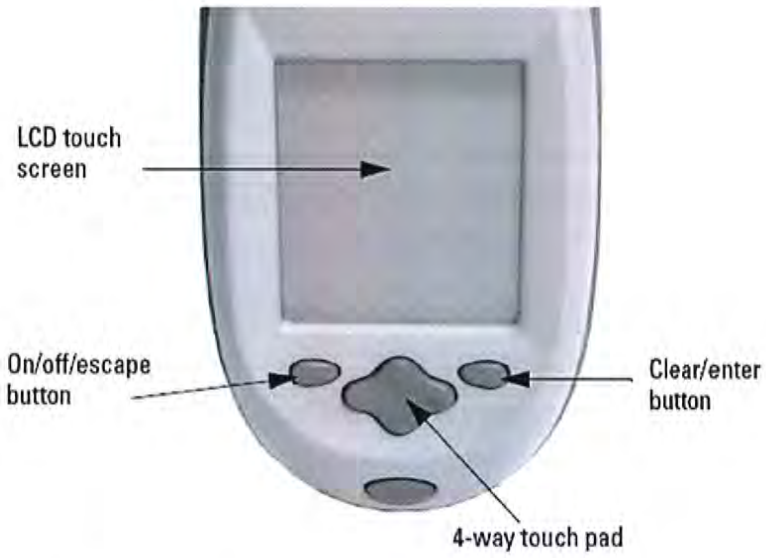


Figure 0-2. The Control Panel

Push and hold the **on/off/escape** button for at least 3 seconds to turn the analyzer on. A 'beep' will sound as the power comes on. Push the **on/off/escape** button and hold it down for about 10 seconds to shut off power to the analyzer from any screen in the menu system. The analyzer will 'beep' as it shuts down.

You also have the option of operating the analyzer, including navigating the menu system, by using the built in touch screen with or without the stylus. To select a menu option with the stylus, tap on the icon once. The touch screen icons have the same functionality as the four way touch pad, the **on/off/escape** button, and the **enter** button. This User's Guide will refer to the process of choosing a course of action by selecting an icon from a menu, either using the touch screen or using the control panel buttons, as "selecting."

Selecting the **Return** icon works everywhere throughout the User Interface to bring you back to the previous menu from the current menu in the menu system. Use the on/off/escape button to return to the **Main Menu**.

Instrument Startup

To turn on the analyzer, depress the **on/off/escape** button on the control panel for approximately 3 seconds, until you hear a beep.

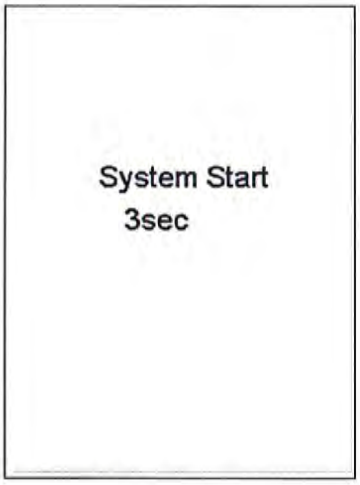


Figure 0-3. System Start Screen

On startup, the screen will be replaced by a **Restart** screen (see Figure 0-3) which will automatically count down from 9 to 0 in increments of one second.

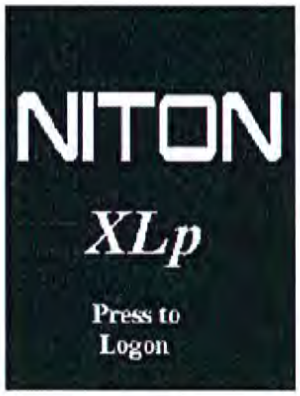


Figure 0-4. Logon Screen

When the **Restart** is complete, the **Restart Screen** will be replaced by the **Logon** screen (see Figure 0-4.) Tap anywhere on this screen to continue.

The **Logon Screen** will be replaced by a **Warning Screen**, advising you that this analyzer produces radiation when the lights are flashing. You must acknowledge this warning by selecting the "Yes" button before logging on. Selecting the "No" button will return you to the **Logon Screen**.

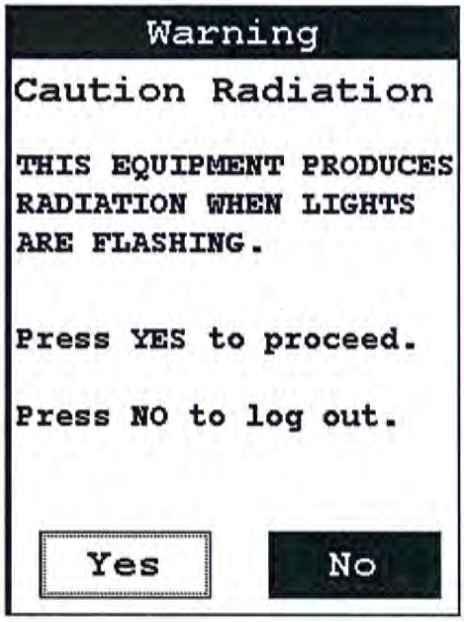


Figure 0-5. Warning Screen

After selecting the "Yes" button, the **Virtual Numeric Keypad** becomes available for you to log onto the analyzer.

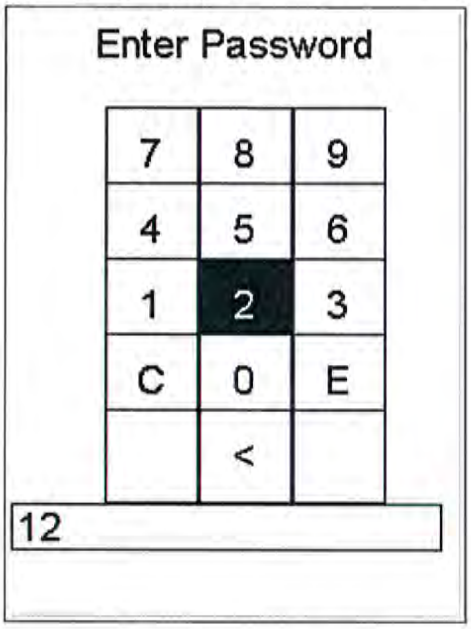


Figure 0-6. Virtual Numeric Keypad for Logon

Select your 4 digit security code, followed by the enter (E) key. The temporary password assigned by default is 1-2-3-4, followed by the "E" key. After you have completed the log on procedure, the word "USER" will appear on the bottom of the screen, then the **Main Menu** will appear. Note that security codes are editable. Please see the NDT manual for instructions on creating user-definable passwords.

Check the **date/time**. The time should be set correctly for accurate and verifiable record keeping (see **Set Time and Date** on page 2-9).

Your analyzer can be stored safely in temperatures of up to 122°F (50°C) and is designed to operate at temperatures of up to 122°F (50°C). You will not be able to take a measurement if the analyzer overheats. If it is hot to the touch, you should allow it to cool before testing.

Screen Contrast

To adjust the contrast of your analyzer's touch screen display, press and hold the left and right ends of the 4 way Touch Pad until a beep sounds. After the beep, you may press the up portion of the 4 way Touch Pad to darken the screen, and the down portion to lighten the screen. When you have the display adjusted so that it is best for you, press the "Enter" button to save the current setting.

Chapter 1 Radiation Safety

CAUTION NITON Analyzers are not intrinsically safe analyzers in regard to sparking. All pertinent Hot Work procedures should be followed in areas of concern. ♦

The NITON XLp Analyzer was designed so that virtually no measurable radiation external to any part of the instrument can escape when the shutter is closed. When the instrument is used in accordance with it's instructions, it is completely safe, that is, there is minimal radiation exposure, even with the shutter open.

NITON XLp Analyzers contain sealed ^{109}Cd and/or ^{55}Fe and/or ^{241}Am radioactive isotope sources. The source is designed to remain secure even under extreme conditions, so that even if the instrument is broken, crushed or burned, there should be no leakage of radioactive material.

During manufacturing, each sealed isotope source is locked in place in a solid tungsten alloy source holder. The source is completely secure in its housing because the aperture at the closure end of the housing is smaller than the source and is completely sealed. The source assembly is secured in the instrument's case, which is fitted with tamper-proof screws.

Human exposure to radiation is typically measured in REMs, or in one-thousandths of a REM, called milliREMs (mREM). For a given source of radiation, three factors will determine the radiation dosage you receive from the source:

1. **Duration of Exposure**

   The longer you are exposed to a source of radiation the more radiation strikes your body and the greater the dose you receive. Dosage increases in direct proportion to length of exposure.

2. **Distance from The Source**

   The closer you are to a source of radiation, the more radiation strikes you. The dosage increases in inverse-squared relation to your distance from the source of radiation. For example, the radiation dose one inch from a source is nine times greater than the dose three inches from the

source, and 144 times greater than the dose one foot (12 inches) from the source. For another example, the radiation dose one meter from the source of radiation is 100 times lower than the dose at 10 cm from the source of radiation. Keep your hand and all body parts away from the front end of the analyzer when the shutter is open to minimize your exposure.

3. Shielding

Note Your instrument emits virtually no radiation with the shutter closed because the ^{109}Cd and/or ^{55}Fe and/or ^{241}Am sources are thoroughly shielded in every direction. This shielding absorbs nearly all of the radiation produced by the source – except when the shutter is open during testing. With the shutter open, the instrument emits a maximum directed radiation beam of approximately 315 mREM/hr intensity. Always hold your instrument so that the radiation beam is not aimed at yourself or at anyone else. Supplied or optional test stands add shielding for analysis.Wearing a dosimeter badge does not protect you against radiation exposure. A dosimeter badge measures your exposure. ♦

Note Pregnant workers may want to take special precautions to reduce their exposure to radiation. Qualified scientists have recommended that the radiation dose to pregnant women should not exceed a total of 500 mREM/gestation period. See U.S. NRC Regulatory Guide 8.13 "Instruction Concerning Prenatal Radiation Exposure". ♦

Table 1-1 lists typical radiation doses encountered in daily life and lists the annual occupational radiation dosage limits for adults set forth in 105CMR 120.200.

Table 1-1. Typical Radiation Dosages (NCRP, 1987)

Category	Dose in mREMs	Dose in mSv
Average total dose in US (annual)	360 mREM	(3.6 mSv)
Average worker exposure (annual)	210 mREM	(2.1 mSv)
Average exposure for underground miner (annual)	400 mREM	(4.0 mSv)
Exposure for airline crew (1,000 hours at 35,000 ft)	500 mREM	(5.0 mSv)
Additional from living in Denver at 5300' (annual)	25 mREM	(0.25 mSv)
Additional from 4 pCi/l radon in home (annual)	1,000 mREM	(10.0 mSv)
Typical chest x-ray	6 mREM	(0.06 mSv)

Table 1-1. Typical Radiation Dosages (NCRP, 1987)

Category	Dose in mREMs	Dose in mSv
Typical head or neck x-ray	20 mREM	(0.2 mSv)
Typical pelvis/hip x-ray	65 mREM	(0.65 mSv)
Typical lumbar spine x-ray	30 mREM	(1.3 mSv)
Typical upper G.I. x-ray	245 mREM	(2.45 mSv)
Typical barium enema x-ray	405 mREM	(4.05 mSv)
Typical CAT scan	110 mREM	(1.10 mSv)
Minimum detectable dose on a standard film badge	5 mREM/QTR	(0.05 mSv)
Annual occupational dosage limits:		
Maximum allowable for the general public (annual)	100 mREM	(1.0 mSv)
Annual Occupational Dose Limits for Adults:		
Whole body	5,000 mREM/yr	(50 mSv)
For a pregnant worker (during gestation period)	500 mREM	(5.0 mSv)
For a minor	500 mREM	(5.0 mSv)
Eye dose equivalent	15,000 mREM	(150 mSv)
Shallow dose equivalent to the skin or any extremity or organ	50,000 mREM	(500 mSv)

As noted above, the allowable limit in the U.S. for occupational exposure is 5,000 mREM/year (50 mSv/year) for a whole-body and 50,000 mREM (500 mSv) for shallow penetration of extremities. Extremity exposure from a properly used NITON XRF analyzer will be less than 100 mREM per year, (1.0 mSv per year) even if the analyzer is used as much as 2,000 hours per year, with the shutter open continuously.

Note NCRP is the National Council on Radiation Protection and Measurements. ♦

How to Use Your NITON XLp

The NITON XLp was designed to be as safe as possible. However, we strongly recommend that you follow these precautions to insure your safety and the safety of those around you.

WARNING! Always be aware of the location of your instrument's radioactive sources and the direction of their beam of X-rays. The location of the sources is at the front end of the instrument. ♦

Note Open the shutter only to analyze a sample. The shutter can only be opened after the user has logged on to the analyzer using the password. ♦

During testing, a strong beam of radiation is continuously emitted through the kapton window at the front of the analyzer (see Figure 1-1). There will be some radiation at the front and top-front of the analyzer. There is negligible radiation at the handle of the analyzer.

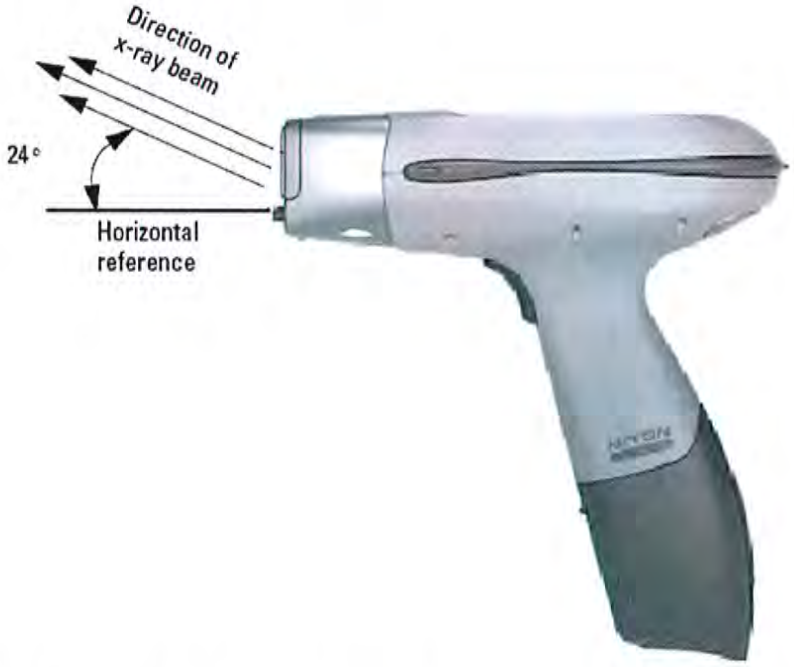


Figure 1-1. Direction of x-ray beams.

WARNING! Always treat radiation with respect. Do not hold your analyzer near the Kapton Measurement Window during testing. Never point your analyzer at yourself or anyone else when the shutter is open. ♦

SHUTTER SAFETY

Note Under no circumstances should the shutters be open when the instrument is not in use!

The 3 warning lights on your instrument will go on when one of the shutters is open and stay on as long as one of the shutters remains open.

WARNING! In the unlikely event that the shutter becomes stuck in the open position, remove the battery (see Battery Pack and Battery Charger - Routine Maintenance Guidelines - Note: All shutters should close immediately and remain locked in the closed position when the battery pack is not attached to the instrument), replace the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department at (800) 875-1578 or +1-978-670-7460. ♦

WARNING! If your LCD Touch Screen displays the message "SHUTTER DOES NOT OPERATE", remove the battery (see Battery Pack and Battery Charger - Routine Maintenance Guidelines - Note: All shutters should close immediately and remain locked in the closed position when the battery pack is not attached to the instrument), replace the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department at (800) 875-1578 or +1-978-670-7460. ♦

Monitoring your Radiation Exposure

There is virtually no measurable radiation from a NITON XLp analyzer when its shutters are closed. The maximum dosage to which you are exposed when properly operating your NITON XLp is <0.1 mREM/hr on the fingers of the hand holding the instrument, with the shutters open.

As an additional precaution to ensure that your radiation exposure is always minimal, NITON recommends that you determine and follow your state or country's specific regulations concerning radiation monitoring. A dosimeter badge is usually worn close to the parts of your body that are most sensitive to radiation, including your reproductive organs and your eyes. These badges are available from many companies. Two companies offering dosimeters in the USA are:

Landauer, Inc.

2 Science Road

Glenwood, IL 60425-9979.

Proxtronics

5795-B Burke Centre Highway

Burke, VA 22015

These companies are listed for information purposes only. NITON does not endorse the services of these companies.

NITON recommends you ask your radiation dosimeter badge supplier for a quarterly schedule. Monthly schedules are also available.

Leak Tests

The radioisotope source shielding on your NITON XLp analyzer is designed to hold up even under extreme conditions, including the instrument being crushed or burned. The continued effectiveness of the instrument's radiation shielding should be tested every six months by performing a thorough leak test.

In the USA, this is a regulatory requirement: Thermo Scientific's license requires that leak tests be repeated once every six months. Leak test kits, which include complete instructions for performing the test, are available

from several vendors. These vendors typically remind you when it is time to perform the next leak test on your instrument. Please follow the test kit instructions carefully, and promptly mail the test samples to the laboratory.

The diagram in Figure 1-2 shows the test wipe locations.

All source shutters must be closed while performing a leak test!

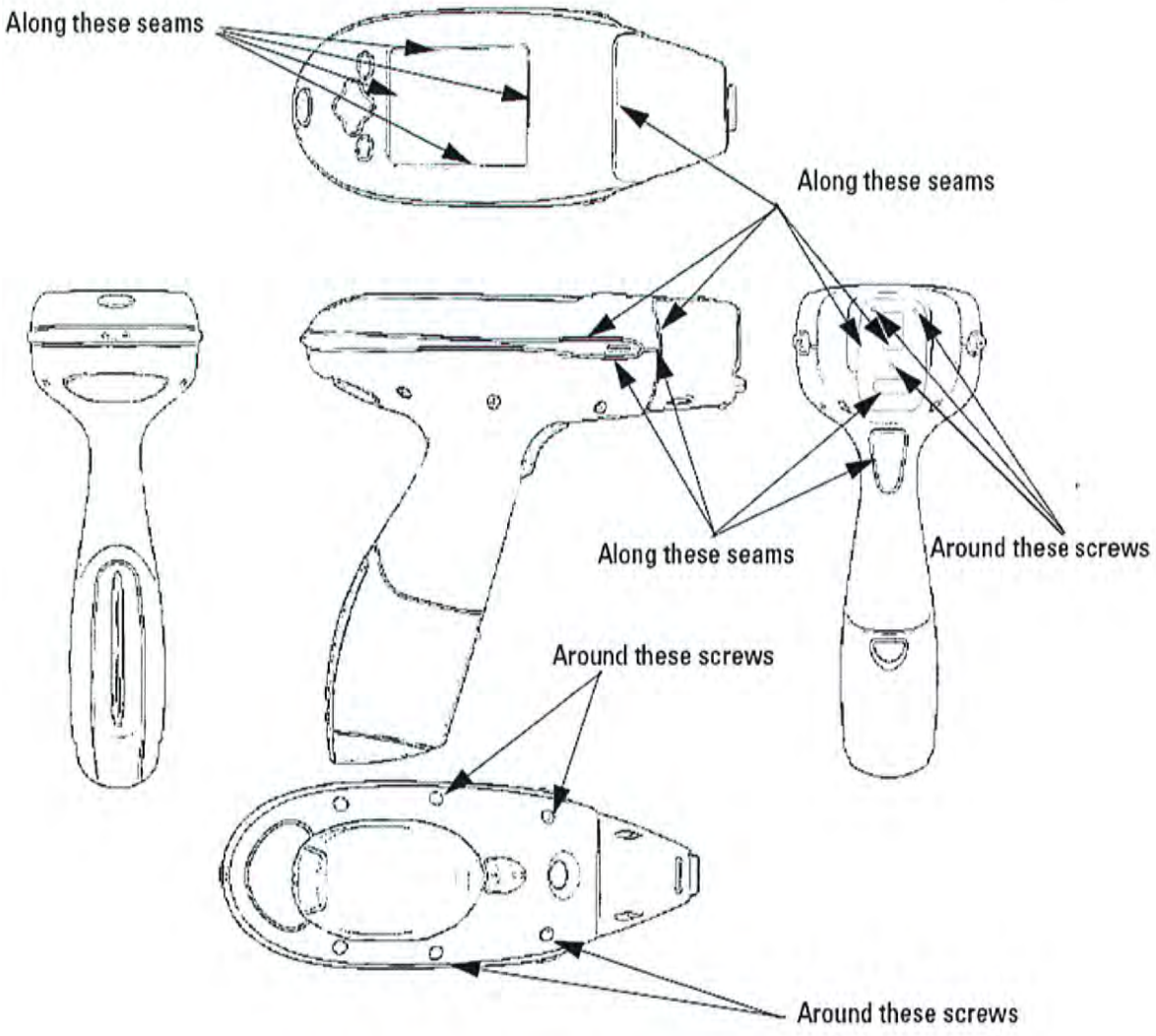


Figure 1-2. Locations of seams for leak testing

The following laboratories supply leak test kits:

Applied Health Physics

2986 Industrial Blvd.

Bethel Park, PA 15102

Tel: (412) 835-9555

Stan A. Huber Consultants

200 N. Cedar Road

New Lennox, IL 60451

Tel: (800) 383-0468

Valley Safety Services

330 Old Enfield Road

Belchertown, MA 01007

Tel: (413) 323-9571

Please check with your local NITON representative if you are located outside the USA

Procedures for Analyzer Loss or Damage

IN THE USA

If your analyzer is damaged, destroyed, lost, or stolen, call:

The Office of Radiological Safety for your state's Dept. of Health.

Telephone:_____

OUTSIDE THE USA

If your analyzer is damaged, destroyed, lost, or stolen, call:

The proper authorities for your location.

Telephone:_____

Also, notify the NITON Analyzer service department using one of the numbers below:

During Regular Business Hours

Calling from within the USA - (800) 875-1578 (toll-free)

Calling from outside the USA - +1-978-670-7460

During Evenings and Weekends, From Anywhere

CALL ONLY WHEN YOU HAVE A RADIOLOGICAL EMERGENCY

- Ken Martin - Mobile Phone Number - +1 (617) 901-3125
- Jim Blute - Mobile Phone Number - +1 (978) 790-8269

If your analyzer is lost, stolen, or damaged in a car accident, also, immediately notify your State Police Department:

Telephone: _____

If your instrument is damaged in a fire or an explosion, also immediately, notify your local Fire Department.

Telephone:_____

Note Please fill in the phone numbers on these pages today. Keep copies where you can find them in case of an emergency. ♦

XLp Radiation Dose Profile - Wood Substrate

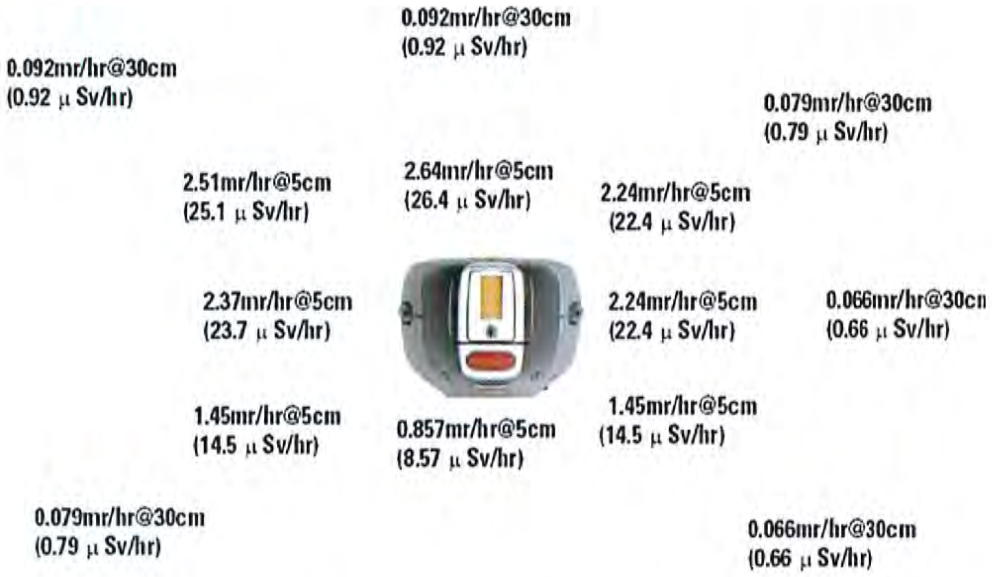


Figure 1-3. XLp 300 series radiation profile

Table 2: XLp 300A/700A Series In-Beam Dose Rates - Shutter Open

Isotope	Activity	5 cm	30cm	100 cm	Trigger
Cd-109	50	369	14.8	1.71	0.053

Note Not to scale. ♦

Note Profile conducted by: James Blute, CHP, Corporate RSO, 04/05/04. ♦

Note Substrate 3/4" thick pine. ♦

Note Profile was taken with the strongest source and the least dense substrate to show the worst case scenario. Any other combination of source strength and substrate would result in lower dose rates from scatter radiation. ♦

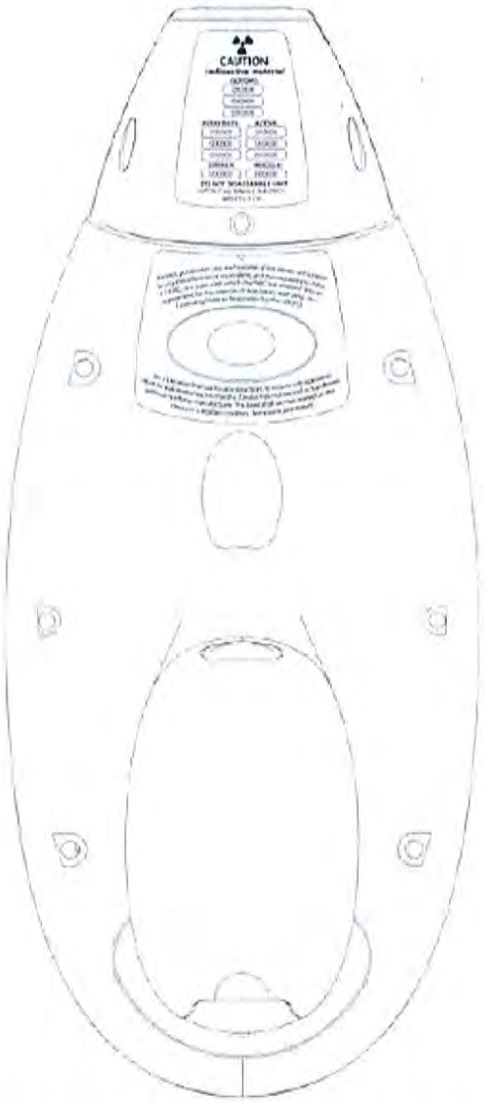


Figure 1-4. View of the Underside of the XLp Analyzer Showing Proper Placement of General and State Specific Radiation Labels

Chapter 2 The Menu System

The Niton XLp Menu System enables you to perform critical tasks, including taking readings and viewing data, with a minimum number of steps. Menus are presented as small pictures called icons which, when selected, will do one of three things:

- Toggle between two different functions or views, such as turning backlighting on or off.
- Present a subsidiary or sub-menu which will allow you access to more choices.
- Present a screen which allows you to view data, change settings, or control the analyzer.

Icons which appear on your Niton analyzer screen display in dark gray (grayed out) represent features which are not enabled, and therefore cannot be selected. In order to enable features on your Niton analyzer which are currently not enabled, please contact Thermo Scientific in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, or your local Authorized Niton Analyzer Service Center.

Icons which appear on your Niton analyzer screen display with a diagonal line through them represent features which are currently turned off. When an icon with a diagonal line through it is selected, the diagonal line through that icon will automatically be erased, and the feature corresponding to that icon will become enabled.

The NAV Menu

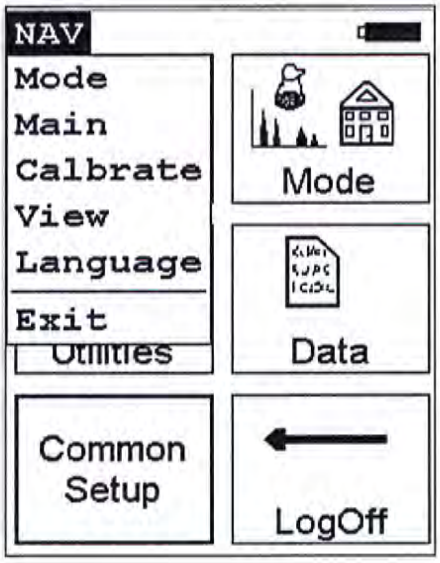


Figure 2-1. The NAV Menu

The Navigation Menu, or NAV Menu, is available in all screens, though only through the touch screen interface. Within a menu, the particular options available from the NAV Menu may change with the context. For example, within the **View Menu**, the **NAV Menu** changes options depending on the mode you are currently using.

Access the **NAV Menu** by selecting the word **NAV** in the screen. A drop-down menu of choices will appear. Selecting an option from the **NAV Menu** will take you directly to a particular menu, no matter where you are in the menu hierarchy. Selecting the "**View**" option from the **NAV Menu**, for example, will bring you directly to the Data Menu.

Selecting the **"Language"** option will load the **Language Screen,** allowing you to change the language from the default English to French, Spanish, Portuguese, or German.

Figure 2-2. The Language Setting Screen

Select the language you want from the Nav Menu.

Figure 2-3. The Main Menu in Spanish

The **NAV Menu** cannot be selected through the Control Panel.

The Battery Life Indicator

The Battery Life Indicator is visible on all screens in the menu system. The indicator is visible in the top right portion of the screen, and graphically shows you how much battery life is left, enabling you to change batteries as needed to avoid unexpected shutdowns.

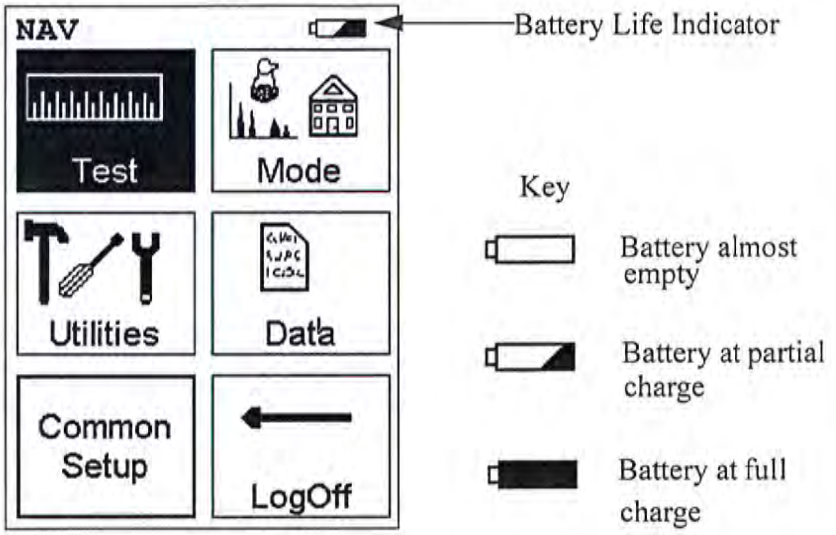


Figure 2-4. Battery Life Indicator

The more black visible in the indicator, the higher the charge. The more white visible in the indicator, the lower the charge. It's best to charge one battery while using the other, to avoid work slowdowns or stoppages due to battery charge conditions.

The Main Menu

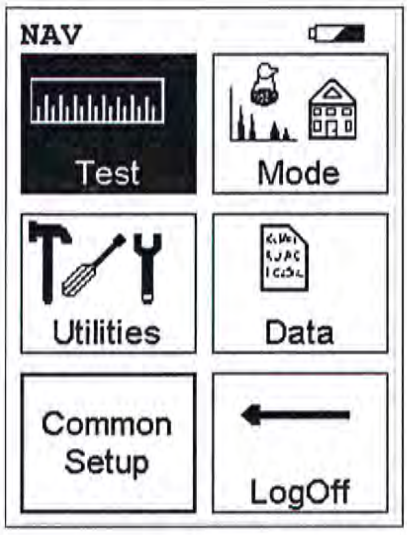
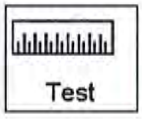


Figure 2-5. The Main Menu

All Niton XLp analyzers functions are accessible from the Main Menu and subsidiary menus. Each of the analyzer's functions represented by an icon on the **Main Menu** screen (Test, Mode, Utilities, Data, and Common Setup) may be selected by choosing the appropriate icon. When one of these Main Menu icons is selected, the function specific sub-menu appropriate to that icon will be displayed.



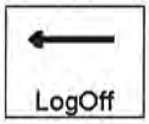
In order to test samples in the previously used test mode, simply select the **Test** icon. Your Niton XLp analyzer will operate in the testing mode that is currently selected. Choose an appropriate test mode for the samples you are testing by selecting a mode from the **Mode Menu**.

Access the **Mode Menu** by selecting the **Mode** icon from the **Main Menu** screen. The analyzer will remember the last mode used on the analyzer, and will use that mode by default unless another mode is selected.

Access the **Utilities Menu** by selecting the **Utilities** icon from the **Main Menu** screen. The **Utilities Menu** enables you to view analyzer specifications; set the date and time; and auto-calibrate the analyzer electronics and the touch screen display.

Access the **Data Menu** by selecting the **Data** icon from the **Main Menu** screen. The **Data Menu** allows you to view readings, and allows you to view the alloy library and stored signatures.

Access the **Common Setup Menu** by selecting the **Common Setup** icon from the **Main Menu** screen. The **Common Setup Menu** allows you to turn on or off the liquid crystal display backlight, turn on or off the integrated bar code scan engine, to enable and configure source utilization, and to enable or disable the printer.



Return to the **Logon screen** by selecting the **Logoff** icon. The **Logon Screen** logs you out and allows you to login again, preventing casual unauthorized access to your analyzer.

The Mode Menu

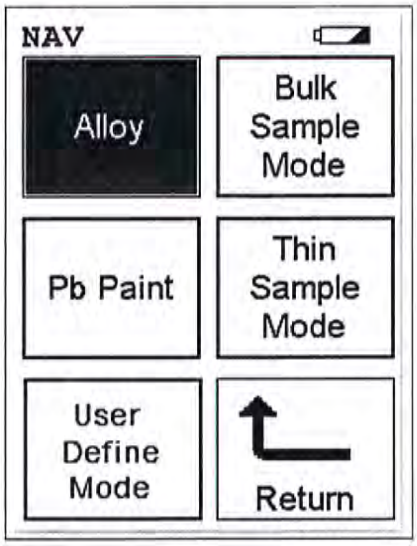
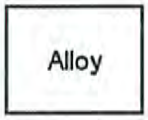
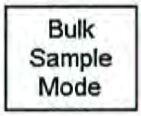


Figure 2-6. The Mode Menu

The **Mode Menu** enables you to select any one of the sample test modes that have been installed on your Niton XLp analyzer.



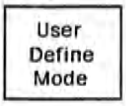
Select the **Alloy** icon to access any one of the alloy testing modes. The Alloy Modes enable you to test metal alloys for chemical composition and/or alloy identification. The Alloy Testing Modes are detailed in Chapter 3 of this User's Guide.



Select the **Bulk Sample Mode** icon to access Bulk Sample Mode. The **Bulk Sample Mode** allows you to analyze plastics, electronics, soil, mining, and other thick samples for metallic composition and/or contamination. The **Bulk Sample Mode** is detailed in Chapter 6 of this User's Guide.

The **Pb Paint Mode** allows you to determine lead (Pb) loading in paint samples in units of mg/cm^2, and to determine if paint samples are above or below the user-defined action level for lead in the shortest time possible. The **Pb Paint Mode** is detailed in Chapter 4 of this User's Guide.

Select the **Thin Sample Mode** icon to access the **Thin Sample Mode Menu**, where you may choose a thin sample testing mode appropriate to the samples you will be testing. The Thin Sample Testing Modes allow you to analyze thin samples, such as dust wipes and air filters, and coatings for elemental analysis. The Thin Sample Test Modes are detailed in Chapter 5 of this User's Guide.



Select the **User Definable Mode** icon to access **User Definable Mode.** The **User Definable Mode** allows you to create your own protocols and methods for your own use. The User Definable Mode is detailed in Chapter 7 of this User's Guide.

The Utilities Menu

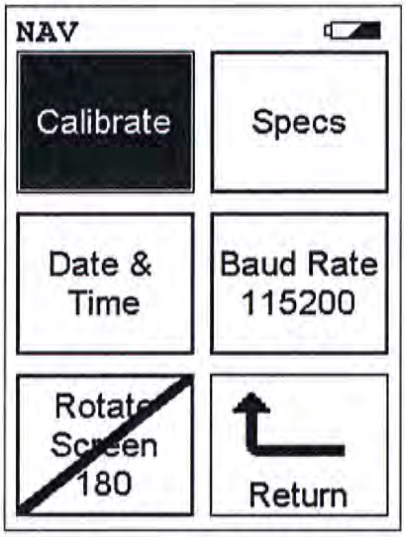
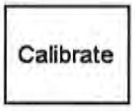


Figure 2-7. The Utilities Menu

To access the **Utilities Menu**, select the **Utilities** icon from the **Main Menu** display. The **Utilities Menu** enables you to view your Niton analyzer specifications, set the date and time, auto-calibrate your Niton analyzer's detector or touch screen display, Rotate the display on the LCD screen, or change the communication (baud) rate for the RS-232 port.



Select the **Calibrate** icon to access the **Calibrate Menu**. The Calibrate Menu allows you to calibrate the detector or to calibrate the touch screen interface.

Select the **Specs** icon to display the analyzer's specifications. These specifications include your Niton XLp's serial number, software and firmware versions, temperature, bias, and data coprocessors. Press the Close button to return to the Utilities Menu.

Select the **Date & Time** icon to set the date and time as needed for different time zones, daylight savings time, or any other reason. The date and time are factory preset prior to shipping. The format used is month/day/year - MM/DD/YY, and hour/minute - HH/MM, for the 24 hour clock.

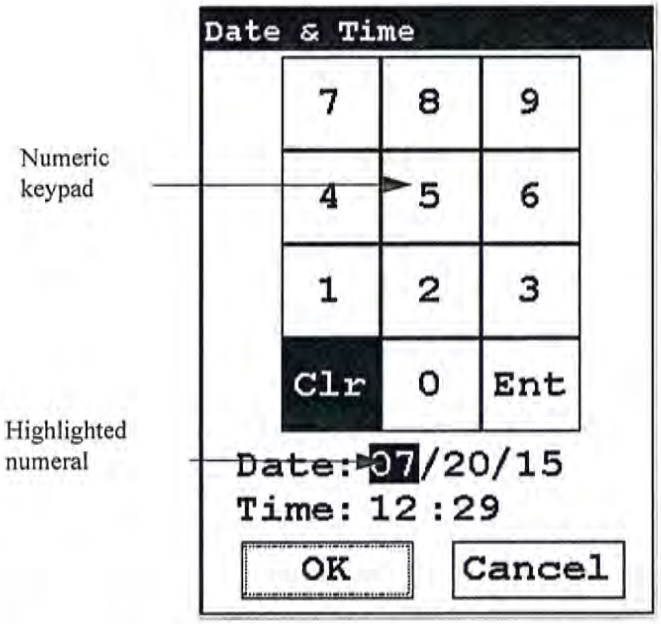


Figure 2-8. Setting the Date & Time

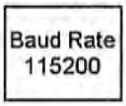
When the **Date & Time** icon is selected, the **Date & Time Screen** comes up on your XLp's LCD Screen. Initially, the first character of the month is highlighted in reverse video (white on black), as in the sample display shown here. To change a character, select the digit you want to replace the character with from the virtual numeric keypad displayed on the screen, then select the Enter (Ent) character from the virtual numeric keypad. Your Niton XLp will then accept the entry and automatically advance to the next digit. To skip a character, simply select the Enter (Ent) character from the virtual numeric keypad without selecting a replacement character.

For example, on the sample display, if you wish to change the "06" of the month to "07", the display appears with the first character (0) highlighted. Select the Enter (Ent) character to skip the zero. The "6" will now be highlighted. Select the "7" digit from the virtual numeric keypad, then select the Enter (Ent) key from the virtual numeric keypad. The change is accepted and the next digit is highlighted. Continue to select the Enter (Ent) symbol from the virtual numeric keypad to skip over the remaining characters of the date and time until the last character is reached. When you select the Enter (Ent) key from the virtual numeric keypad to confirm the last character, the word "SUCCESS" will appear beneath the Time field, and you will be returned to the Main Menu. The date is given in month/day/year format.

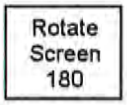
CAUTION Your XLp analyzer must have the correct time and date set in order to test samples correctly using the ^{109}Cd radioisotope source. Please make sure that the time and date are set correctly before testing. ♦

WARNING! It is important that the date and time information displayed on the **Date and Time Screen** is correct. If either the date or time is incorrect, the information stored with your readings will be incorrect. In addition, an incorrect date prevents the instrument from properly compensating for normal source decay - causing erroneous analysis results for instruments equipped with 109Cd. This information must be correct before proceeding with testing. ♦

Note The analyzer will automatically return you to the **Main Menu** when the entry is complete. ♦



Select the **Baud Rate** icon to change the communications rate for the RS-232 port. Selecting the **Change Baud Rate** icon will toggle the communications rate between 115 kbaud and 38 kbaud. The default 115 kbaud rate may be too fast for some older computers, and dropping the rate to 38 kbaud will insure proper communications between your PC and your Niton XLp Analyzer.



Select the **Rotate Screen 180** icon to toggle the orientation of the screen between right side up and upside down.

This is used primarily when your analyzer is placed in the NITON Environmental Test Stand, because the analyzer is inverted when properly installed in the Test Stand.

The Data Menu

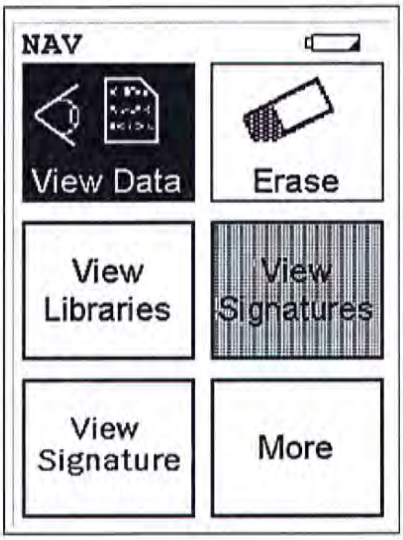
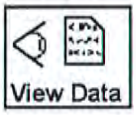


Figure 2-9. The Data Menu - First Page

To access the **Data Menu**, select the **Data** icon from the **Main Menu** display. The **Data Menu** enables you to access readings and libraries, for viewing or manipulation.

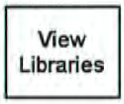


Select the **View Data** icon in order to access data from readings you have already taken. Selecting the **View Data** icon will bring you to the **Data Screen**.

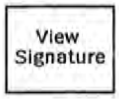
Select the Erase icon to access the **Erase Menu**. The **Erase Menu** allows you to erase your data.

CAUTION Never turn off the analyzer while data is being erased! •

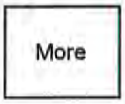
WARNING! Do not attempt to take measurements while downloading readings! This will generate an error requiring a system reset, and may corrupt your stored readings, requiring all stored readings to be erased. •



Select the **View Libraries** icon to access the **Library View Menu**. The **Library View Menu** allows you to view data in the Alloy Grade Library as well as the Superlib and Superstds libraries.



Select the **View Signatures** icon to view data saved as reference sample signatures in Signature ID Mode. When the **View Data** icon is selected, the Results screen of your most recent test is shown on the LCD display.



Select the **More** icon to access the second page of the **Data Menu**.

The Data Menu - Second Page

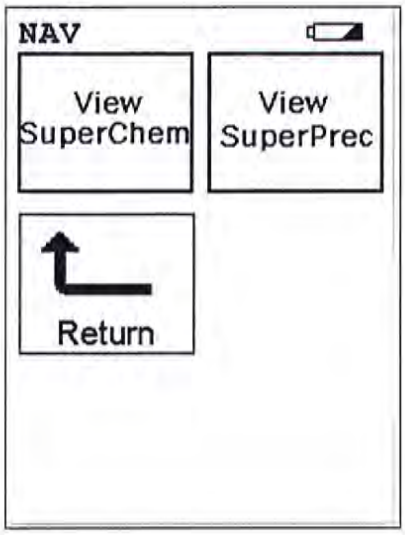
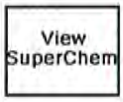
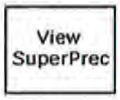


Figure 2-10. The Data Menu - Second Page

To access the **Data Menu Second Page**, select the **More** icon from the **Data Menu** display. The **Data Menu** enables you to access readings and libraries, for viewing or deleting.



Select the **View SuperChem** icon in order to view your SuperChem data. The **View SuperChem** icon allows you to view your **SuperChem Mode** data, and also allows you to delete individual readings.When the **View Data** icon is selected, the Results screen of your most recent test is shown on the LCD display.



Select the **View SuperPrec** icon in order to view your SuperPrec data. The **View SuperPrec icon** allows you to view your **SuperPrec Mode** data, and also allows you to delete individual SuperChem standards.When the **View Data** icon is selected, the Results screen of your most recent test is shown on the LCD display.

The Data Screen

Use the **Data Screen** to view previously taken test result readings. When the **View Data** icon is selected, the Results screen of your most recent test is shown on the LCD display.

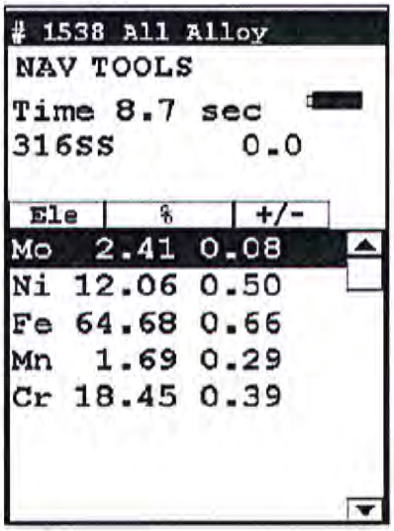


Figure 2-11. The Results Screen

Using the buttons on the control panel, you may view different readings or additional data for individual readings.

Scrolling Down Through the Complete Listing of Elements

Pressing the "Left" position on the 4-way touch pad of your Niton analyzer will display the previous reading, or if the first reading is currently displayed, the last reading. Pressing the "Right" position on the 4-way touch pad will display the next reading, or if the last reading is currently displayed, the first reading in memory. Niton analyzers can store between 3000 to 6000 readings.

You can also look at the complete x-ray spectra for each reading stored in the analyzer's memory.

Sorting Elements

The Sort Buttons, which double as column headings, can be used to re-sort the data in different ways. The **Data Screen** always begins as a Standard Sort, as you have defined it. Selecting the appropriate sort button once

toggles the sort order to High-to-Low. Selecting the sort button again toggles the sort order to Low-to-High. To return to the Standard Sort, view a different reading and return.

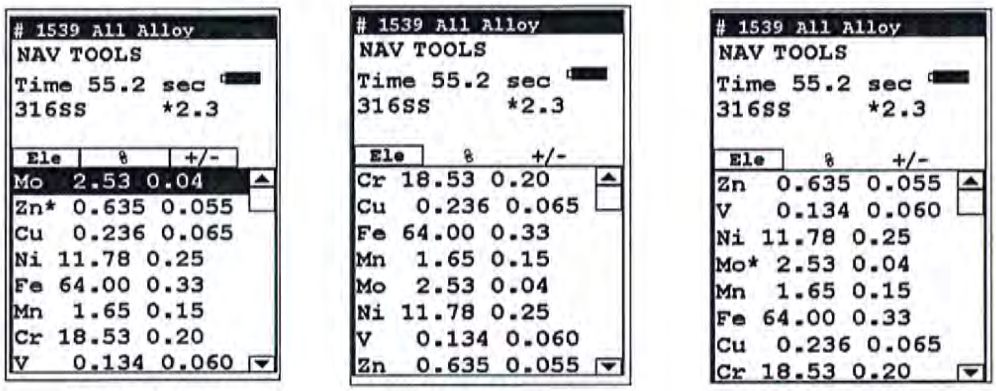


Figure 2-12. Element Sorts

Element Sorts Element sorts are performed alphanumerically based on the element name.

Composition Sorts Composition sorts are performed numerically based on the percentage of composition.

Error Sorts Error sorts are performed based on the range of error in the reading.

Spectrum Graph

For any reading result, simply use the **NAV Menu** to gain access to the reading's spectrum graph. Selecting Spectra will show a graphed spectrum of this reading, called SpectraView. SpectraView can be a useful tool for rapid, qualitative analysis of sample chemistries. See "SpectraView" on page A-7 for details.

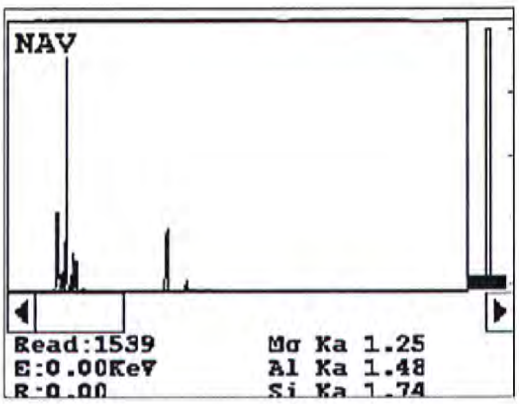


Figure 2-13. The SpectraView Screen

The Data Entry Screen

Selecting **Data Entry** will access the identifying criteria for each reading. In this scrolling screen, you can see the identifying criteria you input into the **Data Entry Screen** describing the conditions of the reading analysis sample and other data concerning the reading. The parameters are context sensitive - they change depending on the current mode.

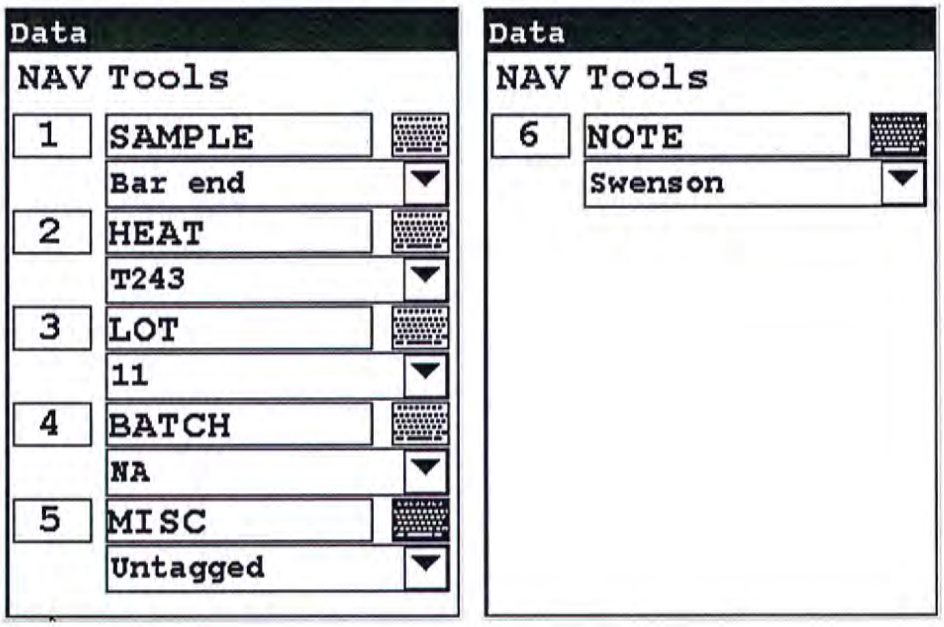


Figure 2-14. Sample Setting Parameters - Alloy Mode

The Calibrate Menu

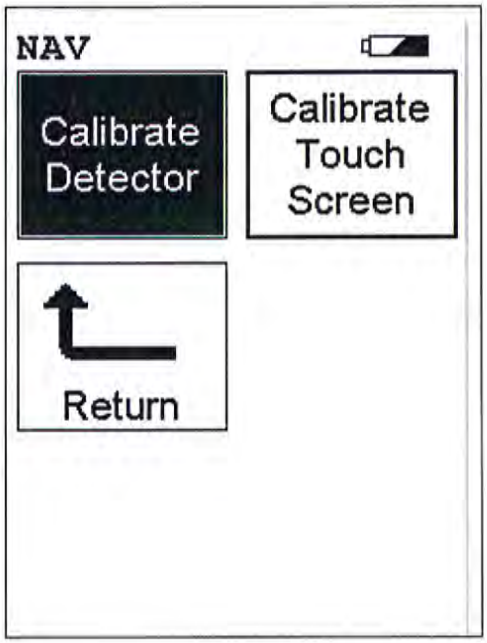


Figure 2-15. The Calibrate Menu

To access the **Calibrate Menu**, select the **Calibrate** icon from the **Utilities Menu**. The **Calibrate Menu** enables you to calibrate your analyzer's electronics.

Select the **Calibrate Detector** icon to begin a standard calibration of your analyzer's detector. Once you select the **Calibrate Detector** icon, calibration will begin immediately. The analyzer is programmed to calibrate for a specific, predetermined period in order to ensure proper operation of your Niton XLp analyzer in the field.

CAUTION Avoid any vibration, loud noise, strong electronic fields, or other possible interference when your analyzer is calibrating its detector. ♦

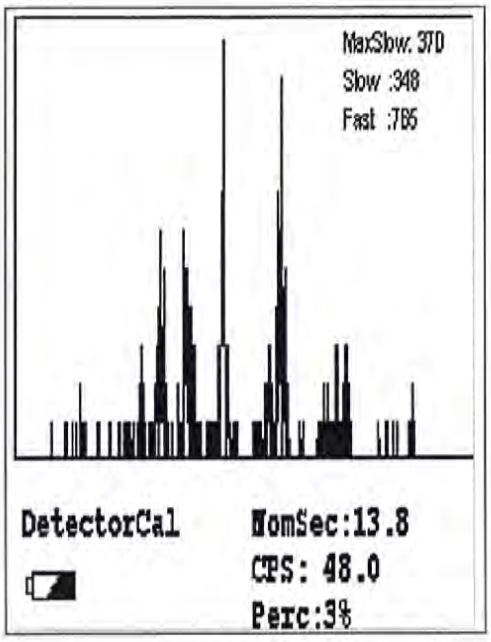


Figure 2-16. Detector Calibration Screen

The analyzer calibration screen will be displayed until calibration is complete. After the calibration has finished, the calibration results will be displayed. Press the on/off/escape button or the **Return** icon to return to the **Main Menu**. In order to insure good test results, it is essential that you calibrate your Niton XLp300 Series Lead-based-Paint Analyzer's detector daily, and if a check sample test reveals discrepancies in the reading.

Calibrate Touch Screen

Select the **Calibrate Touch Screen** icon to re-calibrate the analyzer's touch screen display. This procedure establishes the display boundaries for the touch screen interface. When the **Calibrate Touch Screen** icon is selected, the display will show the message: "Calibrate Touch Screen". There will be a small cross in the upper left-hand corner of the display. Tap on this cross with the stylus, and the cross will disappear and reappear in the lower left-hand corner of the screen. Tap on the cross again, and it will reappear in the lower right-hand corner of the screen. Tap on the cross again and it will reappear in the top right-hand corner of the screen. Tap on the cross once more, and you will be presented with the **Calibrate Menu**

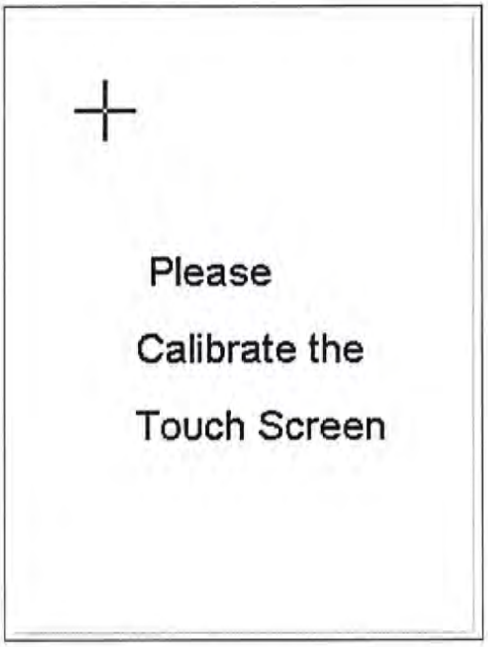


Figure 2-17. The Touch Screen Calibration Screen

The View Libraries Menu

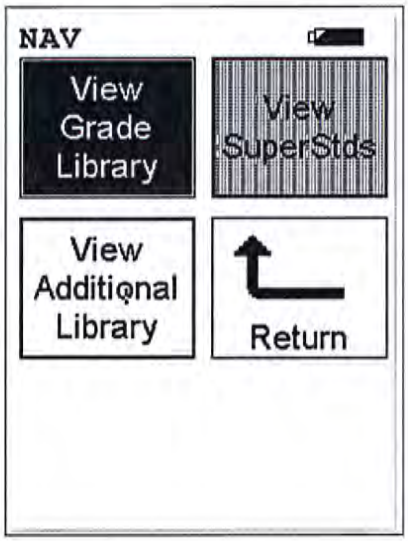
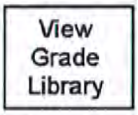
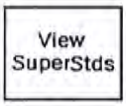


Figure 2-18. The View Libraries Menu

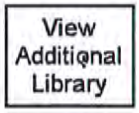
To access the **View Libraries Menu**, select the **View Libraries** icon from the **Data Menu**. The **View Libraries Menu** enables you to view reference data from your analyzer's libraries.



Select the **View Grade Library** icon to view your default Alloy Grade Library, which is set by Thermo Scientific. The entries in the Grade Library serve as a reference for chemistry based analysis. The library entries allow the analyzer to work properly "out of the box" without needing time-consuming pre-analysis. Please refer to the NDT User Guide for information on modifying the Grade Library.



Select the **View SuperStds** icon in order to view your SuperStds Alloy library.



Select the **View Additional Library** icon in order to view your Additional Library alloys, which you may have uploaded to your Analyzer with the NDTI utility. If you have not uploaded an additional library, the **View Additional Library** icon will be grayed-out and the View Additional Library function will be unavailable.

The Common Setup Menu

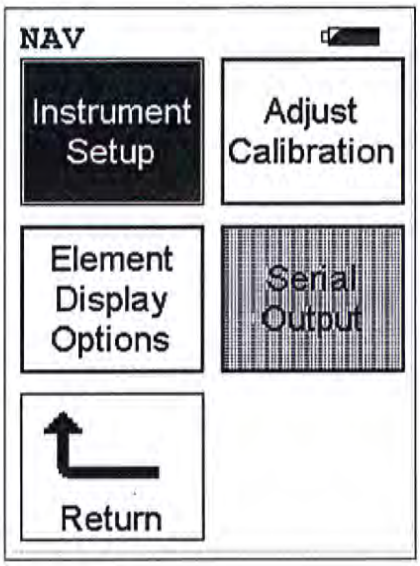
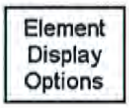


Figure 2-19. The Common Setup Menu

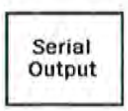
To access the **Common Setup Menu**, select the **Common Setup** icon from the **Main Menu** display. The **Common Setup Menu** gives you access to various Niton XLp analyzer functions, including touch screen display backlighting and the built in bar code scanner, and enables you to configure the way the sources contained in your Niton XLp analyzer are used in sample testing.

Select the **Instrument Setup** icon to access the **Instrument Setup Menu**. The **Instrument Setup Menu** enables you to toggle the display backlight on or off, configure the Bluetooth wireless connection, configure trigger/proximity button use, or configure sources.

Select the **Adjust Calibration** icon to access the **Calibration Adjustment Setup Screen**. The **Calibration Adjustment Setup Screen** enables you to recalibrate your analyzer for more effective analysis in the various different modes available to you. This option is only recommended for use after consulting with Niton Applications



Select the **Element Display Options** icon to bring up the **Element Display Menu**. The **Element Display Menu** enables you to control the sorting of elements in the sample display, and the pass-fail threshold for sample analysis.



Select the **Serial Output** icon to access the **Serial Output Menu**. The **Serial Output Menu** allows you to adjust the parameters for downloading data across a serial line to a computer, printer, or other computing device.

The Element Display Menu

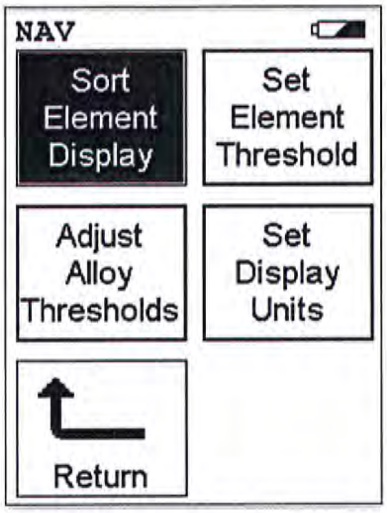
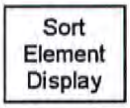


Figure 2-20. The Element Display Menu

To access the **Element Display Menu**, select the **Element Display** icon from the **Common Setup Menu**. The **Element Display Menu** enables you to control element sorting parameters and adjust elemental thresholds.



Select the **Sort Element Display** icon to configure sorting criteria used for analysis display. Selecting the **Sort Element Display** icon opens up the **Sort Criteria Screen**. Select the mode you wish to change, and the **Sorting Options Screen** will appear.

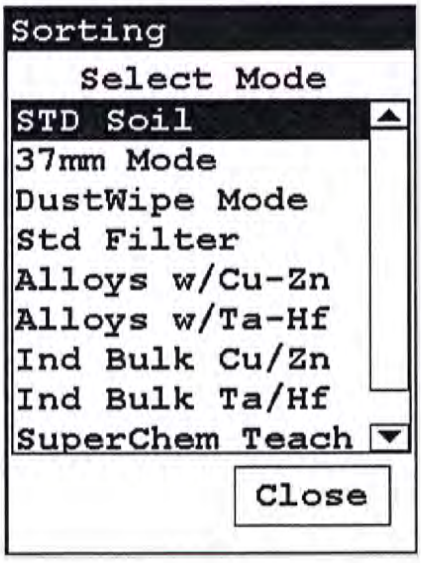


Figure 2-21. The Sort Element Display

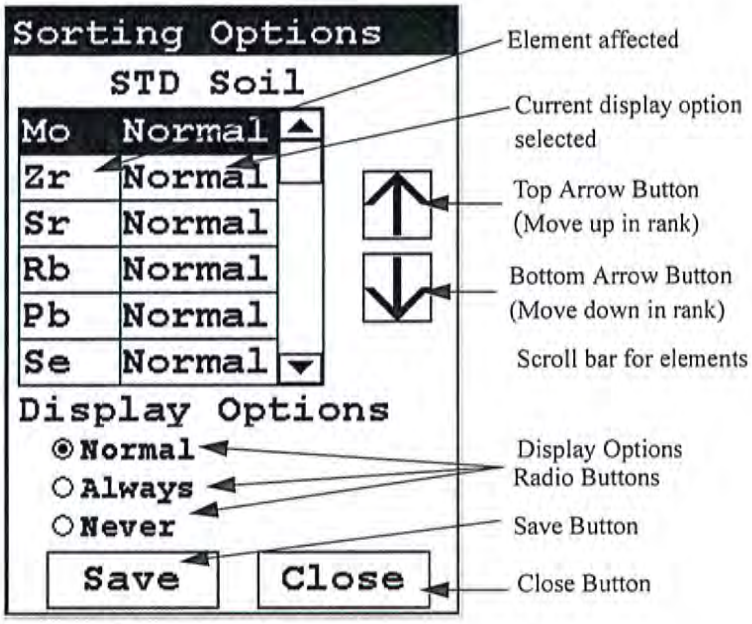


Figure 2-22. The Sorting Options Screen

On the left of the display are elements, each with its currently selected display option beside it to the right. The element list is ranked by importance, with the most important element on top, and each one lower down of less importance than the one above it.

By selecting an element and using the arrow buttons to the right of the list, you can change its ranking. Use the Top Arrow Button to move an element one rank closer to the top with each click. Use the Bottom Arrow Button to move an element one rank closer to the bottom with each click.

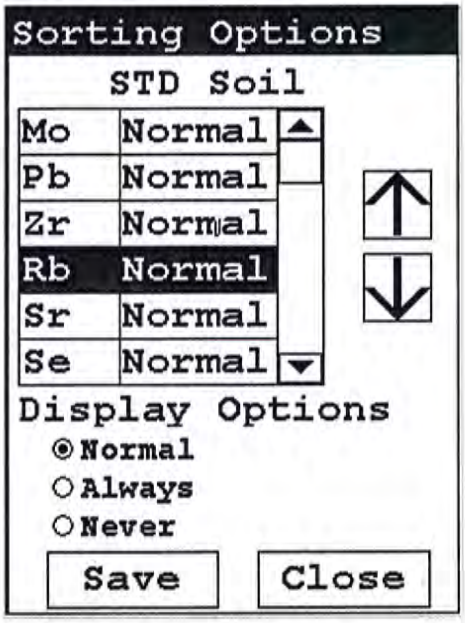


Figure 2-23. Changed Sort Order

The Display Options Radio Buttons allow you to change the display status of any element to one of three states:

- Normal - The standard state. Element displays only when the elemental value is greater than the limit of detection.
- Always - Always display the results for this element. Use this state for elements critical to all of your analyses.
- Never - Never display the results for this element. Use this state for elements which are unimportant to your work. This makes your instrument display less complex.

Select the element you want to change, then select the radio button corresponding to your choice of display status. The currently selected element is displayed in white on black.

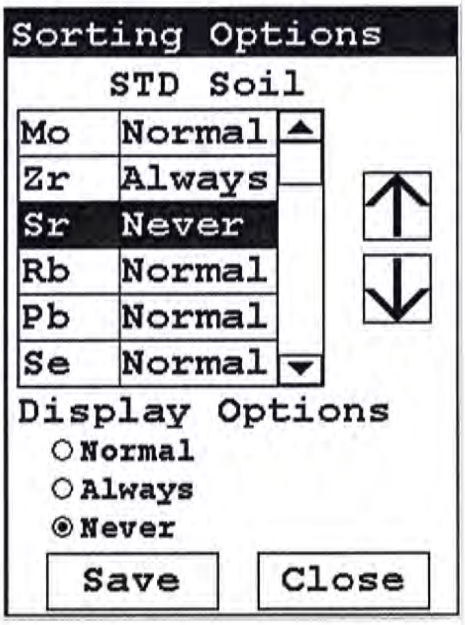


Figure 2-24. Changed Display Options

Select the Save Button to save your current status as the new default. After saving, you will go back to the **Element Display Menu**.

Select the Close Button to exit without saving. When you select the Close Button after changing the display state of any element, a screen will open asking you if you want to save the changes you made. Selecting "Yes" will save these changes as the new default. Selecting "No" will return you to the **Element Display Menu** without saving the changes.

Figure 2-25. Save Changes

Select the **Set Element Threshold** icon to configure pass and fail criteria for elemental analysis. Selecting the **Set Element Threshold** icon opens the **Set Threshold Screen**.

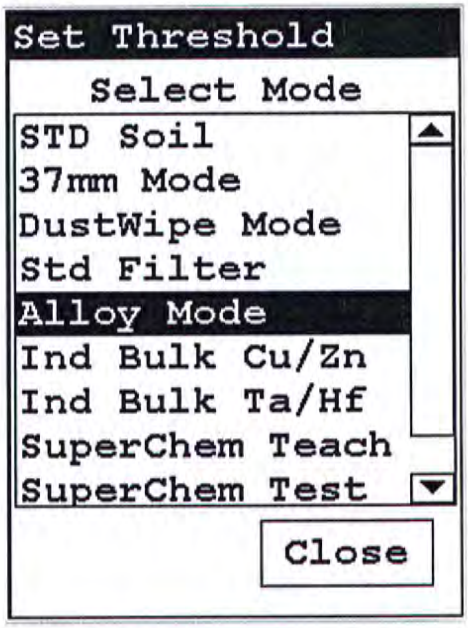


Figure 2-26. Set Threshold Screen

Select the mode you wish to work with from the scrollable list. This will open up the **Pass/Fail Settings Screen** for that mode.

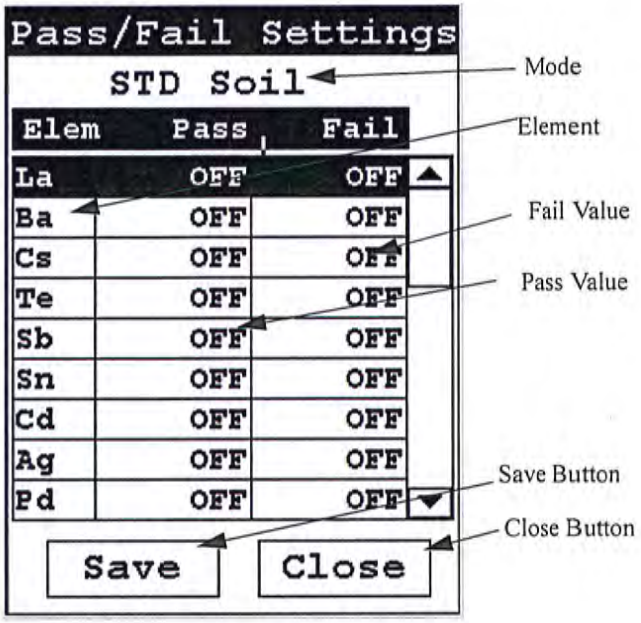


Figure 2-27. Pass/Fail Settings Screen

Selecting the Pass Value will open up the Pass Editor for the selected element.

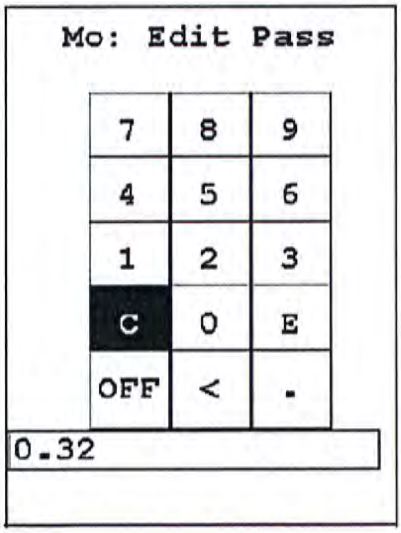


Figure 2-28. The Pass Editor

The Pass Editor is very similar to the Logon Screen. The "C" button clears the field, and the "<" button clears the last numeral. Select the numerals you want, then press "E" to enter the number. "OFF" resets the value to "OFF"

Selecting the Fail Value will open up the Fail Editor for the selected element.

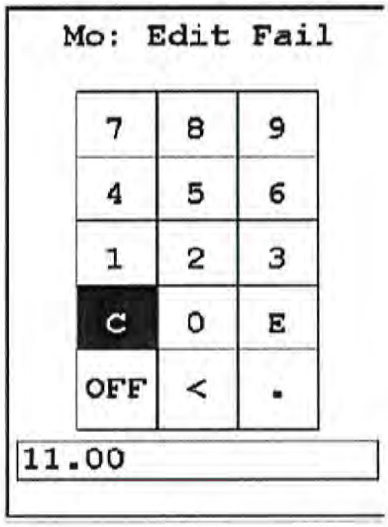


Figure 2-29. The Fail Editor

The Fail Editor works the same as the Pass Editor.

When you press the "E" button in either editor, you are returned to the Pass/Fail Settings Screen, with your new values in place.

Selecting the "OFF" button not only sets the value to "OFF" but also saves the new value.

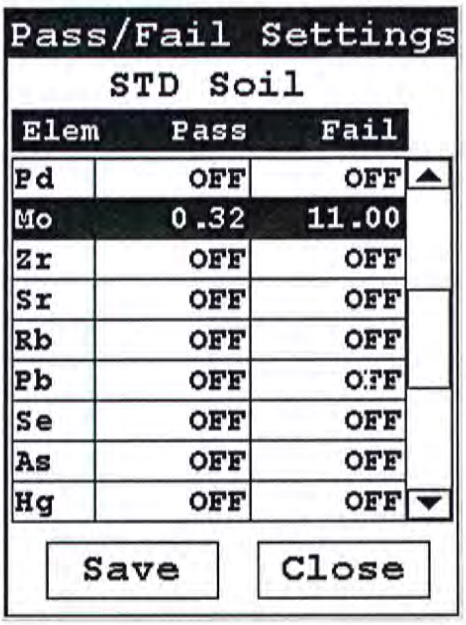


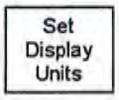
Figure 2-30. ThePass-Fail Settings Screen with new parameters

Select the Save Button to save your current status as the new default. After saving, you will go back to the **Element Display Menu**.

Select the Close Button to exit without saving. When you select the Close Button after changing the display state of any element, a screen will open asking you if you want to save the changes you made. Selecting "Yes" will save these changes as the new default. Selecting "No" will return you to the **Element Display Menu** without saving the changes.

Figure 2-31. Save Changes Screen

Select the **Adjust Alloy Threshold** icon to configure pass and fail criteria for alloy analysis. Selecting the **Adjust Alloy Threshold** icon opens the **Alloy Threshold Screen**.



Select the **Set Display Units** icon to choose between ppm (parts per million) and percentage (hundredths of whole) displays when taking readings. Selecting the **Set Display Units** icon opens the **Display Units Screen**.

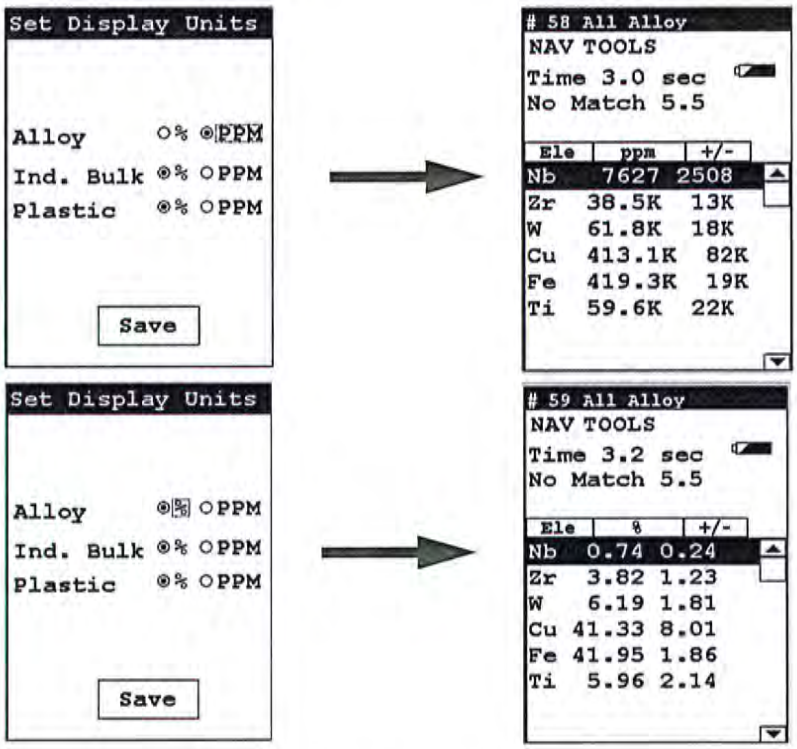


Figure 2-32. Display Units Screen and Associated Results Screens

The Display Units Screen

The **Display Units Screen** allows you to select either PPM or Percentage display on the **Results Screen** for **Alloy**, **Industrial Bulk**, or **Plastic Modes**. Select the radio button for the preferred display unit, then select the Save button, and subsequent results will be shown in that unit type.

Note Readings will retain the unit type used when the reading was taken, even if the units are changed. ♦

Cutoff numbers set the allowable limits in identifications of analyzed samples. The higher the cutoff is set, the easier a match can be made in the Alloy Analysis, Signature ID, and SuperChem modes.

The Erase Menu

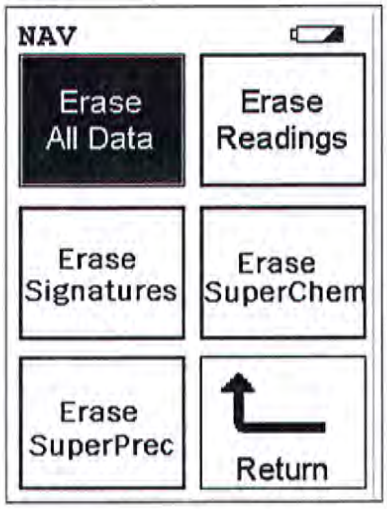
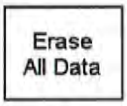
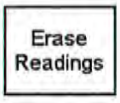


Figure 2-33. The Erase Menu

To access the **Erase Menu**, select the **Erase** icon from the **Data Menu**. The **Erase Menu** enables you to delete data from your analyzer, allowing you to gain storage space for more recent data. You should only erase data which you have already transferred to permanent storage, or when requested to do so by Thermo service personnel.

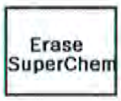


Select the **Erase All Data** icon to erase all data, including signatures and SuperChem reference readings, from your analyzer. Selecting the **Erase All Data** icon will bring up a confirmation screen Figure 2-34) asking you "Are you sure?" with options to select "YES" or "NO". Selecting "YES" will erase all reading data from your analyzer. Selecting "NO" will return you to the **Erase Menu**.

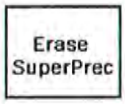


Select the **Erase Readings** icon to erase all accumulated test readings from your analyzer. Selecting the **Erase Readings** icon will bring up a confirmation screen Figure 2-34 asking you "Are you sure?" with options to select "YES" or "NO". Selecting "YES" will erase all test reading data from your analyzer. Selecting "NO" will return you to the **Erase Menu**.

Select the **Erase Signatures** icon to erase all accumulated alloy signatures from your analyzer. Selecting the **Erase Signatures icon** will bring up a confirmation screen Figure 2-34 asking you "Are you sure?" with options to select "YES" or "NO". Selecting "YES" will erase all signature data from your analyzer. Selecting "NO" will return you to the **Erase Menu**.



Select the **Erase SuperChem** icon to erase accumulated SuperChem reference readings from your analyzer. Selecting the **Erase SuperChem** icon will bring up a confirmation screen Figure 2-34 asking you "Are you sure?" with options to select "YES" or "NO". Selecting "YES" will erase all SuperChem reference reading data from your analyzer. Selecting "NO" will return you to the **Erase Menu**.



Select the **Erase SuperPrec** icon to erase accumulated SuperPrec reference readings from your analyzer. Selecting the **Erase SuperPrec** icon will bring up a confirmation screen Figure 2-34 asking you "Are you sure?" with options to select "YES" or "NO". Selecting "YES" will erase all SuperPrec reference reading data from your analyzer. Selecting "NO" will return you to the **Erase Menu**.

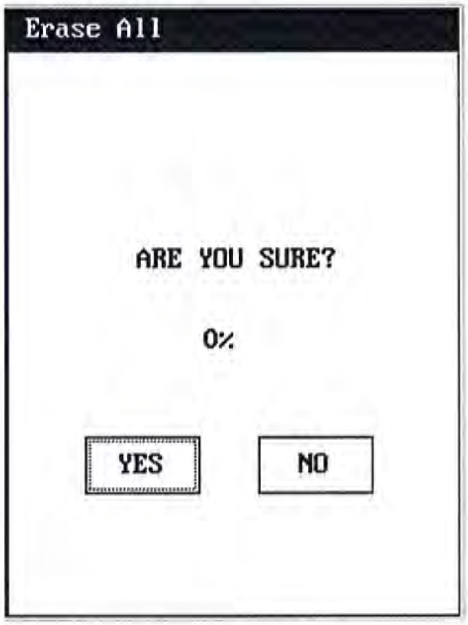


Figure 2-34. Confirmation Screen

The confirmation screen has a progress meter which tells you how far along your erasure is at any given time.

The Instrument Setup Menu

Select the **Backlight** icon to toggle the display backlight on or off.

CAUTION Enabling the backlight will reduce your battery pack operating time, requiring more frequent recharges. ♦

Select the **Wireless Setup** icon to access the **Wireless Setup Menu**. The **Wireless Setup Menu** enables you to configure the Bluetooth wireless networking for your analyzer.

Select the **Hardware Setup** icon to access the **Hardware Setup Screen**. The **Hardware Setup Screen** enables you to configure trigger, safety interlock, and proximity button use.

Select the **Source Config** icon to access the **Source Config Screen**. The **Source Config Screen** enables you to completely configure parameters and use of your analyzer's excitation sources.

Select the **Printer Setup** icon to access the **Printer Setup Screen**. The **Printer Setup Screen** enables you to configure printer parameters.

The Hardware Setup Screen

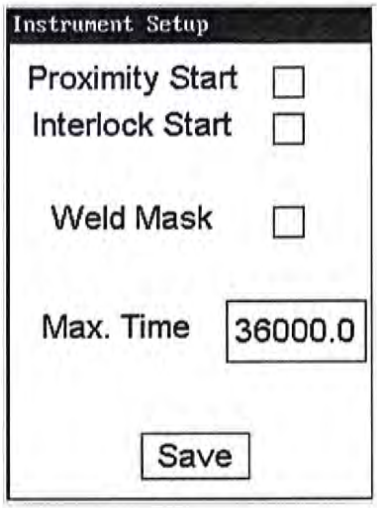


Figure 2-35. The Hardware Setup Screen

The **Hardware Setup Screen** enables you to toggle various options on or off, as well as select certain hardware dependant modes. Selecting an empty checkbox enables the option and places a check in the box. Selecting a checked box disables the option and clears the box.

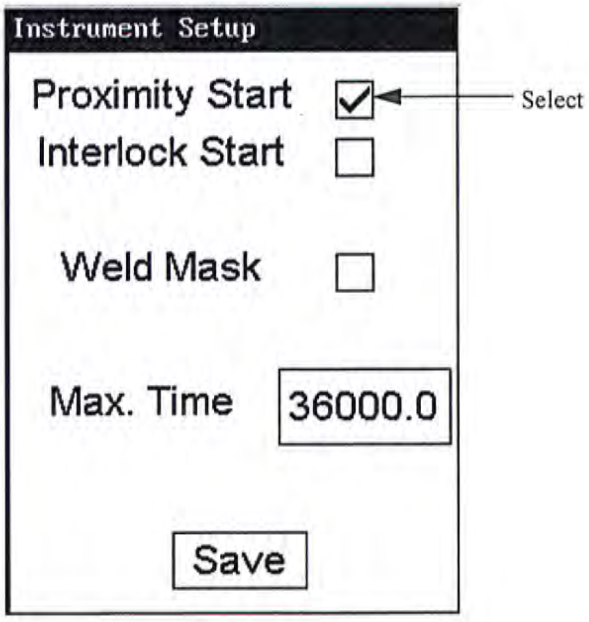


Figure 2-36. Selecting Options

Select the Proximity Start checkbox to toggle the use of the front proximity button. This enables the proximity button to be used to start taking a sample on contact. Some nations have laws or regulations which prohibit use of this feature. In this case, the feature will be disabled before shipping.

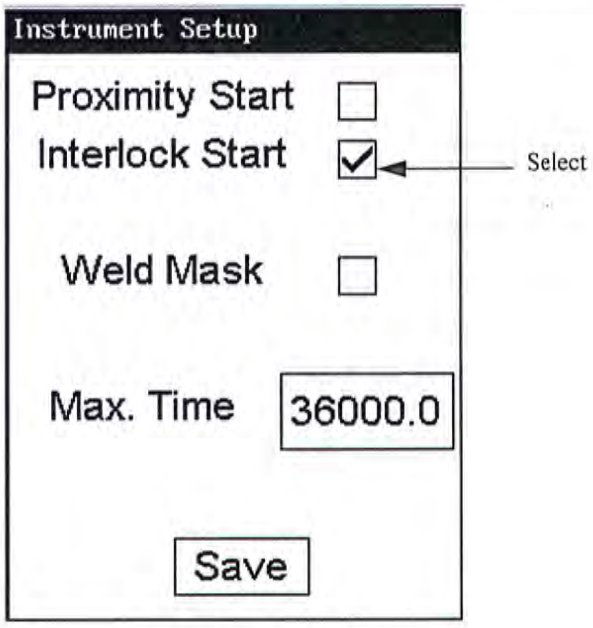


Figure 2-37. Selecting Other Options

Select the Interlock Start checkbox to toggle the use of the rear interlock button. This requires the interlock button to be used to start taking a sample on contact. Enabling the "Interlock Start" feature allows the user to start an analysis by depressing the rear interlock button on the analyzer.

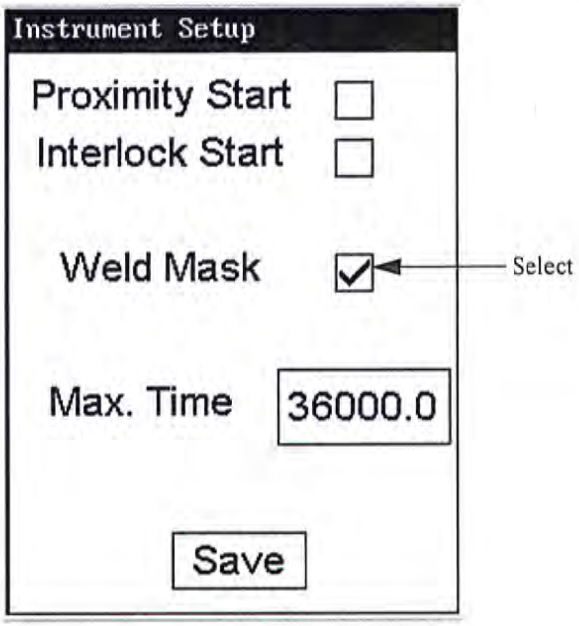


Figure 2-38. Select the Welding Mask Checkbox

Select the **Weld Mask** checkbox to toggle the use of the Welding Mask function. This reconfigures the calibration of the analyzer, enabling you to take readings using the Welding Mask.

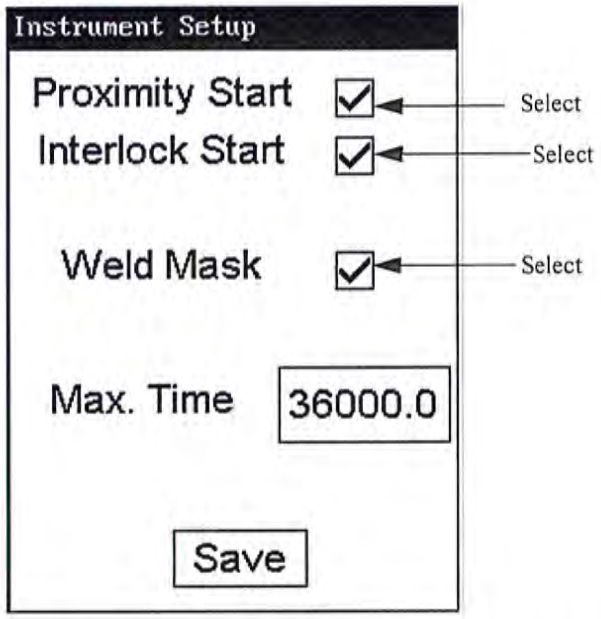


Figure 2-39. Selecting Option Combinations for Multiple Effects

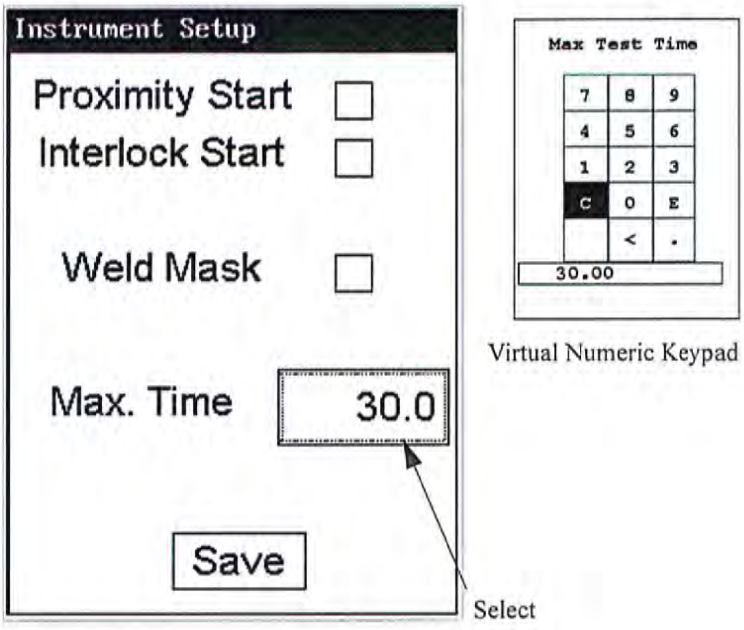


Figure 2-40. Changing the Max Time Parameter

Select the numbers box in the Max Time field to change the maximum seconds per reading. A virtual numeric keypad will appear, allowing you to set the number to whatever value you want, up to the maximum of 36000. When the max testing time is reached during an analysis, the analyzer reading will be automatically ended. Your analyzer will continue switching filters as needed until you terminate the reading or the Max Time is reached.

The Source Config Screen

Multi-source tests are used to either preferentially excite specific elements for increased sensitivity, or to cover a wider element range.

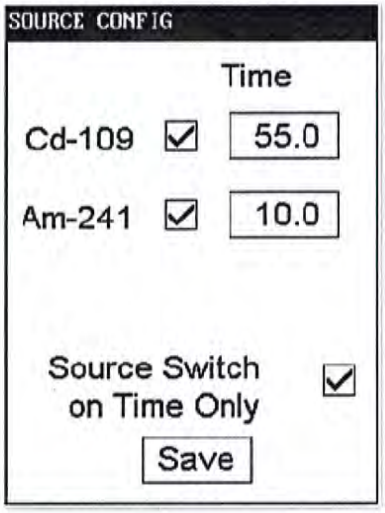


Figure 2-41. The Source Config Screen

The **Source Config Screen** enables you to directly enable or disable any source, or control the time that a source irradiates the sample before auto-switching to another source.

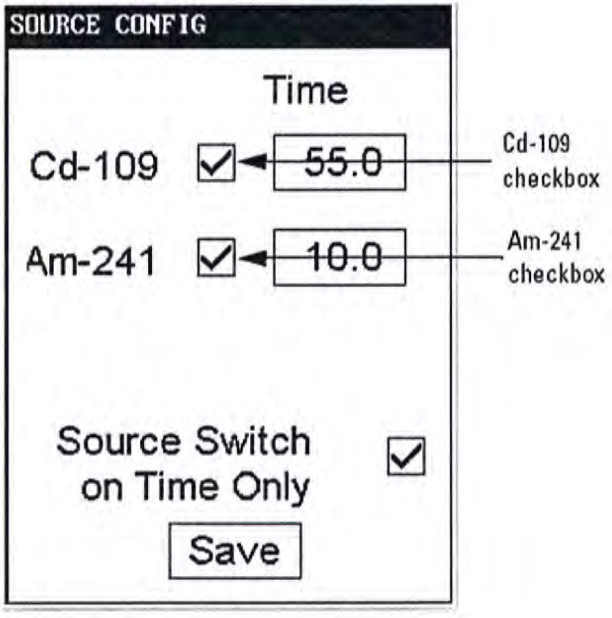


Figure 2-42. The Source Checkboxes

Select the checkbox next to the source you want to use to determine exactly which of the sources contained in your Niton Analyzer is used for sample testing. Selecting an empty checkbox will enable that source and place a check into the box as an indicator. Selecting a checked box will disable the source and clear the box.

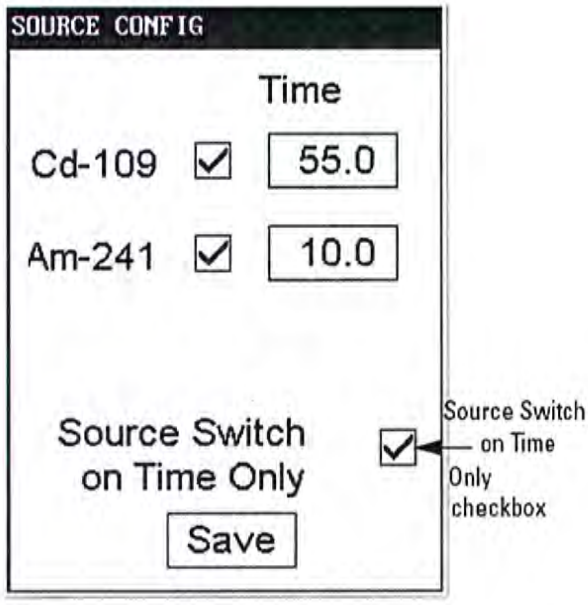


Figure 2-43. The Source Switch on Time Only Checkbox

Select the Source Switch on Time Only checkbox to toggle **Time Switch Only Mode** on or off. In **Time Switch Only Mode**, the Niton XLp analyzer will ignore the Alloy Library and only switch sources according to the time interval you set in the Time field for each source.

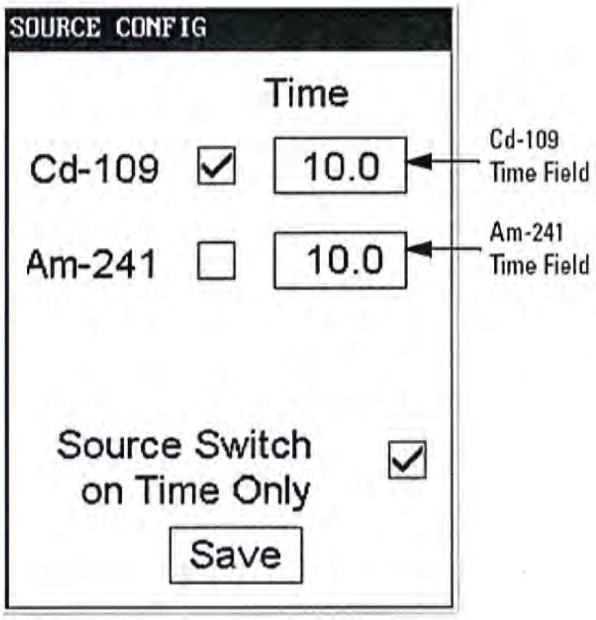


Figure 2-44. The Source Time Fields

Select the Time field for the intended source to change the source switch time for that source. This enables you to set the number of seconds each enabled source is allotted before auto-switching will occur when needed during sample testing. Your Niton XLpanalyzer will auto-switch from one source to another when the testing time for that source is greater than or equal to the time you have chosen, and the identified alloy is flagged as needing the switch in the Niton Alloy Library.

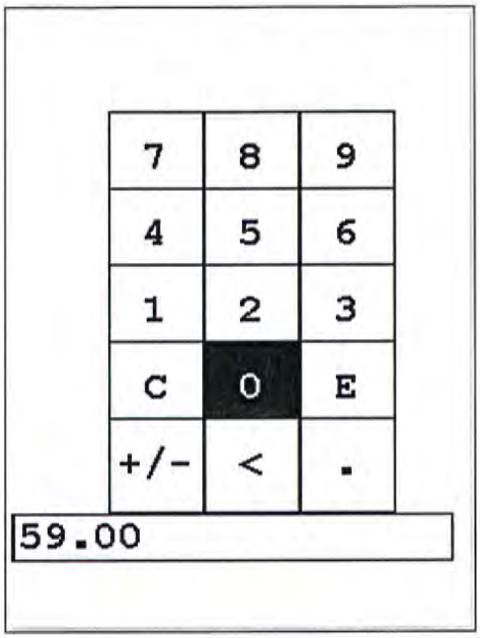


Figure 2-45. The Source Time Editor

Select the "C" key to clear the current time, then from the virtual numeric key pad, select each digit you want to input, then select "E" to enter.

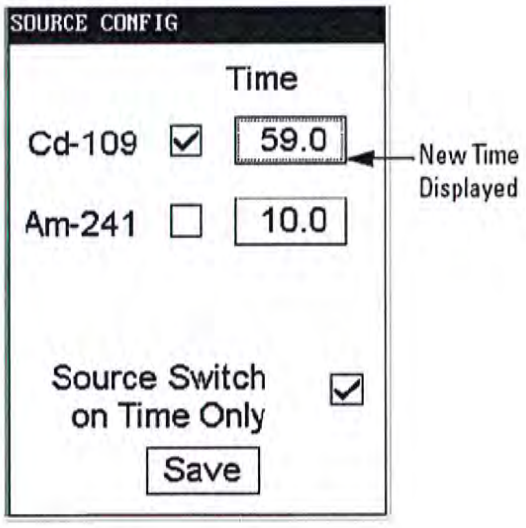


Figure 2-46. New Time Displayed

The Printer Setup Screen

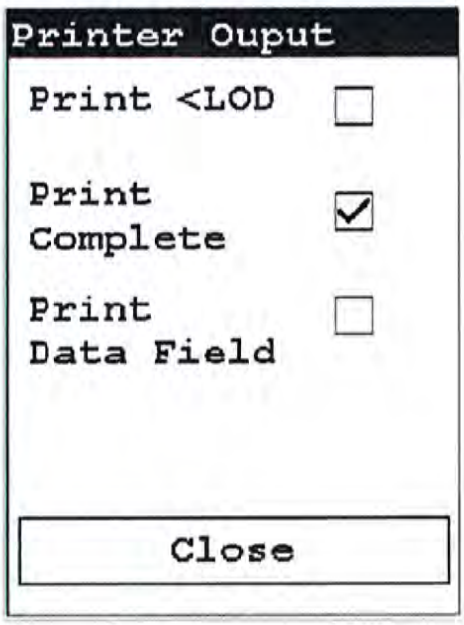


Figure 2-47. The Printer Setup Screen

The Printer Setup Screen allows you to adjust which sections of your reading data are sent to your optional printer. By default, your analyzer prints the detected list, reading number, reading length, reading mode and any applicable measurement data such as Alloy match grade names. You can select any combination of options on the Printer Setup Screen to change what is printed.

Print < LOD Selecting this option will enable printing of readings which are lower than the Limit of Detection.

Print Complete Selecting this option will enable printing of all the data fields in the reading.

Print Data Field Selecting this option will enable printing of all entered data fields.

Industrial Bulk Calibration Factors

Note Your NITON Analyzer may not be equipped with Industrial Bulk Mode. If you do not have Industrial Bulk Mode enabled on your analyzer, this section will not apply to you. ♦

Use Industrial Bulk mode when you expect that the element of interest to you will be more than 1% of the total sample. If you expect that the element of interest to you will be less than 1% of the total sample, use Standard Soil mode.

Within Industrial Bulk mode there are two sub-modes; Industrial Bulk (Cu / Zn / Pb) and Industrial Bulk (Ta / Hf / Re). Use Industrial Bulk (Ta / Hf / Re) mode only if you are specifically looking for compounds that contain those elements. If you are not specifically looking for compounds that contain tantalum, hafnium, or rhenium, then start by using Industrial Bulk (Cu / Zn / Pb) mode. This mode is more common than Industrial Bulk (Ta / Hf / Re) mode.

The XRF Analyzer can quickly identify and measure the presence of many elements, but it can not detect the lighter elements, such as carbon and silicon. However, concentrations of these lighter elements can affect the measurement of the heavier elements. Calibration factors are used to correct for the presence of these light elements in the sample matrix.

Industrial Bulk mode lets you calculate your own site-specific calibration factors and add them to the XRF Analyzer.

When you begin using the XRF Analyzer, NITON recommends that you test the XRF Analyzer against known samples of the material of interest, and add calibration factors, if needed.

When you change to a material that has a different matrix, such as moving from a matrix with high carbon content to a matrix with high silicon content, NITON recommends that you again test the XRF Analyzer against known samples. If the XRF Analyzer still provides results that are accurate enough for your needs, you can continue to use it without changing the calibration factors. However, if the change in the matrix of the sample causes a change in the results from the XRF Analyzer, such that they are no longer accurate enough for your needs, then calculate new calibration factors for the new sample type.

Displaying the Calibration Factors

To display the calibration factors:

1. **From the Main menu of the XRF Analyzer, select Common Setup.**

2. **Select Adjust Calibration.**

3. **Select the mode in which to adjust the call factors.**

Figure 2-48. Select Mode

Once you have selected the mode yoou want to edit, the CalFactor Screen fr that mode will appear.

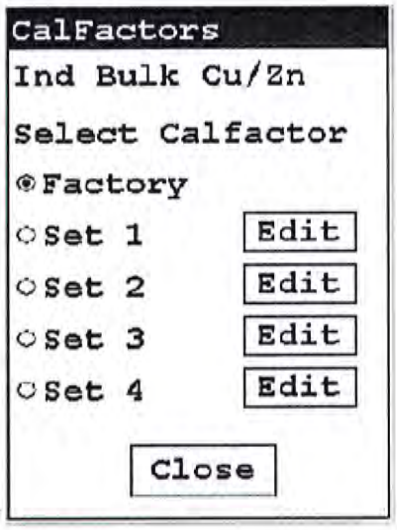


Figure 2-49. CalFactors Screen (for Inustrial Bulk Cu/Zn/Pb)

There are 5 CalFactor sets, four of which you can edit. Initially, before you edit them, CalFactor sets 1 through 4 are copies of the Factory setting, which is itself not editable. Select the set you want to edit, then select the EDIT button. An information screen pops up briefly before the editing screen appears.

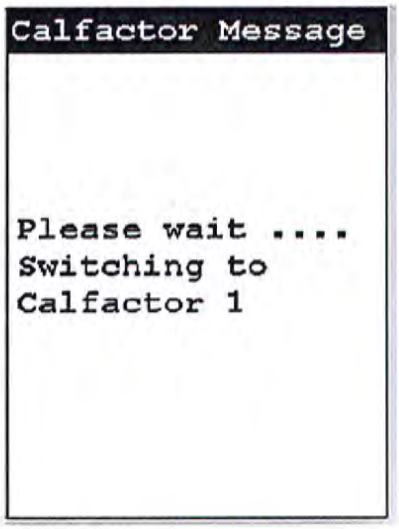


Figure 2-50. Information Screen

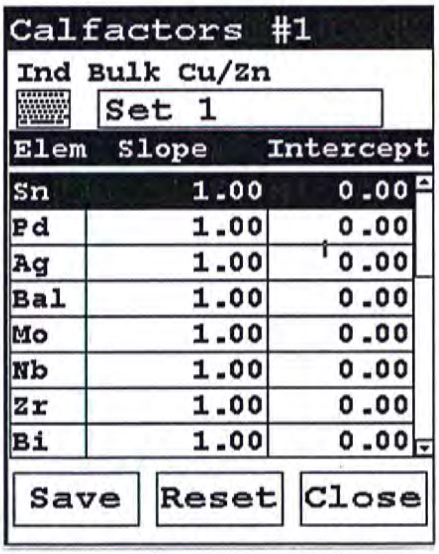


Figure 2-51. CalFactors Edit Screen

The Calibration Factors Edit Screen

To edit the element you are interested in, select the intersection of the row of that element with the Slope or Intercept columns - for example, if you want to edit the slope of Selenium, select the 1.00 default slope appearing in the Sn row. A Slope Editor for that element will appear.

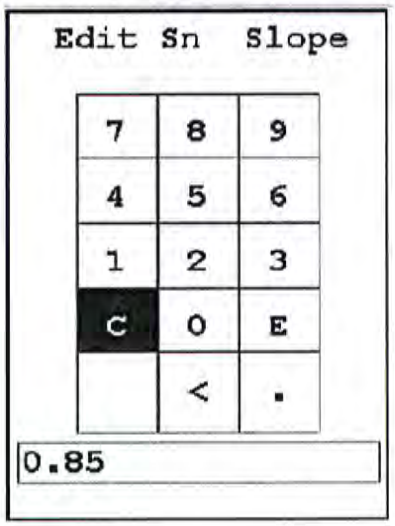


Figure 2-52. Slope Editor (Selenium)

The Slope Editor is a virtual numeric keypad, which works much like the login keypad. Clear the default or old data from the box with the "C" key, select the numbers to input the new slope, and select the "E" key to save the new value. You will be returned to the CalFactors Screen, and the new slope will be there.

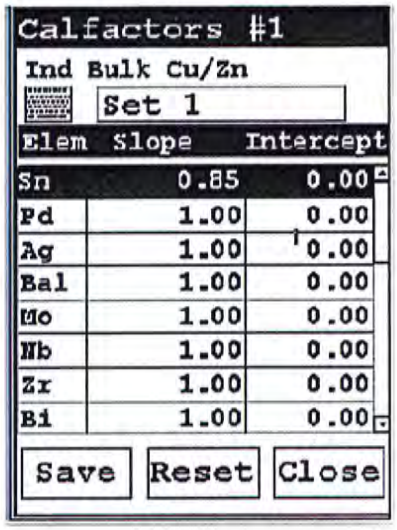


Figure 2-53. New Slope as Edited

Like the Slope Editor, the Intercept Editor is a virtual numeric keypad, which works much like the login keypad. Clear the default or old data from the box with the "C" key, select the numbers to input the new intercept, and select the "E" key to save the new value. You will be returned to the CalFactors Screen, and the new intercept will be there.

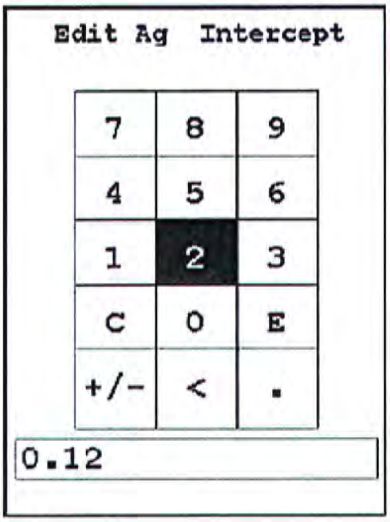


Figure 2-54. Intercept Editor

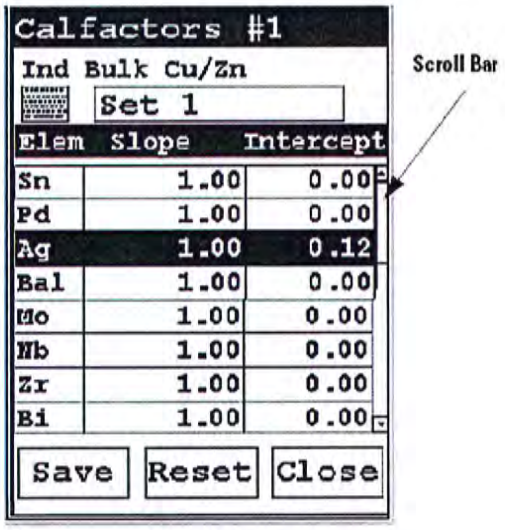


Figure 2-55. Intercept as Edited

- You can use the scroll bar to mover around in the list of elements.

- Selecting the Save button will save the new CalFactor set.
- Selecting the Reset Button will copy the Factory Setting over the current setting, resetting the CalFactors back to Factory Standard.
- Selecting the Close button will exit the CalFactors Edit screen and return you to the CalFactors screen.

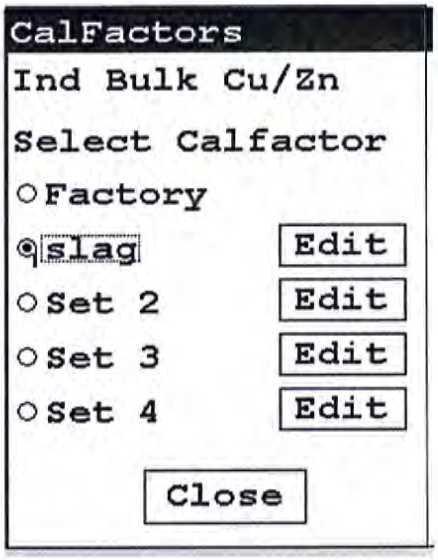


Figure 2-56. Renamed CalFactor Set

To change the name of the set, select the keyboard icon to load the Virtual Keyboard at the top of the CalFactors Edit Screen, and rename the set. Set names can be up to 9 characters long.

Adjusting the Calibration Factors

Calibration factors are used to adjust for consistently high or consistently low readings from the XRF Analyzer.

They are calculated by graphing the data points created by plotting the percentage of each element as indicated by standard samples, against the percentage of each element as reported by the XRF Analyzer.

Then the linear regression function is used to calculate the slope and intercept for a straight line that best fits these data points. The slope and intercept of this line are the calibration factors.

Calculating calibration factors requires several steps:

1. **Resetting the Calibration Factors**
2. **Measuring Standard Samples**
3. **Calculating Calibration Factors**
4. **Adding the Calibration Factors to the XRF Analyzer**

These steps are described in detail below.

Resetting the Calibration Factors

To reset the calibration factors to the defaults:

1. **Display the calibration factors. See "Displaying the Calibration Factors" on page 2-48 for detailed instructions.**
2. **Press the Escape button to switch to the Calibration Factors Menu.**
3. **Press RES on the Calibration Factors Menu. The XRF Analyzer resets the calibration factors to the defaults.**

Measuring the Standard Samples

Use the XRF Analyzer to take readings of samples for which you already know the composition of the sample. It is important that the known composition of these samples be accurate. These samples provide the baseline to which the XRF Analyzer is adjusted. If the known composition is inaccurate, the XRF Analyzer will also be inaccurate.

For each sample, take a reading of at least 120 seconds. Make a note of the reading numbers for these samples.

For each sample, the XRF Analyzer reports the percentage by weight for the elements present in the sample. With the calibration factors set to the defaults, these percentages will differ from the known percentages, but are used to calculate the calibration factors.

Note Readings from only three samples are required for each element, but increasing the number of readings also increases the accuracy of the results.

Calculating Calibration Factors

Using the data that you collected by measuring the standard samples, you now need to plot the percentage of each element as indicated by the standard samples, against the percentage of each element as reported by the XRF Analyzer. Then use the linear regression function to calculate the slope and intercept for a straight line drawn through a graph of those data points.

The slope and intercept for this line are the calibration factors for the element.

You may use any tools that you prefer to make this calculation. This document shows one possible method, using Excel.

Note The XRF Analyzer reports percentages of elements, and the calibration factors are based on percentages of elements. If your standard samples indicate percentages of oxides, see "Oxide Conversion" on page 2-63 to convert percentages of oxides to percentages of elements.

Calculating Calibration Factors Using Excel

Note NITON also provides CorrectCalc™, an Excel-based software program that calculates the calibration factors. It also provides some additional calculations and a graph of your data. For more information, see the CorrectCalc Manual, which is provided as a PDF file on the NDT Software CD.

To calculate calibration factors:

1. **Open Excel. To do this:**
   a. Click the Start button.
   b. Select Programs.

c. Click Microsoft Excel.

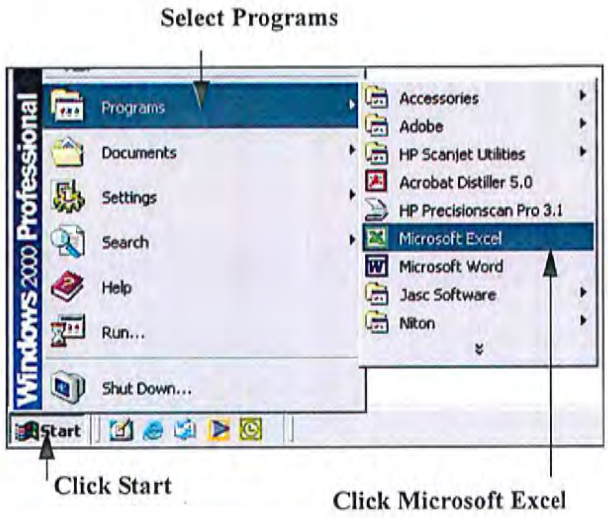


Figure 2-57. Starting Excel

1. **In the first column, enter all the percentages by weight for the element as reported by the XRF Analyzer**

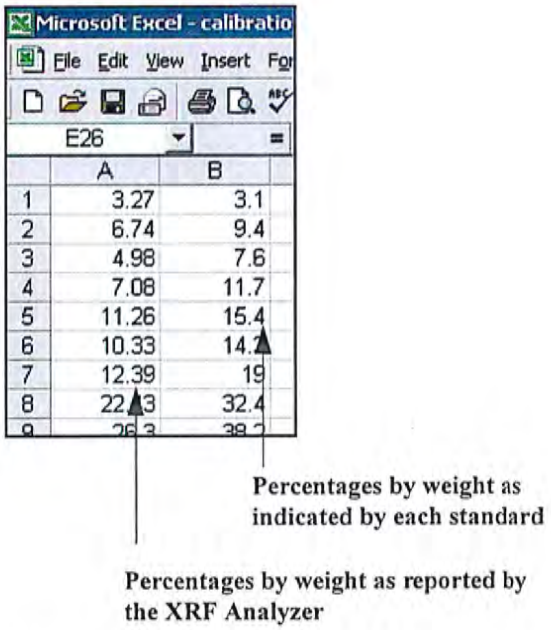


Figure 2-58. Adding Data

2. In the second column, enter all the percentages by weight for the element as indicated by each standard.

3. Use the cursor to highlight all the numbers in both columns.

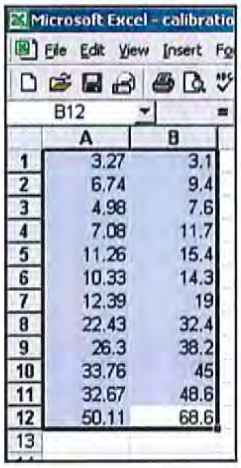


Figure 2-59. Setting Up the Chart

4. Click Chart Wizard.

5. Select the Scatter Chart with data points that are not connected by lines.

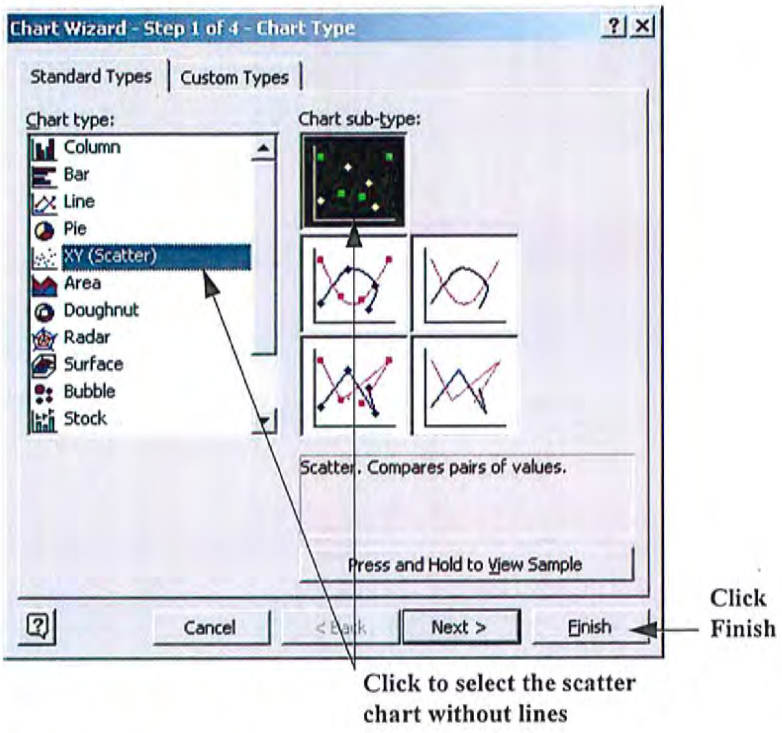


Figure 2-60. Selecting the Scatter Chart

6. **Click Finish.**

7. **Right-click on one of the data points.**

8. **Click Add Trendline on the pop-up menu.**

Figure 2-61. Adding the Trendline

Note If you do not have Add Trendline as an option on this menu, see "Adding the Analysis ToolPak to Excel:" on page 2-64 for instructions.

a. On the Type tab, click Linear.

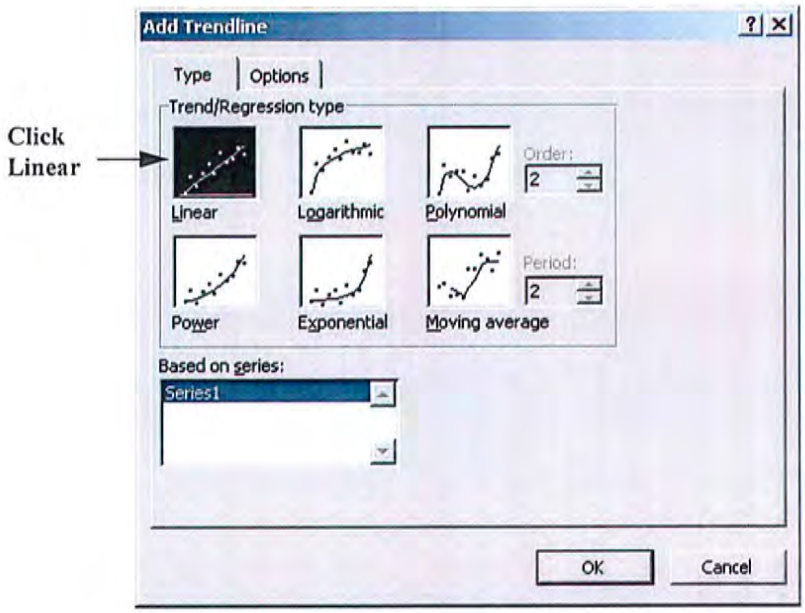


Figure 2-62. Selecting Linear Trendline

b. On the Options tab, check the boxes for "Display equation on chart" and "Display R-squared value on chart."

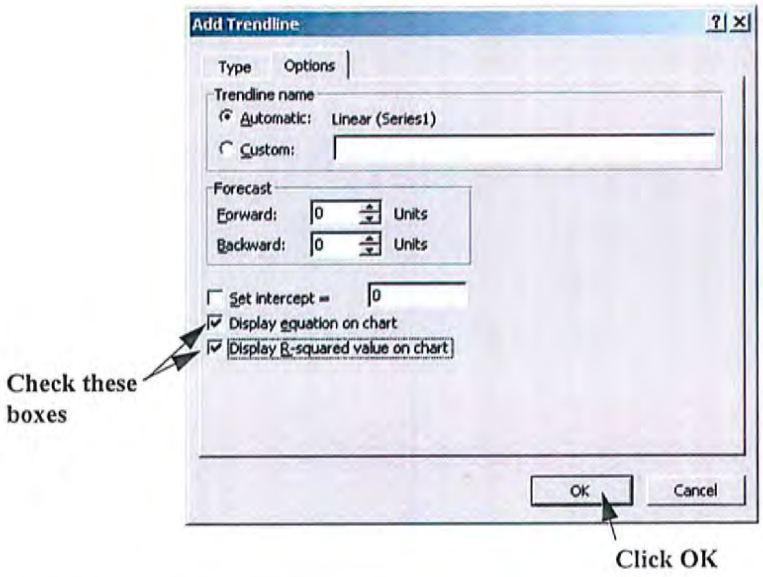


Figure 2-63. Selecting Options

c. Click OK.

d. The equation shows the slope and intercept for the trend line. These are the calibration factors that you enter into the XRF Analyzer.

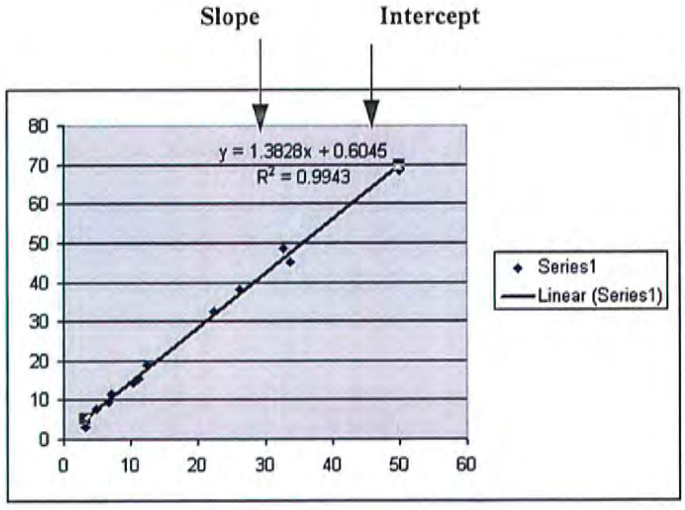


Figure 2-64. Slope and Intercept Displayed

Note If the intercept is negative in the equation, be sure that you enter the intercept as negative in the XRF Analyzer.

9. **Repeat these steps to find the slope and intercept for every element that you are interested in.**

Oxide Conversion

If the composition of your standard sample is specified in percentage of oxide, multiply that percentage by the conversion factor in the table to find the percentage of the element. For example, if the standard sample contains 4% TiO_2, it contains 2.398% Ti:

4 * 0.5995 = 2.398

Table 2-1. Table of Oxide Percentages

Oxide	Conversion Factor
TiO_2	0.5995
V_2O_5	0.5602
Cr_2O_3	0.6842
Mn_3O_4	0.7203
MnO	0.7745
Fe_2O_3	0.6994
FeO	0.7773
Co_3O_4	0.7342
NiO	0.7858
CuO	0.7988
ZnO	0.8034
PbO	0.9283
Fe_2O_3	0.6994
Bi_2O_3	0.8970
ZrO_2	0.7403
MoO_3	0.6665
WO_3	0.7930
Ta_2O_5	0.8190
Nb_2O_5	0.6990
SnO_2	0.7876

If your standard sample contains a compound that is not listed in the table, you can calculate the percentage of the element using this formula:

$$\frac{\text{(molecular weight of the element)}}{\text{(molecular weight of the compound)}} * \text{(\% of compound)} = \text{(\% of element)}$$

Adding the Analysis ToolPak to Excel:

1. **Within Excel, select Add-Ins from the Tools menu.**

Figure 2-65. Selecting Add-Ins

2. **Click the box for Analysis ToolPak**

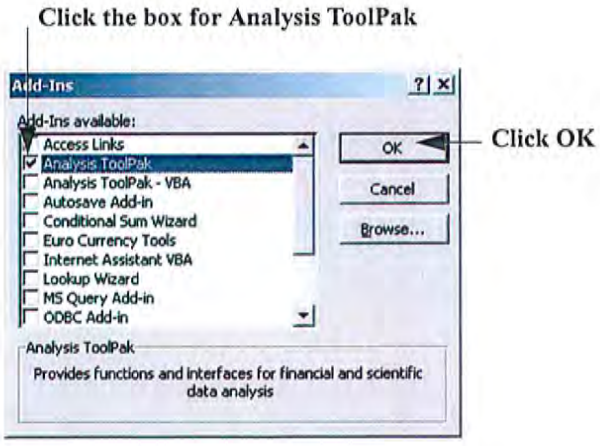


Figure 2-66. Selecting Analysis ToolPak

3. **Click OK.**

Note If you need more information about the Analysis ToolPak, see the documentation for Excel.

Certified Values for Bulk Standards

The three NIST soil samples included with your bulk testing kit (Low, Medium, and High) are certified to the following specifications in these selected elements:

Table 2-2. Low Standard - NIST #2709 (Discontinued)

Element	Mass Fraction %	Variation %	Element	Mass Fraction µg/g	Variation µg/g
Aluminum	7.50	± 0.06	Antimony	7.9	± 0.6
Calcium	1.89	± 0.05	Arsenic	17.7	± 0.8
Iron	3.50	± 0.11	Barium	968.0	± 40
Magnesium	1.51	± 0.05	Cadmium	0.38	± 0.01
Phosphorus	0.062	± 0.005	Chromium	130.0	± 4.0
Potassium	2.03	± 0.06	Cobalt	13.4	± 0.7
Silicon	29.66	± 0.23	Copper	34.6	± 0.7
Sodium	1.16	± 0.03	Lead	18.9	± 0.5
Sulphur	0.089	± 0.002	Manganese	538.0	± 17.0
Titanium	0.342	± 0.024	Mercury	1.40	± 0.08
		± Nickel		88.0	± 5.0
		± Sel	enium	1.57	± 0.08
		± Si	lver	0.41	± 0.03
		± S	trontium	231.0	± 2.0
		+	Thallium	0.74	± 0.05
		± V	anadium	112.0	± 5.0
			Zinc	106.0	± 3.0

The complete Certificate of Analysis can be obtained from the NIST web site at: http://patapasco.nist.gov/smcatalog/certificates/2709.pdf

Table 2-3. Non-Certified Values - NIST Standard 2709 (Discontinued

Element	Mass Fraction %	Element	Mass Fraction µg/g
Carbon	1.2	Cerium	42.0
		Cesium	5.3
		Dysprosium	3.5
		Europium	0.9
		Gallium	14.0
		Gold	0.3
		Hafnium	3.7
		Holmium	0.54
		Iodine	5.0
		Lanthanum	23.0
		Molybdenum	2.0
		Neodymium	19.0
		Rubidium	96.0
		Samarium	3.8
		Scandium	12.0
		Thorium	11.0
		Tungsten	2.0
		Uranium	3.0
		Ytterbium	1.6
		Yttrium	18.0
		Zirconium	160.0

Table 2-4. Medium Standard - NIST # 2711 (Discontinued)

Element	Mass Fraction %	Variation %	Element	Mass Fraction µg/g	Variation µg/g
Aluminum	6.53	± 0.09	Antimony	19.4	± 1.8
Calcium	2.88	± 0.08	Arsenic	105.0	± 8.0
Iron	2.89	± 0.06	Barium	726.0	± 38.0
Magnesium	1.05	± 0.03	Cadmium	41.7	± 0.25
Phosphorus	0.086	± 0.007	Copper	114.0	± 2.0
Potassium	2.45	± 0.08	Lead	1162.0	± 31.0
Silicon	30.44	± 0.19	Manganese	638	± 28.0
Sodium	1.14	± 0.03	Mercury	6.25	± 0.19
Sulphur	0.042	± 0.001	Nickel	20.6	± 1.1
Titanium	0.306	± 0.023	Selenium	1.52	± 0.14
		± Si	lver	4.63	± 0.39
		± S	trontium	245.3	± 0.7
		± Tha	llium	2.47	± 0.15
		± V	anadium	81.6	2.9
		+	Zinc	350.4	4.8

The complete Certificate of Analysis can be obtained from the NIST web site at: http://patapasco.nist.gov/smcatalog/certificates/2711.pdf

Table 2-5. Non-Certified Values - NIST Standard 2711 (Discontinued)

Element	Mass Fraction %	Element	Mass Fraction µg/g
Carbon	2.0	Bromine	5.0
		Cerium	69.0
		Cesium	6.1
		Chromium	47.0
		Cobalt	10.0
		Dysprosium	5.6
		Europium	1.1
		Gallium	15.0
		Gold	0.03
		Hafnium	7.3
		Holmium	1.0
		Indium	1.1
		Iodine	3.0
		Lanthanum	40.0
		Molybdenum	1.6
		Neodymium	31.0
		Rubidium	110.0
		Samarium	5.9
		Scandium	9.0
		Thorium	14.0
		Tungsten	3.0
		Uranium	2.6
		Ytterbium	2.7
		Yttrium	25.0
		Zirconium	230.0

Table 2-6. High Standard - NIST # 2710

Element	Mass Fraction %	Variation %	Element	Mass Fraction µg/g	Variation µg/g
Aluminum	6.44	± 0.08	Antimony	38.4	± 3.0
Calcium	1.25	± 0.03	Arsenic	626.0	± 38.0
Iron	3.38	± 0.10	Barium	707.0	± 51.0
Magnesium	0.853	± 0.042	Cadmium	21.8	± 0.2
Manganese	1.01	± 0.04	Copper	2950.0	± 130.0
Phosphorus	0.106	± 0.015	Lead	5532.0	± 80.0
Potassium	2.11	± 0.11	Mercury	32.6	± 1.8
Silicon	28.97	± 0.18	Nickel	14.3	± 1.0
Sodium	1.14	± 0.06	Silver	35.3	± 1.5
Sulfur	0.240	± 0.006	Vanadium	76.6	± 2.3
Titanium	0.283	± 0.010	Zinc	6952.0	± 91.0

The complete Certificate of Analysis can be obtained from the NIST web site at: http://patapasco.nist.gov/smcatalog/certificates/2710.pdf

Table 2-7. Non-Certified Values - NIST Standard 2710

Element	Mass Fraction %	Element	Mass Fraction µg/g
Carbon	1	Bromine	6
		Cerium	57
		Cesium	107
		Chromium	39
		Cobalt	10
		Dysprosium	5.4
		Europium	1
		Gallium	34
		Gold	0.6
		Hafnium	3.2
		Holmium	0.6
		Indium	5.1
		Lanthanum	34
		Molybdenum	19
		Neodymium	23
		Rubidium	120
		Samarium	7.8
		Scandium	8.7
		Strontium	330
		Thallium	1.3
		Thorium	13
		Tungsten	93
		Uranium	25
		Ytterbium	1.3
		Yttrium	23

Table 2-8. NITON Analyzers Soil Standards Substitutions Comparisons

Element	NCS DC 73308	NIST Low	Element	TILL-4	NIST Medium
Ag	0.27 +/- 0.02	0.41 +/- 0.03	Ag		4.6 +/- 0.4
As	25 +/- 3	17.7 +/- 0.8	As	111	105 +/- 8
Au (0.3)			Au	5 ppb	(0.03)
Ba	42 +/- 7	968.0 +/- 40.0	Ba	395	726 +/- 38
Be	0.9 +/- 0.2		Be	3.7	
Bi	0.38 +/- 0.04		Bi	40	
Br	2.4 +/- 0.5		Br	8.6	(5)
Ca (%)	0.28	1.89 +/- 0.05	Ca (%)	0.89	2.88 +/- 0.08
Cd	1.1	0.38 +/- 0.01	Cd		41.7 +/- 0.25
Ce	38 +/- 4	(42)	Ce	78	(69)
Co	15.3 +/- 1.1	13.40 +/- 0.70	Co	8	(10)
Cr	136 +/- 10	130.00 +/- 4.00	Cr	53	(47)
Cs	2.3 +/- 0.5	(5.3)	Cs	12	(6.1)
Cu	22.6 +/- 1.3	34.6 +/- 0.7	Cu	237	114.0 +/- 2.0
Eu	0.47 +/- 0.04	(0.9)	Eu	<1.0	(1)
Er	1.3 +/- 0.2		Er	3.2	
Fe (%)	2.7	3.50 +/- 0.11	Fe (%)	3.97	2.89 +/- 0.06
Ga	6.4 +/- 0.7	(14)	Ga		(15)
Gd	2.2 +/- 0.2		Gd		
Ge	0.40 +/- 0.06		Ge		
Hf	1.8 +/- 0.4	(3.7)	Hf	10	(7.3)
Hg	0.28 +/- 0.03	1.4 +/- 0.08	Hg		6.3 +/- 0.2
I	1.6 +/- 0.3		I		
In	0.067 +/- 0.016		In		(1.1)
K (%)	1041 ppm	2.03 +/- 0.06	K (%)	2.70	2.45 +/- 0.08
La	13.0 +/- 0.9	(23)	La	41	(40)
Li	13.0 +/- 0.5		Li	30	
Mn	1010 +/- 29	538.0 +/- 17.0	Mn	490	638 +/- 28
Mo 1.2	+/- 0.1	(2.0)	Mo 16		(1.6)
Nb	6.8 +/- 1.3		Nb	15	

Table 2-8. NITON Analyzers Soil Standards Substitutions Comparisons

Element	NCS DC 73308	NIST Low	Element	TILL-4	NIST Medium
Nd	11.8 +/- 1.1		Nd	30	
Ni	30 +/- 2	88.0 +/- 5.0	Ni	17	20.6 +/- 1.1
P	271 +/- 15	0.062% +/- 0.005%	P	880	0.086% +/- 0.007%
Pb	27 +/- 2	18.9 +/- 0.5	Pb	50	1162 +/- 31
Rb	9.2 +/- 1.5	(96)	Rb	161	(110)
S (%)	(90 mg/kg)	0.089 +/- 0.002	S (%)	0.08	0.042 +/- 0.001
Sb	6.3 +/- 0.6	7.9 +/- 0.6	Sb	1	19.4 +/- 1.8
Sc	4.1 +/- 0.4	(12.0)	Sc	10	(9.0)
Se		1.57 +/- 0.08	Se		1.5 +/- 0.1
Si (%)	41.55	29.66 +/- 0.23	Si		
Sm	2.4 +/- 0.2		Sm	6.1	
Sr	25 +/- 3	231.0 +/- 2.0	Sr	109	245.3 +/- 0.7
Ta	0.44 +/- 0.12		Ta	1.6	
Tb	0.42 +/- 0.07		Tb	1.1	
Th	5.0 +/- 0.3	(11.0)	Th	17.4	(14.0)
Ti (%)	0.127 +/- 0.007	0.342 +/- 0.024	Ti (%)	0.484	0.306 +/- 0.023
U	2.1 +/- 0.2	(3.0)	U	5	(2.6)
V	107 +/- 5	112.0 +/- 5.0	V	67	81.6 +/- 2.9
W	1.6 +/- 0.3	(2.0)	W	204	(3.0)
Y	14 +/- 2	(18.0)	Y	33	(25.0)
Yb	1.2 +/- 0.2		Yb	3.4	
Zn	46 +/- 4	106.0 +/- 3.0	Zn	70	350.4 +/- 4.8
Zr	70 +/- 6	(160.0)	Zr	385	(230.0)

values in mg/kg unless otherwise specified

uncertified values in parentheses

Replacement Standards for Discontinued NIST Samples

Table 2-9. Low Standard - NCS DC 73308 (Replaces NIST 2709)

Element	Mass Fraction µg/g	Variation µg/g
Ag	0.27	± 0.02
As	25	± 3.
Au		±
B	26	±4
Ba	42	±7
Be	0.9	± 0.2
Bi	0.38	± 0.04
Br	2.4	± 0.5
Cd	1.12	± 0.08
Ce	38	± 4
Cl	[53]	±
Co	15.3	± 1.1
Cr	136	± 10
Cs	2.3	±0 .5
Cu	22.6	+1.3
Dy	2.2	± 0.3
Er	1.3	± 0.2
Eu	0.47	± 0.04
F	149	± 25
Ga	6.4	± 0.7
Gd	2.2	± 0.2
Ge	0.40	± 0.06
Hf	1.8	± 0.4
Hg	0.28	± 0.03
Ho	0.45	± 0.07
I	1.6	± 0.3
In	0.067	± 0.016
La	13.0	± 0.9
Li	13.0	± 0.5
Lu	0.19	± 0.03
Mn	0.41010	± 29

Table 2-9. Low Standard - NCS DC 73308 (Replaces NIST 2709)

Element	Mass Fraction µg/g	Variation µg/g
Mo	1.2	± 0.1
N	[360]	±
Nb	6.8	± 1.3
Nd	11.8	± 1.1
Ni	30	± 2
P	271	± 15
Pb	27	± 2
Pr	3.2	± 0.4
Rb	9.2	± 1.5
S	[90]	±
Sb	6.3	± 0.6
Sc	4.1	± 0.4
Se	0.28	± 0.05
Sm	2.4	± 0.2
Sn	1.4	± 0.3
Sr	25	± 3
Ta	0.44	± 0.12
Tb	0.42	± 0.07
Te	0.08	± 0.02
Th	5.0	± 0.3
Ti	1270	± 70
Tl	0.21	± 0.05
Tm	0.20	± 0.03
U	2.1	± 0.2
V	107	± 5
W	1.6	± 0.3
Y	14	± 2
Yb	1.2	± 0.2
Zn	46	± 4
Zr	70	± 6

Data enclosed in brackets [x.y] are reference values.

Table 2-10. Medium Standard - TILL-4 (Replaces NIST 2711)

Element	Mass Fraction %	Element	Mass Fraction µg/g
Fe	3.97	As	111
LOI (500°C)	4.4	Au	0.005
S	0.08	Ba	395
		Be	3.7
		Bi	40
		Br	8.6
		Ce	78
		Co	8
		Cr	53
		Cs	12
		Cu	237
		Eu	<1.0
		Er	3.2
		Hf	10
		La	41
		Li	30
		Lu	0.5
		Mn	490
		Mo	16
		Nb	15
		Nd	30
		Ni	17
		P	880
		Pb	50
		Rb	161
		Sb	1.0
		Sc	10
		Sm	6.1
		Sr	109
		Ta	1.6
		Tb	1.1

Table 2-10. Medium Standard - TILL-4 (Replaces NIST 2711)

Element	Mass Fraction %	Element	Mass Fraction µg/g
		Th	17.4
		Ti	4840
		U	5.0
		V	67
		W	204
		Y	33
		Yb	3.4
		Zn	70
		Zr	385

Chapter 3 Alloy Testing

Using Alloy Testing Mode

Alloy Testing Mode is not enabled on your NITON XLp analyzer. For more information on this feature please contact NITON's Customer Service Department in the United States, Toll free, at (800) 875-1578, or outside the United States at + 1-978-670-7460 or your authorized NITON Service Center.

Chapter 4 Pb Paint Mode

Using Pb Paint Mode

Your NITON XLp-300 Series Lead-in-Paint Analyzer is designed to detect and quantify the amount of lead present in painted surfaces. Your analyzer also tracks important information about your site readings using the integrated bar-code scanner or the analyzer's control panel.

Lead Paint inspection guidelines and protocols are specific to the country and state in which the testing is being performed. For the HUD testing guidelines and the NITON Analyzer Performance Characteristic Sheet (PCS), please go to www.hud.gov

The Pb Paint Mode Menu

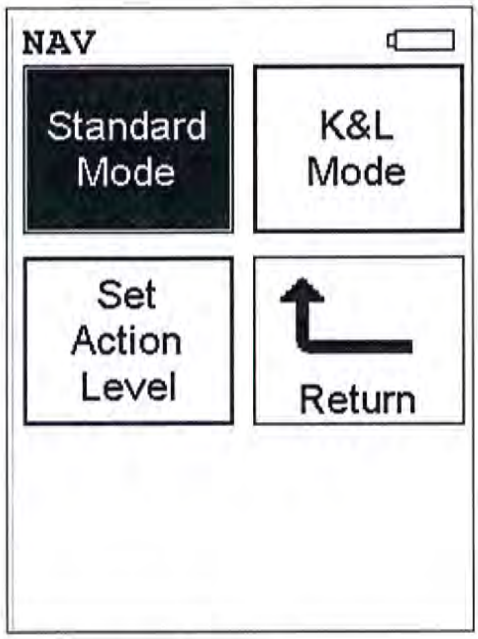
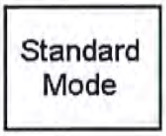
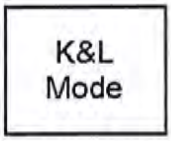


Figure 4-1. The Pb Paint Mode Menu

All NITON XLp-300 Series Lead-in-Paint Analyzer Mode functions are accessible from the Pb Paint Mode Menu and subsidiary menus. Each of the instrument functions represented by an icon on the Pb Paint Mode Menu screen may be selected by choosing the appropriate icon. When one of these Pb Paint Mode Menu icons is selected, the function specific sub-menu appropriate to that icon will be displayed. All Lead-In-Paint modes use both L and K shell analysis simultaneously.



The Standard Mode icon allows you to select the Lead-in-Paint Standard Mode. Standard Mode is a qualitative analysis designed for 95% confidence level as to whether the sample is above or below the Action Level. This mode tends to give very fast readings. as it terminates the test as soon as 95% confidence has been achieved.



The K+L Mode icon allows you to select the Lead-in-Paint K+L Mode. K+L Mode is a quantitative analysis which allows you to determine the statistical confidence of the reading to a 95% Confidence Level while allowing you the flexibility of continuing the test for as long as you wish up to the (user-definable) maximum test time.

The Set Action Level icon allows you to set the action level for Lead-in-Paint to match the official action level of your locality.

The Standard Mode Menu

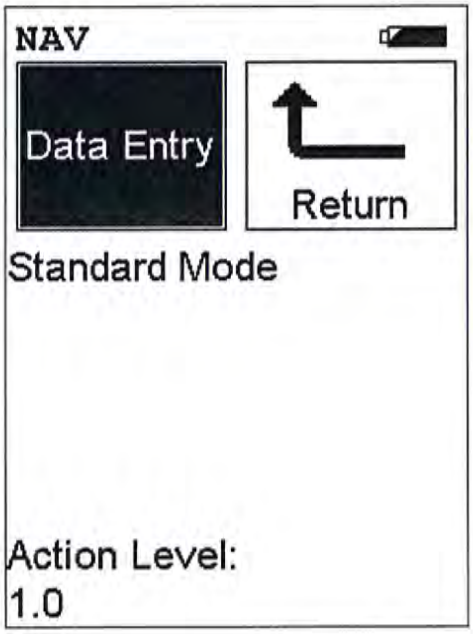


Figure 4-2. The Standard Mode Menu

All NITON XLp Standard Mode functions are accessible from the Standard Mode Menu and subsidiary sub-menus. From the **Standard Mode Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **Standard Mode Menu**to go to the "The Data Entry Screens" on page 4-10, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

The K+L Mode Menu

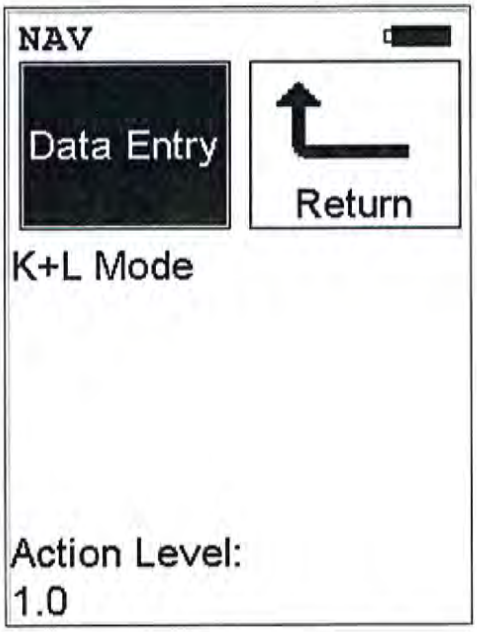


Figure 4-3. The K+L Mode menu

All NITON XLp K+L Mode functions are accessible from the K+L Mode Menu and subsidiary sub-menus. From the **K+L Mode Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **K+L Mode Menu**to go to the "The Data Entry Screens" on page 4-10, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

The Set Action Level Screen

The Set Action Level Screen is accessible from the Paint Mode Menu. When the Set Action Level icon is selected, the Set Action Level Screen will be displayed.

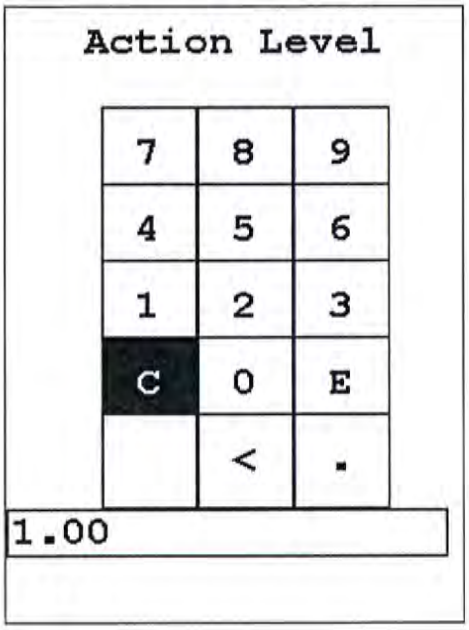


Figure 4-4. The Set Action Level Screen

To change the preset Action Level to match the Action Level set by your locality, select the "C" key from the Virtual Numeric Keypad to clear the current Action Level value, select the new Action Level value using the numeric keys, then select "E" to enter the new Action Level value. The Action Level will be changed to the new value.

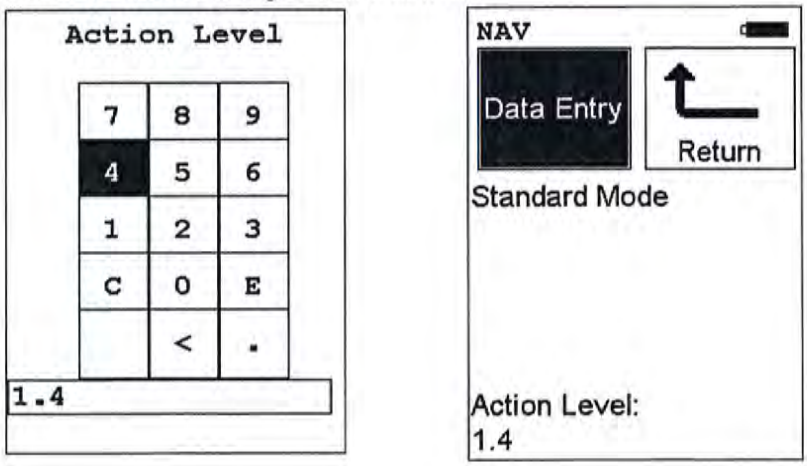


Figure 4-5. Changing the Action Level

Taking a Reading

CAUTION After being powered on, the NITON 300 Series Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your instrument warm up for ten minutes after start up, before testing is begun. ♦

There are five different methods of operation for taking a sample measurement, and your analyzer will be configured to use one of those methods, depending on the regulatory requirements of your locality. These methods are:

- Trigger-Only method. With the Trigger-Only method, you only need to place the measurement window close to the sample to be analyzed and pull the trigger for sample analysis to be initiated.

- Trigger-and-Proximity-Sensor method. With the Trigger-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the instrument, then pull the trigger for sample analysis to be initiated.

- Momentary-Trigger-Touch-and-Proximity-Sensor method. With the Momentary-Trigger-Touch-and-Proximity-Sensor method, you must place the measurement window against the surface to be analyzed to engage the proximity sensor on the front of the instrument, then pull the trigger.

- The trigger may be released and the reading will continue until you release the proximity button, or other criteria (such as Max Time) are reached.

- Trigger-and-Interlock method. With the Trigger-and-Interlock method, you need to place the measurement window close to the sample to be analyzed, press and keep pressing the interlock button at the rear of the instrument with your free hand, then pull the trigger for sample analysis to be initiated.

- Trigger-Interlock-and-Proximity-Sensor method. With the Trigger-Interlock-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the instrument, press and keep pressing the interlock button at the rear of the instrument with your free hand, then pull the trigger for sample analysis to be initiated.

With any of these methods, analysis will stop if any one of the preconditions are violated. For example, with the Trigger-Interlock-and-Proximity-Sensor method, if the trigger or the Proximity Sensor or the Interlock is released, the reading will stop immediately, and the shutters will close.

After your NITON XLp analyzer is calibrated, initiate a sample reading using the appropriate method. If you attempt to initiate a sample reading using a different method, the analyzer will inform you that one or more of the preconditions need to be met in order for sample analysis to begin.

Note The three LED lights will blink during calibration or whenever there is a shutter open. ♦

WARNING! The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, all the shutters will close, and the measurement will end. The three LED lights will stop blinking, the shutters will close, and the measurement will end. The flashing of the LED lights is not synchronized to minimize power consumption. ♦

WARNING! The three LED warning lights are designed to blink only during a measurement, where one or more of the shutters are open and the trigger depressed. If the LED lights blink at any other time, disconnect the battery pack immediately, place the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, or your local Authorized NITON Analyzers Service Center. ♦

Your NITON Analyzer will display the Results Screen throughout the duration of each reading, The Results Screen is updated regularly throughout the reading. When the reading is complete, a final screen update will appear, and your NITON analyzer will display the final results of the measurement which has just been completed.

Standard Mode Results Screen

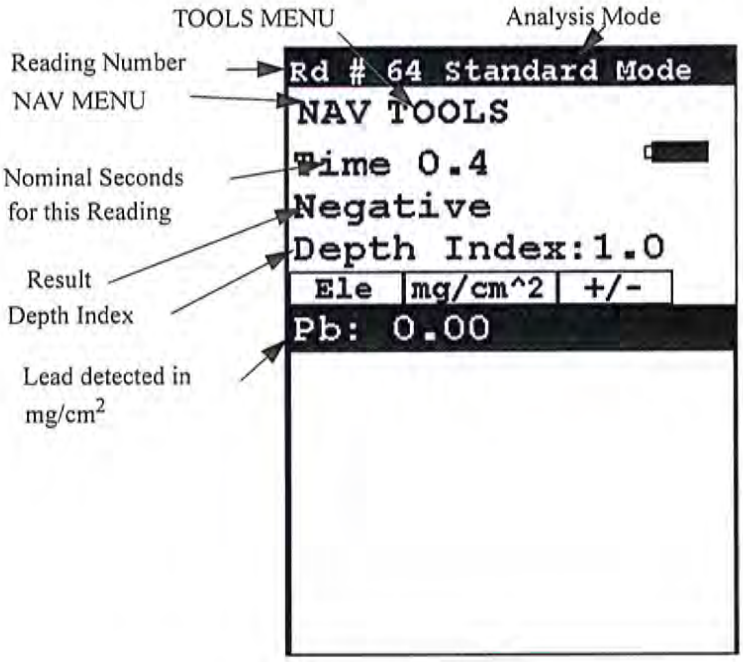


Figure 4-6. Standard Mode Results Screen

Before the analyzer has reached a determination, the result will be shown as "Inconclusive". Your analyzer will beep twice when a result is reached then terminate the reading. The display will change from "Inconclusive" to show the result, either "Positive" for lead detected above the action level, or "Negative" for no lead or lead below the action level. It will also display the amount of lead detected in mg/cm^2.

The Depth Index is a numerical value indicating the amount of non-leaded paint covering the lead (if any) detected by the analyzer.

- A Depth Index of less than 1.5 indicates a reading very near the surface.
- A Depth Index between 1.6 and 4.0 indicates a moderate depth.
- A Depth Index of greater than 4.0 indicates a deeply buried reading.

K+L Mode Results Screen

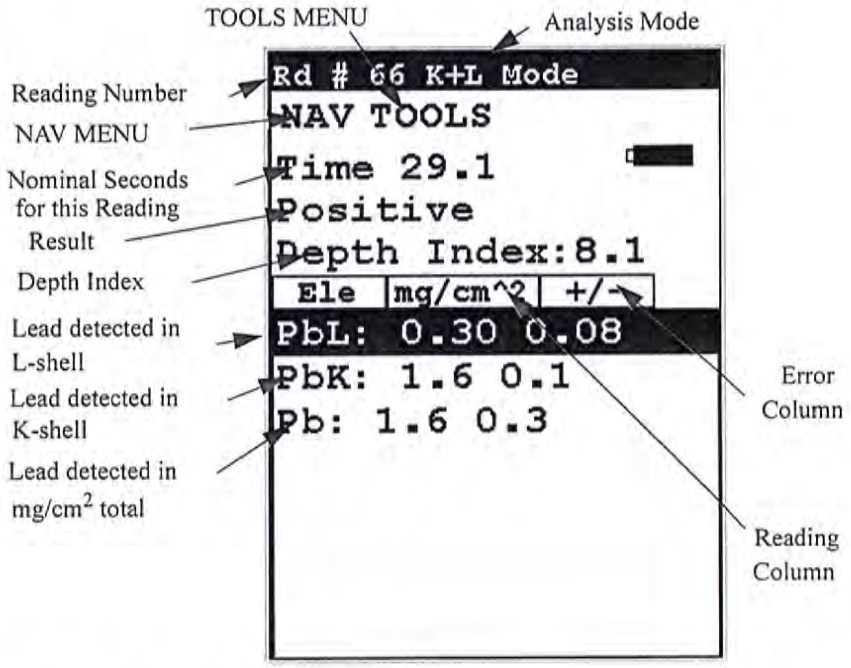


Figure 4-7. K+L Mode Results Screen

The K+L Mode Results Screen will show the same basic display throughout the reading. Your analyzer will beep twice to indicate it has reached a conclusion, and the Result field will change from "Inconclusive" to "Positive" or "Negative", but the reading will continue for as long as you continue to hold down the trigger and depress the Proximity Sensor.

The K+L Results Screen shows the reading results and error for both K and L-shell readings. The "Lead Detected in mg/cm^2 total" field is the result of the judgement of your analyzer as to which reading, K or L-shell, best represents the true condition.

The **Depth Index** is a numerical value indicating the amount of non-leaded paint covering the lead (if any) detected by the analyzer.

- A Depth Index of less than 1.5 indicates a reading very near the surface.
- A Depth Index between 1.6 and 4.0 indicates a moderate depth.
- A Depth Index of greater than 4.0 indicates a deeply buried reading.

The Data Entry Screens

The Data Entry Screens allow you to set the values for various parameters tracked by the system along with the actual analysis results. This screen allows you to input data in several different fields, or categories, concerning your sample, in several different ways:

- By selecting the Virtual Keyboard button and typing the parameter in using the **Virtual Keyboard**.
- By scanning in the parameter name using the integrated bar code scanner.
- By creating a new, or editing your analyzer's existing, '.ndf' file through the NDT program. You can then select from the various custom options you have created using the Drop-down List button.

These fields are saved along with the subsequent reading, and allow you to associate important information about the sample directly with the reading, so that you have a full description of the sample tied into the reading itself.

These parameters all describe the particular test target to be analyzed. The location of the target in the site, the type of target, the surface and substrate, the condition of the surface, and the inspector performing the test are some of the parameters tracked by the analyser.

Navigating the Data Entry Screen

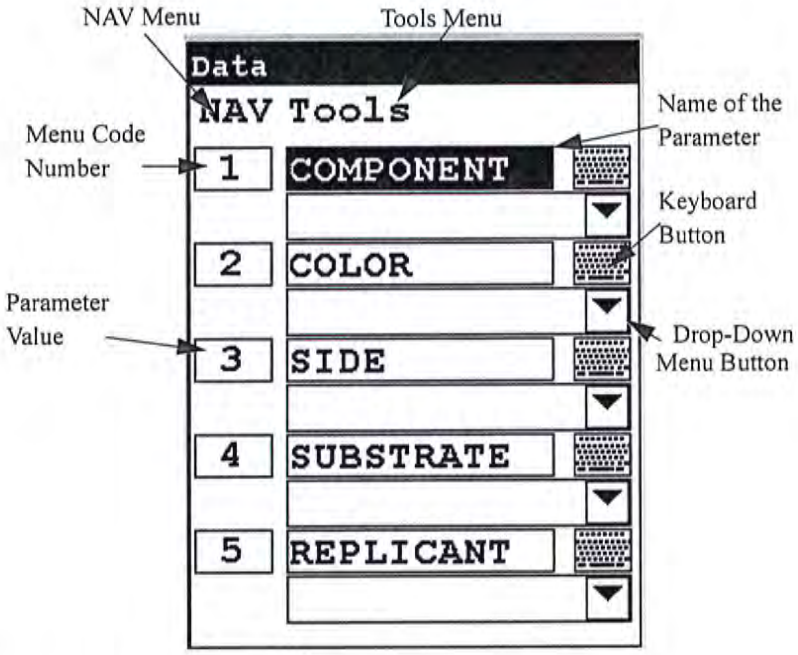


Figure 4-8. Navigating the Data Entry Screen

Above is a sample of the Data Entry Screen. To navigate through the Data Entry Screen and select your parameter settings, you can use the touch screen display or 4-way touch pad and control panel buttons.

Navigating the Data Entry Screen

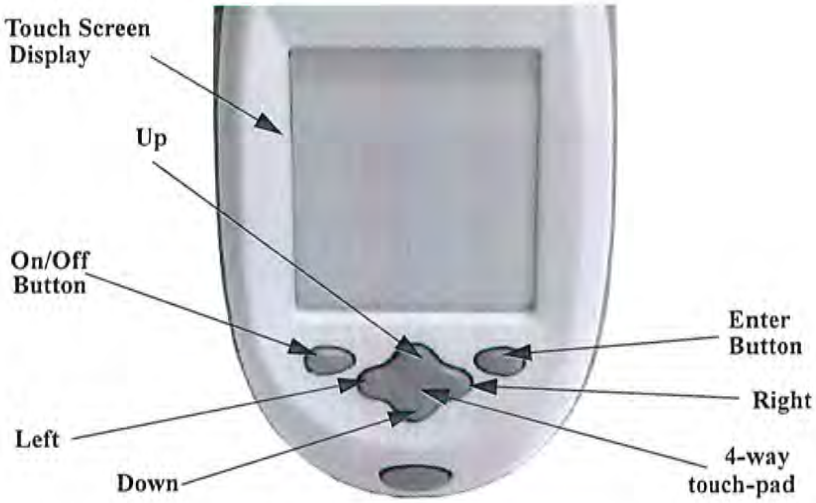


Figure 4-9. The Control Panel

The following description of screen navigation using the control panel assumes that the analyzer is held so that the display is held upright as in Figure 4-9.

- To move from column to column, use the Right and Left portion of the 4-way touch pad.
- To move from row to row, use the Up and Down portions of the 4-way touch pad.
- To select the highlighted option, press the Enter button on the control panel.

The *Data Entry* Screen is divided into sections of 5 setting parameters. By using the Down portion of the 4-way touch pad when you are on the last row of a section, the display will change to the next section. By using the Up portion of the 4-way touch pad when you are on the first row of a section, the display will change to the previous section.

By selecting the *On/Off* button, you can exit the *Data Entry* Screen.

Using The Data Entry Screen

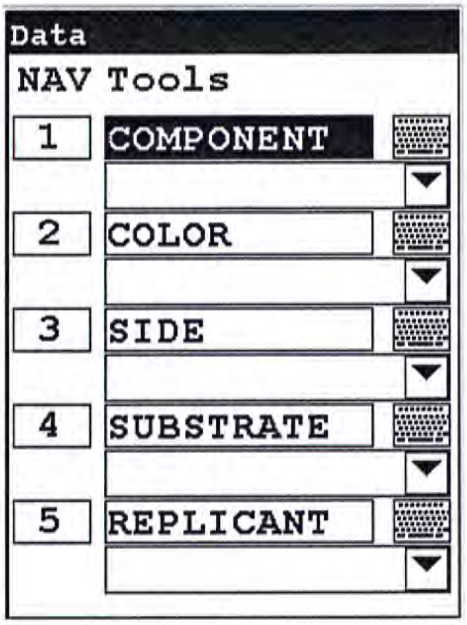


Figure 4-10. The Data Entry Screen - First Section

This is the first section of the Data Entry Screen. There are five parameters in this section.

- Selecting Component allows you to input the reading location Component parameter.
- Selecting Color allows you to input the reading location Color parameter.
- Selecting Side allows you to input the reading location Side parameter.
- Selecting Substrate allows you to input the reading Substrate parameter.
- Selecting Replicant allows you to input the reading Replicant parameter.

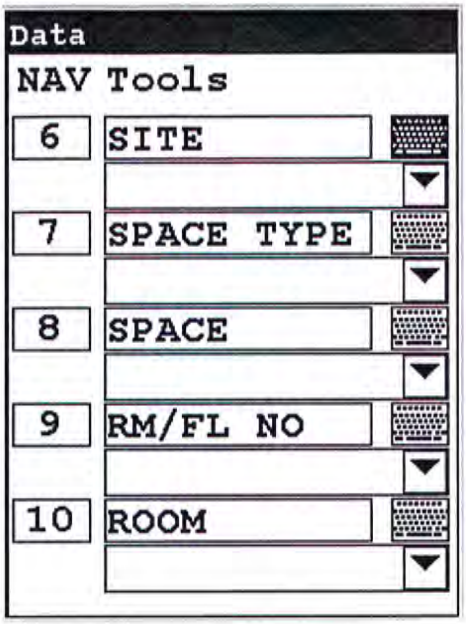


Figure 4-11. The Data Entry Screen - Second Section

This is the second section of the Data Entry Screen. There are five parameters in this section.

- Selecting Site allows you to input the reading Site parameter.
- Selecting Space Type allows you to input reading the reading location Space Type parameter.
- Selecting Space allows you to input the reading location Space parameter.
- Selecting Rm/Fl No allows you to input the reading location Rm/Fl No parameter.
- Selecting Room allows you to input the reading Room parameter.

Figure 4-12. The Data Entry Screen - Third Section

This is the third section of the Data Entry Screen. There is one parameter in this section.

- Selecting Inspector allows you to input the reading Inspector parameter.

- Selecting the Menu Code Number will initiate a bar code scan to input pre-printed bar code parameter values

- Selecting the Drop-Down Menu Button will access the particular Drop-Down Menu for that parameter, allowing you to select the parameter value from a pre-determined list.

- Selecting the Keyboard button allows you to input a parameter value as required using the Virtual Keyboard.

Lead in Consumer Products

Your analyzer is capable of screening toys, children's jewelry, and other consumer products for the presence of Lead (Pb). The primary goal of screening is not to accurately analyze the chemical composition of all components or parts of the product, but to identify and segregate products containing prohibited elements.

Consumer Products SOP

The following is the Standard Operating Procedure (SOP) for finding lead in consumer products.

Preparatory Tasks

1. **Attach a charged battery to the analyzer and turn it on. Follow the screen instructions and Log On as the operator.**

2. **Wait five (5) minutes before using the analyzer, allowing the instrument's electronics to stabilize.**

3. **Verify that the date is set properly for data tracking purposes.**

4. **From the Main Menu, select Utilities -> Instrument Specs. The date will be displayed for verification. To escape back to the Main Menu, press the On/Off/Escape button. If the date is incorrect, correct it prior to proceeding.**

5. **If testing will be performed onsmall samples, mount the analyzer in its optional test stand. Any sample that does not completely cover the analyzer measuring window (aperture) is considered small. Also, samples that have a form of thin sheet, such as foil, paper, fabric, etc., should also be analyzed in a test stand.**

(Optional) Connect the analyzer to a computer via the included serial cable or Bluetooth wireless module. Consult the Guide to NDTr and the Bluetooth Installation Guide document for details, if necessary.

6. **During analysis and detector calibrations, it is important to ensure that the analyzer is not exposed to strong electromagnetic fields, including those produced by computer monitors, hard drives, cellular telephones, walkie talkies, etc. Keep a minimum two (2) feet (0.7 meters) distance between the analyzer and electronic devices.**

7. **From the Main Menu, select Utilities‡ Calibrate -> Calibrate Detector.**

   a. Detector calibration standardizes the detector. After starting the process, no further user interaction is required during this operation. Note the "Res" figure displayed following the detector calibration. This number, usually <500eV, is an evaluation of the detector resolution and should be consistent with past calibration results. This data will download as a stored reading if the user chooses to download readings, so it can be saved for tracking the instrument's performance

   b. If the "Res" number is consistent with past detector calibration results, continue to step 8.

   c. If the "Res" figure varies by more than 20 percent over the average of multiple detector calibrations, move the analyzer away from potential interference sources (as discussed in item 6 above), and repeat the detector calibration steps. If the resolution does not return to its standard range, please contact Thermo Scientific Niton Analyzers toll-free at (800) 875-1578 (US only), +1 978 670-7460 (outside US), via email at niton@thermofisher.com, or your local Niton Analyzer representative for assistance.

Plastics

8. **From the Main Menu, select Mode -> Consumer Goods Mode -> Plastics Mode.**

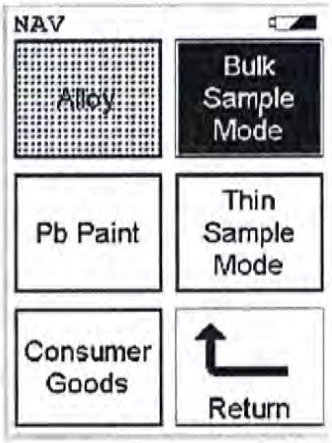


Figure 4-13. Mode Screen

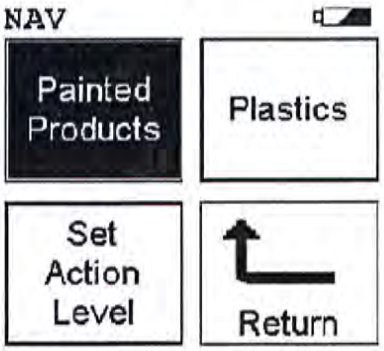


Figure 4-14. Consumer Goods Mode

a. Place the Reference Sample (PN 180-619) in the test stand for analysis, or set the reference sample on a clean surface. The unlabeled side should be presented to the analyzer. Place the front end of the instrument against the sample with the window centered on the sample. Due to the length of this check measurement, it is recommended that a test stand be used. It can be difficult to hold the analyzer steady on the reference sample for the required length of time.

b. Take one to three 60-source second measurements on this reference sample. Ensure that the analyzer has the maximum test time set to 60 (seconds). From the Main Menu, select Common Setup -> Instrument Setup -> Hardware Setup. Edit the Max. Time dialog box to the proper setting. Average the results. Results can be averaged via the TOOLS drop down menu.

c. Compare the averaged results with the acceptable ranges as listed in Table 1. The averaged results should fall within these specified values.

Table 4-1. Plastic "Reference Sample in PE Matrix" Nominal Concentrations and Allowable Ranges

Element	Nominal Concentration* (mg/kg)**	Acceptable Range (±% Rel)	Acceptable Range (mg/kg)
Pb 1	50	15%	127-173

*Please refer to the certificate accompanying the reference sample for actual elemental composition data as the concentration may vary from batch to batch.

** Please note that mg/kg is a unit of measure of concentration equal to ppm by mass, i.e., 1000 ppm is equivalent to 1000 (mg/kg) = 0.1%.

d. If the analyzer reports values within the acceptable ranges of the reference values, it is ready for measurement of unknown samples. Proceed to Analysis of Unknown Samples section.

e. If the analyzer reports values outside the acceptance tolerance ranges specified in Table 4-1, repeat the detector calibration described in step 7, then repeat the reference sample analysis in step 8a

f. If the analyzer again fails to meet the acceptance tolerance ranges specified in Table 4-1, please contact Thermo Fisher Scientific or your local representative for assistance.

If lead is in a surface coating the Painted Products mode should be used.

Caution Only use Painted Products mode for consumer goods (toys, apparel, etc). Do not use Standard Mode or K+L Mode for analysis of painted samples not associated with building materials. Do not use Painted Products mode for analysis of building materials.

Follow the procedure outline in step 8.1, but using the Painted Products standard.

Table 4-2. Painted Products "Lead Reference Sample" Nominal Concentrations and Allowable Ranges

Element	Nominal Concentration* (micrograms/cm2)**	Acceptable Range (±% Rel)	Acceptable Range (micrograms/cm2)
Pb 3	00	15%	255-345

***Please refer to the certificate accompanying the reference sample for actual elemental composition data.**

**** Please note that the standard used (green paint) is reported in milligrams/cm². 0.30 milligrams/cm² is equal to 300 micrograms/cm².**

Action Levels

Painted Products mode allows the XRF instrument to be used to its full advantage, namely as a rapid and non-destructive test. This mode enables a direct positive/negative comparison against the new alternative standard of 2 micrograms/cm² The value of the action level can be set from 2 micrograms/cm² to 9 micrograms/cm², with 2 micrograms/cm² set by default. To change the default value follow the following procedure:

From the Main Menu, select Mode -> Consumer Goods -> Set Action Level

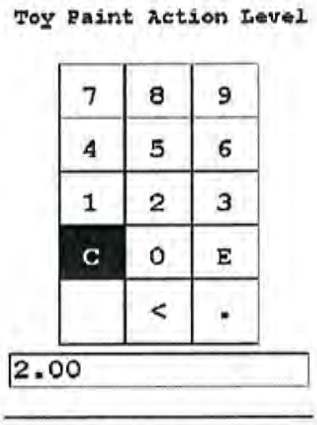


Figure 4-15. Toy Paint Action Level Screen

There is no correlation between micrograms/cm^2 and ppm, although some research indicates that the value of 1 micrograms/cm^2 to 9 micrograms/cm^2 roughly correlates to Pb concentrations in dried paint of 50 ppm to 600 ppm, respectively. However it is important to point out that these are alternate measurements set by the regulators, which means that they don't have to agree with each other to be valid.

When using plastics mode, results are reported in ppm (mg/kg). A pass/fail/inconclusive can be setup to correlate with a certain action levels. Based on the current regulation (as of March of 09), the action level for Pb in consumer products is 600 ppm (300 ppm in August 2009). Given the variability of the samples and the number of possible ways of presenting the sample to the analyzer, the pass criteria should be set lower than 600 ppm. While performing screening activities, we recommend that the identification of any Pb in a sample should alert the user to the need for additional testing to determine true content and location of Pb in the sample; setting the pass/fail level to 100 ppm (factory default) would allow this. To change factory default settings, follow the following procedure:

From the Main Menu, select Common Setup -> Element Display Options -> Set Element Threshold -> Plastics Mode

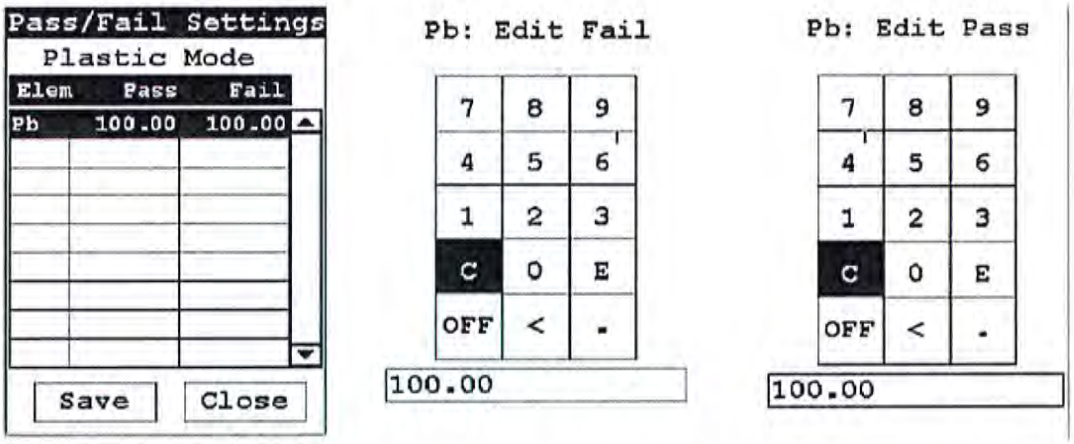


Figure 4-16. Plastics Mode Pass/Fail Action level settings

Analysis of Unknown Samples

Toys and the majority of other consumer products represent a wide variety of material composition, shapes, and functionality. Plastic, wooden, and paper goods should be tested using Plastic Mode. Products and components made of a combination of metallic and nonmetallic parts (materials) should be tested using Plastic Mode. Painted objects should be tested in the Painted Products Mode.

Note The following text applies to the use of a Niton XLp 300 Series XRF analyzer with Consumer Goods mode.

All parts of a product should be screened for the restricted element of lead using the appropriate analytical mode. The primary goal of screening is not to accurately analyze the chemical composition of all components or parts of the product, but to weed out and eliminate from the market products containing lead. Therefore, it is only logical to start testing the product "as is", without disassembly. This is particularly true of the products that are painted because lead can typically be found in paint. Once the presence of lead is confirmed during testing "as is", the product can be sequestered, and further testing may not be necessary. If, however, the testing of the product "as is" produces no evidence of lead, but it is suspected that it may be contained in inaccessible parts of the product for direct "as is" testing, one must consider disassembly of the product for compliance testing at the component level. If the product or its components are large enough, they may be tested directly by pressing the analyzer against their surfaces. Otherwise, measurements should be performed in a test stand.

Due to the large variety of consumer products, only general rules of testing, related mainly to proper sample selection and results interpretation, are outlined here. Detailed testing protocols should be developed for specific cases by the user.

General Testing Protocol

Large Objects

These are the objects that can easily cover the measuring window of the analyzer and are typically at least as large as the analyzer.

1. **Remove the foam from the upper part of the Niton XLp 300 Series analyzer..s carrying case.**

2. **Place the In-Situ Test Guard on the analyzer. This test guard is used to help enable the front proxity button as required during measurement.**

3. **Select the areas or fragments of the sample that is to be tested.**

   a. Based on the type of material, select appropriate analytical mode:

      i. Plastic Mode for all nonmetallic materials (or if uncertain of material)

      ii. Painted Products mode for painted surfaces on materials.

4. **Place the measuring window of the analyzer flush within the area identified on the toy or product for analysis and pull the trigger to start measurement.**

5. **Figures 4-17, 4-18 and 4-19 show examples of positioning the materials to be measured.**

6. **Continue the measurement until the display says PASS or FAIL. While a FAIL may appear immediately when a sample contain high levels of Pb, it is advisable to analyze all samples for a minimum amount of time for consistency purposes. The minimum time may be 30 to 60 source seconds. Experience with continued sampling can help determine what is best for your application.**

7. Figures 4-20 to 4-23 show the results screen for each mode.

8. When testing larger surfaces, it is advisable to take several measurements at different locations. This is especially important if the surface tested is partially painted, plated, or otherwise appears different from location to location, including areas with different color paints. This will help in identifying the location of any Pb found during the screening, whether in the substrate, paint, or both.

9. If the foam is not used for the measurement, insure that the surface holding the object being tested does not contain lead by testing the surface prior to screening unknown samples.

10. The object being tested should be at least one inch (2 centimeters) in thickness.

11. Continue the measurement as planned.

Figure 4-17. Screening a larger object.

Figure 4-18. One possible way of screening a toy.

Figure 4-19. The sweater is placed on the table for measurement. Ensure the table has no lead by first measuring it with the analyzer.

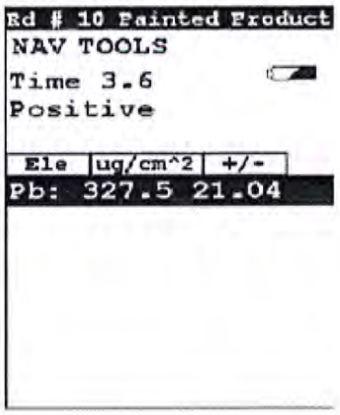


Figure 4-20. The results screen shows a positive result for lead in paint.

Figure 4-21. Inconclusive reading for this plastic item. A high levels of Pb appears to be present but more time is needed for the measurement at the current threshold limit to conclusively determine that the result is positive.

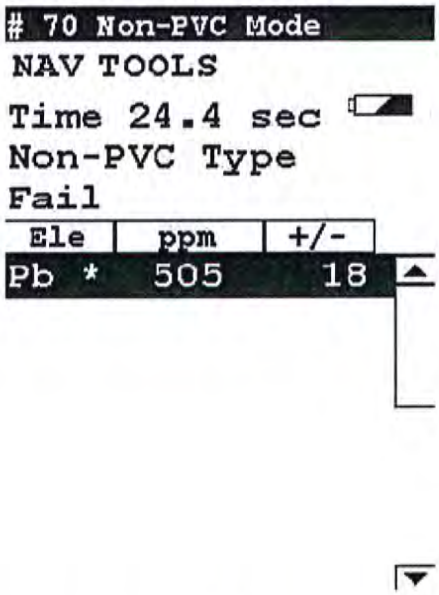


Figure 4-22. The result indicates fail (action level set at 100 ppm)

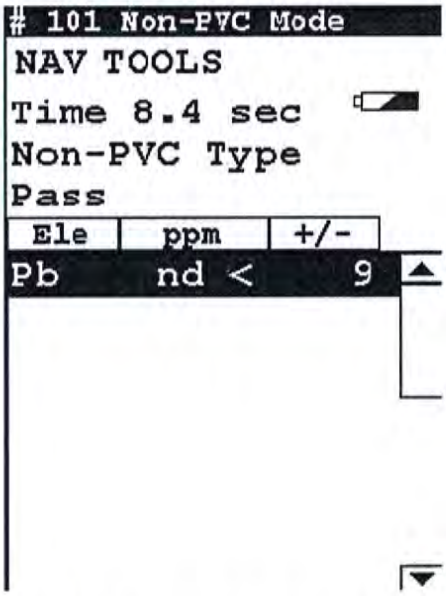


Figure 4-23. The result indicates pass (action level set at 100 ppm)

Small Objects

These are objects that are typically smaller in size than analyzer, but still large enough to cover the measuring window of the analyzer.

1. **The foam may be used as in the protocol for the Large Objects.**

   a. Optionally, place the object in a test stand in such a way that the area selected for testing covers the measuring aperture of the instrument as much as possible. If the toy cannot be manipulated into a position suitable for measurement, one may need to use some small objects to support the object in proper position.

Figure 4-24. The figure is positioned so that the item in Figure 15 is balanced on its side,

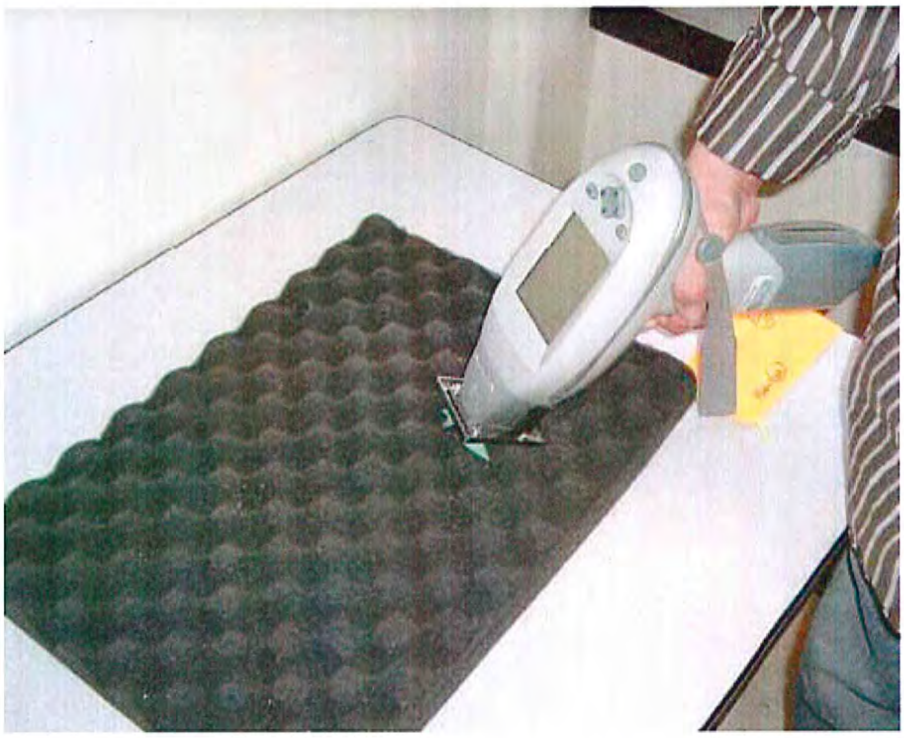


Figure 4-25. The item is placed on the foam supplied in the top of the instrument carrying case

A second option is to use a test stand (not supplied)

Figure 4-26. Niton XLp 300 Series shown with optional portable (part #500-961) and bench-top (part #600-705) test stands

b. First select the areas or fragments of the object that are to be tested.

c. Mount the analyzer in a test stand and connect the analyzer to a PC (via Serial Cable, or via Bluetooth). Start the NDTr Program on PC.

d. Place the object in a test stand in such a way that the area selected for testing covers the measuring aperture of the instrument as much as possible. If the toy cannot be manipulated into a position suitable for measurement, one may need to use some small objects to support the object in proper position. Common, small rubber erasers were found suitable and convenient for such task. Care must be taken to not allow any part of the support to be within the perimeter of the measuring window of the analyzer. Figures 4-27 and 4-28 show examples of how the object can be manipulated into proper testing position.

e. Based on the type of material, select appropriate analytical mode:

   i. Plastic Mode for all nonmetallic materials and combinations of nonmetallic

   ii. Painted Products Mode for painted surfaces.

f. Start the measurement, preferably via a PC using the NDTr software program.

g. Continue the measurement until the display says PASS or FAIL.

h. If possible, take more than one measurement of the sample, preferably in a different location.

2. Continue measurements as planned.

Figure 4-27. The unpainted bulk of the dinosaur's foot

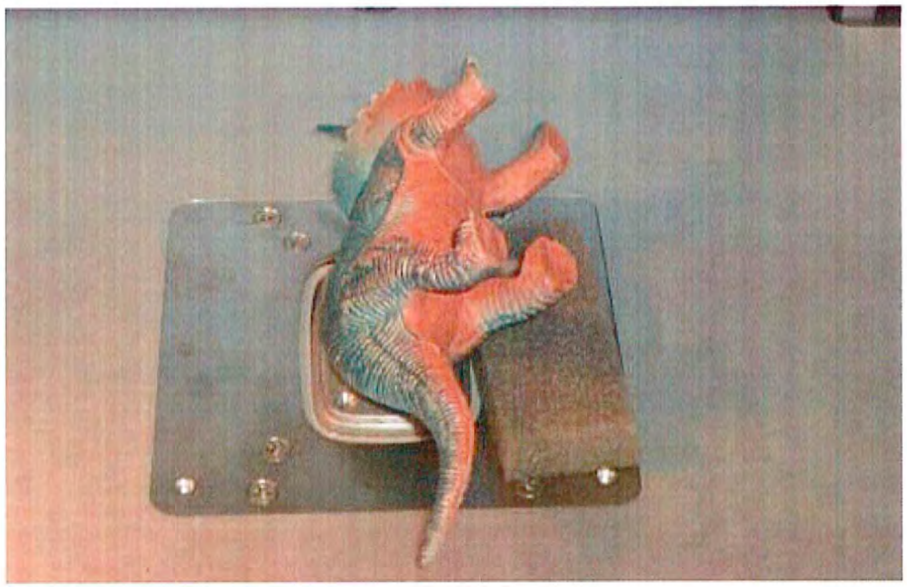


Figure 4-28. Positioning the sample for analysis may require a helping hand

Very Small Objects

These are objects that are not large enough to cover the analyzer measuring window, typically less than 10 mm in any dimension, e.g., beads, small jewelry pieces, chains, etc.

1. **Mount the analyzer in a test stand and connect it to PC (via Serial Port or Bluetooth). Start NDTr software program on user's PC.**

2. **Stretch taut a piece of Mylar film (Part Number 187-462) over the measuring aperture of the analyzer and secure it with tape.**

3. **Place the object in a test stand in such a way that it resides in the center of the measuring window of the analyzer. If available, it is possible to take more than one identical small item and pile a small quantity of them over the measuring window of the analyzer. However, one must be certain that these individual items are identical in composition.**

4. **Based on the type of material, select appropriate analytical mode:**

   a. Plastic Mode for all nonmetallic materials

   b. Painted Products Mode for paint on consumer products.

5. **Start the measurement, preferably using NDTr.**

6. **Continue the measurement until the display says PASS or FAIL.**

7. **Continue measurements as planned.**

Chapter 5 Thin Sample Test Modes

You can use your NITON XLp Environmental Analyzer to measure a wide range of thin samples, such as:

- 25 mm and 37 mm diameter filters used for exposure monitoring and air sampling methods.
- Total Suspended Particulate (TSP) and Particulate Monitoring (PM) filters.
- Dust Wipes.
- Thin coatings, depending on the substrate.
- Filters used for capturing suspended or dissolved metals in liquids

There are specific procedures for specific applications. Methods for many of these applications have been developed and may be selected from the Thin Sample Mode Menu. Other applications, such as analyzing coatings, may require you to develop your own protocol using User-definable Thin Sample Mode, which is also available from the Thin Sample Mode Menu.

Using Thin Sample Test Modes

The methods you can select have been developed to account for the size of the sample and the distribution of the contamination to be analyzed. Contamination captured on filters or wipes is not usually deposited uniformly, and as filters and wipes are also several times larger than the 1 cm x 2 cm measurement window of the instrument, these sample types usually require reading on separate sections for accurate analysis.

Depending on the sample, the individual readings may be first weighted to account for the loading characteristics of the sampling technique, and then all the individual reading in a sequence are either summed or averaged. This result is multiplied by an adjustment factor to account for the size of the sample and the desired units of measurement, so the final results are displayed with an easily interpreted meaning. (e.g. dust wipes, which are used extensively to test for Pb dust on surfaces report in µg/wipe, which is easily converted to µg/foot^2, the units specified in state and federal regulations.) These calculations will be performed automatically when you choose the appropriate Thin Sample Mode from the Thin Sample Mode Menus.

The Tools Menu

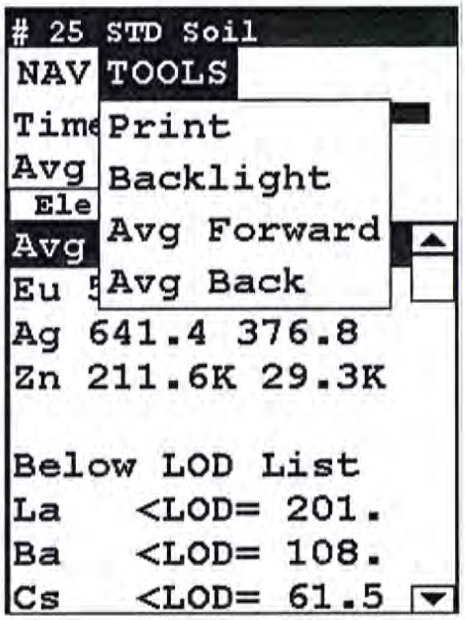


Figure 5-1. The Tools Menu

The Tools Menu enables you to perform common data-related tasks such as printing and averaging. Select a task from the drop down menu to initiate that task.

The Tools Menu, like the NAV Menu, uses context sensitive menus. The following is the most common menu set.

Print

Enables you to print the sample analysis to the attached printer.

Backlight

Enables you to turn backlighting on and off.

Avg Forward

Enables you to average different readings together from this analysis forward. Select Avg Forward to initiate future sample averaging. Avg Forward will set up an automatic personal averaging protocol to be followed until your analyzer is shut down. To begin, select the number of readings you want to average from the virtual numeric keypad. Your analyzer will calculate an average reading after that number of tests, and continue this pattern until stopped. For example, if you select 3 on the virtual keypad, the analyzer will automatically calculate, average, and store a reading for every three tests you take.

Avg Back

Enables you to average different readings together from this analysis backward. Select Avg Back to initiate backwards sample averaging. Avg Back will take a number of readings you select and average their analytical results. The range is counted from the last reading backward by the number of readings selected. If your last reading was #15, selecting 3 would average readings #13, 14, and 15. The average is calculated, displayed, and stored into memory as the next sequential reading number.

Note You cannot average readings taken with different modes - or with different sources if they have different element lists - with either Avg Back or Avg Forward. Alloy and Mining modes use the same element lists with the different sources, so averaging works when switching between sources in either of these modes. Thin Film and Bulk modes both use different element lists for different sources, and readings with different sources cannot be averaged when using either of these modes. ♦

Note The Tools Menu is only available when viewing readings, and the menu is only accessible through the touch screen interface or NDTr. ♦

The range number is selected using a virtual keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the "E" key to enter the number. "C" will clear all, and "<" will clear the last digit entered. The average will automatically be displayed.

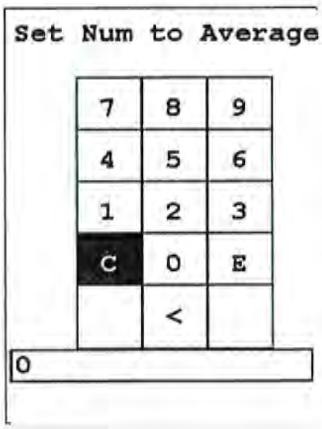


Figure 5-2. Set Range Number Screen

Stop Avg Fwd/Back

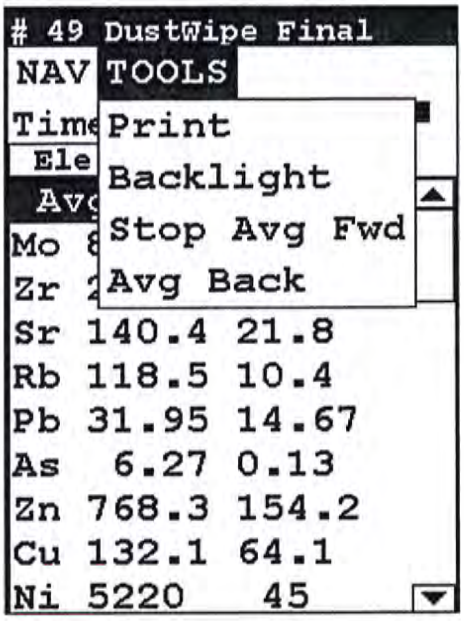


Figure 5-3. Stop Average Tool

Avg Back and Avg Forward are toggles. The option on the Tools Menu changes to its opposite when selected. To stop averaging, select Stop Avg Fwd or Stop Avg Back from the Tools Menu as appropriate.

Example Averaging

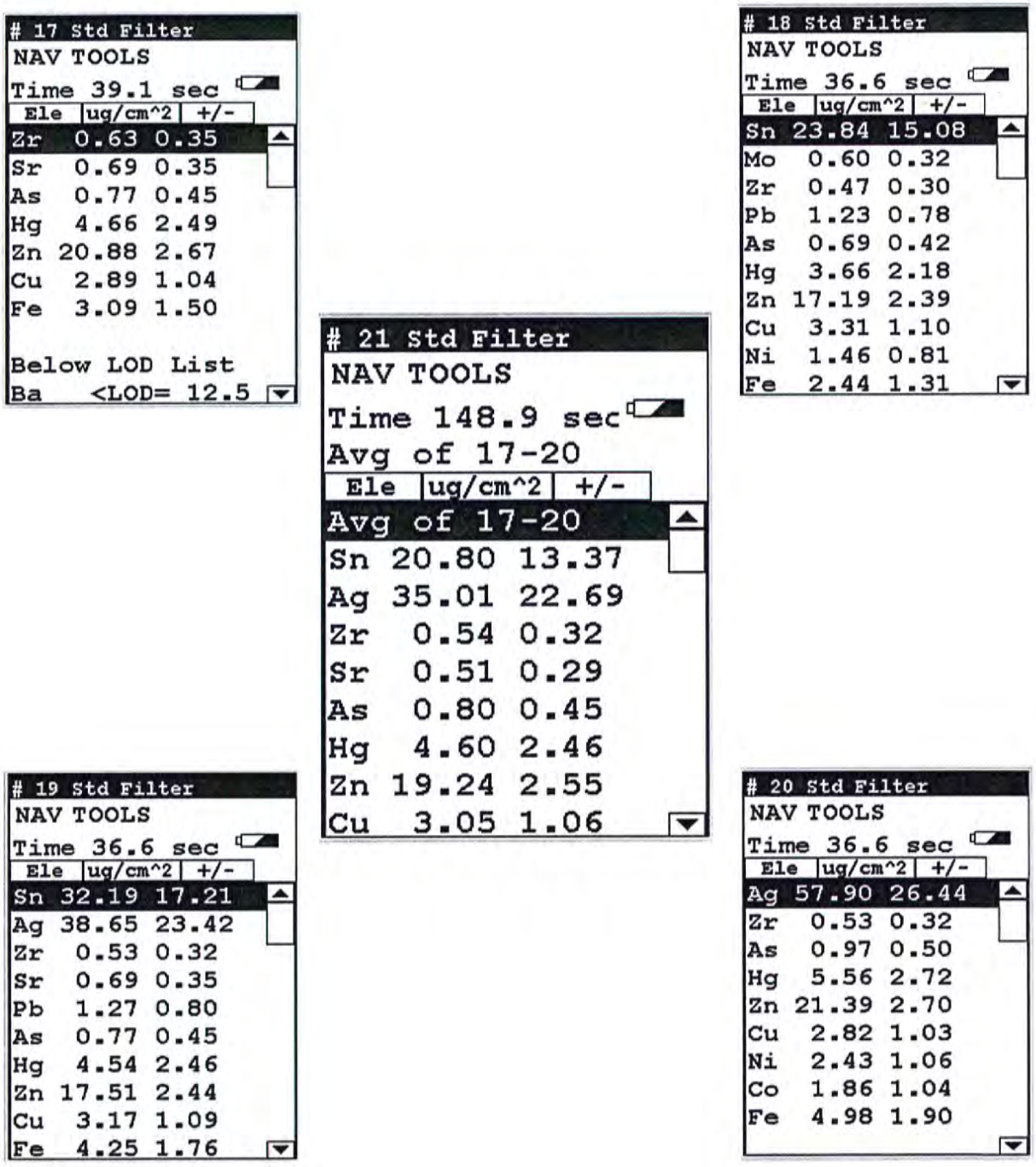


Figure 5-4. Averaging four readings

The Thin Sample Mode Menu

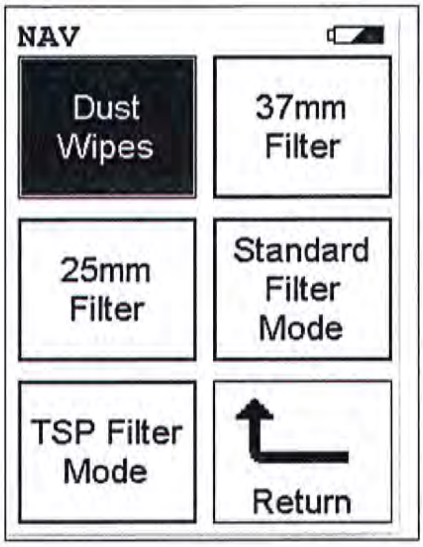
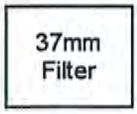


Figure 5-5. The Thin Sample Mode Menu

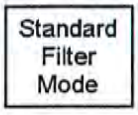
All NITON XLp Thin Sample Mode functions are accessible from the Thin Sample Mode Menu and subsidiary menus. Each of the instrument functions represented by an icon on the Thin Sample Mode Menu screen may be selected by choosing the appropriate icon. When one of these Thin Sample Mode Menu icons is selected, the function specific sub-menu appropriate to that icon will be displayed.

To use the Dust Wipe Testing Mode, simply select the Dust Wipe icon from the Thin Sample Mode Menu to place your instrument into Dust Wipe Testing Mode.



To use the 37mm Filter Testing Mode, simply select the 37mm Filter icon from the Thin Sample Mode Menu to place your instrument into 37mm Filter Testing Mode.

To use the 25mm Filter Testing Mode, simply select the 25mm Filter icon from the Thin Sample Mode Menu to place your instrument into 25mm Filter Testing Mode.



To use the Standard Filter Testing Mode, simply select the Standard Filter Mode icon from the Thin Sample Mode Menu to place your instrument into Standard Filter Testing Mode.

To use the TSP Filter Testing Mode, simply select the TSP Filter Mode icon from the Thin Sample Mode Menu to place your instrument into TSP Filter Testing Mode. At the time this User's Guide was published, the TSP Filter Testing Mode was not available on the NITON XLp Analyzer.

For more information on this feature, please contact Thermo Scientific's Customer Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 or your local Authorized NITON Analyzers Service Center.

Dust Wipe Testing Mode Menu

Figure 5-6. Dust Wipe Mode Testing Menu

From the **Dust Wipe Testing Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **Dust Wipe Testing Menu**to go to the "The Data Entry Screen" on page 5-24, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

The Dust Wipe Mode prompts you to take four readings on a properly folded and bagged dust wipe, which are then averaged to give you a final test result.Dust wipes are usually analyzed for Pb, though many other contaminants may also be detected.

The wipe recommended by NITON is the Lead Wipe. If the use of other brands is desired, consult with NITON prior to any testing.

Note Some brands of wipes are not compatible with the NITON method. •

Lead Wipes are available from Thermo or from:

Lynx Products

Thorofare, New Jersey

(800) 767-6933

The following instructions are appropriate for screening purposes. The instrument displays levels of contamination in µg per wipe. The wipe reflects the levels of contaminants of the area wiped. Current regulations require lead contamination below 40 µg/ft^2 on floors, 250 µg/ft^2 on window sills, and 400 µg/ft^2 in window wells.

Note Check with all local, state and federal regulatory bodies, as the regulations may change from location to location, and from time to time. ♦

Taking a Dust Wipe Sample

NITON assumes that you will follow the HUD guidelines for taking a dust wipe which are summarized here:

1. **Measure a known area of the surface, preferably one square foot**

2. **Wear clean surgical gloves. Wipe the measured square with parallel strokes**

3. **Fold the wipe in half. Wipe in strokes 90° to the original direction.**

4. **Fold the wipe in half again.**

Note Thus far, you have followed one of the HUD procedures for taking a wipe test. The following procedure is specific for testing with your instrument, though it will not affect your results should you decide on submitting the sample to an accredited laboratory. ♦

5. **Fold the wipe in half three more times. You will now have a pad measuring about 1 x 1.5 inches (2.5 x 3.7 cm). It is important to fold the wipe neatly, so that the final wipe is very nearly a neat rectangle measuring about 1 x 1.5 inches.**

6. **Place the folded wipe in one of the plastic baggies provided**

7. **Position the wipe, in the baggie, in the metal dust wipe holder**

8. **The dust wipe is now ready to test. NITON recommends that the plastic bags NOT be re-used, to eliminate the chance of cross-contamination of subsequent wipes.**

Taking Measurements of Your Dust Wipe Sample

Follow the procedure below:

1. **Position the metal dust wipe holderon the number one position of the test stand.**

2. **Position the instrument in the nose cone adaptor and initiate the first measurement.**

3. **Place the wipe in the number two position of the test stand. Take the second measurement.**

4. **Rotate the dust wipe holder 180 degrees (without turning the holder over).**

5. **With the wipe in the number one position, take the third measurement.**

6. **Change the wipe to the number two position. Take the fourth measurement.**

This procedure assures that your analyzer measures the entire area of the folded dust wipe.

Reading The Measurement and Final Results Screens

The Measurement screen is continually updated during each test. When each test is terminated, the screen will update one final time and store the results for future review or downloading. After the fourth step of the test sequence is complete, the Final Results screen will appear.

When the four measurements are complete, the instrument automatically averages the results to yield the average loading in µg/wipe. These results are displayed on the Final Result screen. (Note that Figures 5-7 & 5-8 show multiple elements measured on a dust wipe sample. NITON 300 Series Analyzers will only display results for Pb.)

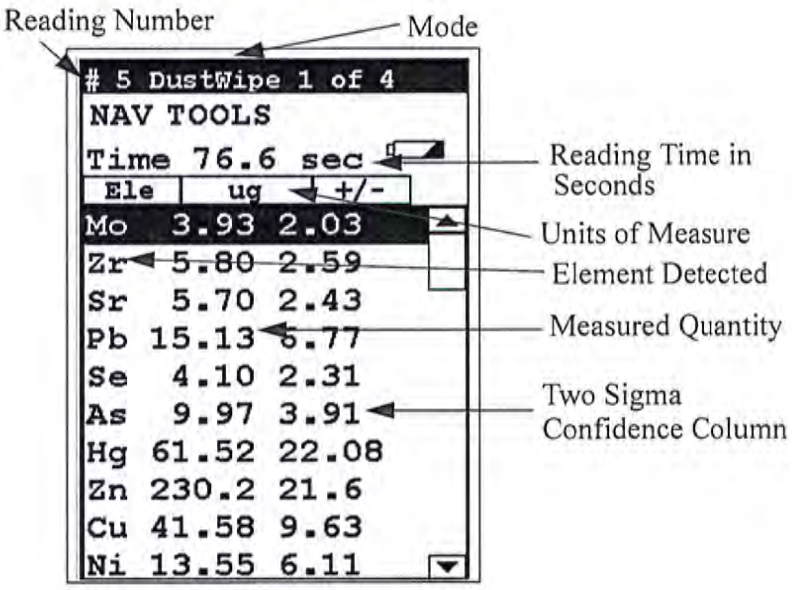


Figure 5-7. Reading the Final Results Screen

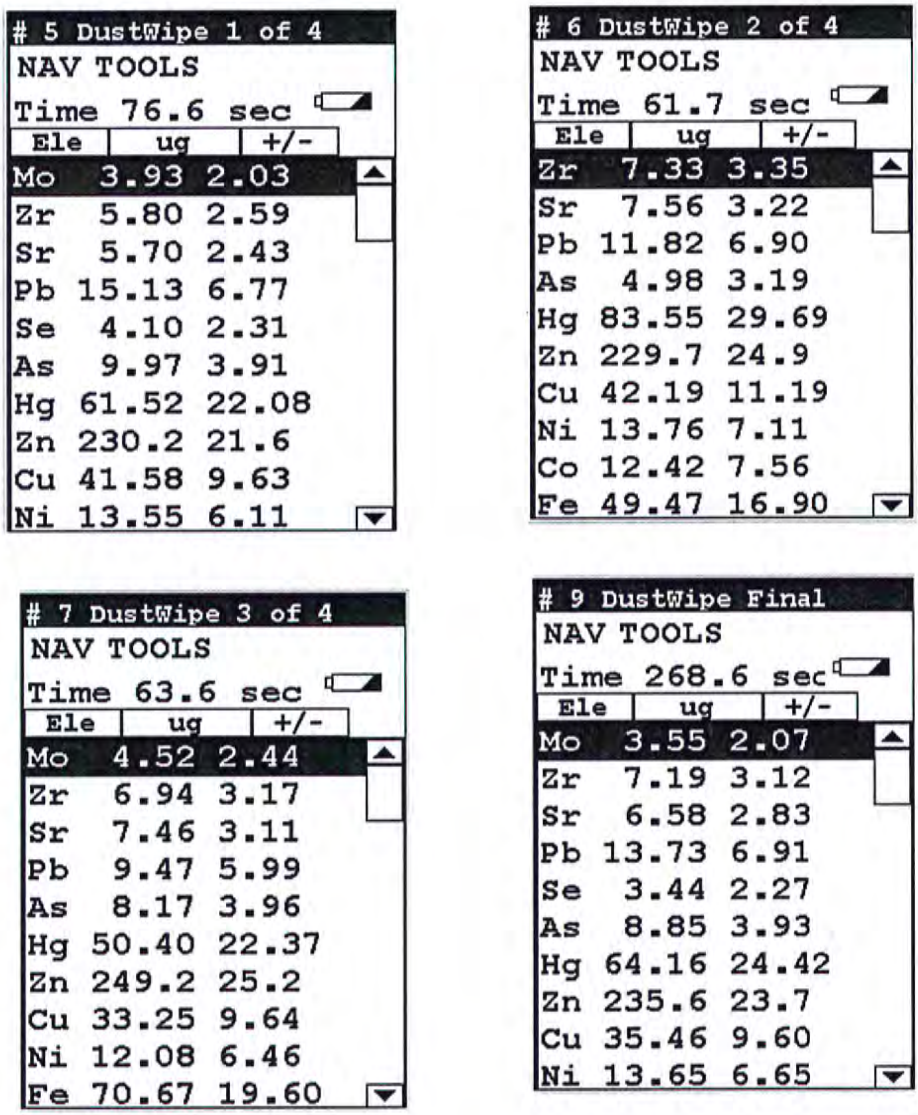


Figure 5-8. All Four Dustwipe Reading Screens

37mm Testing Mode Menu

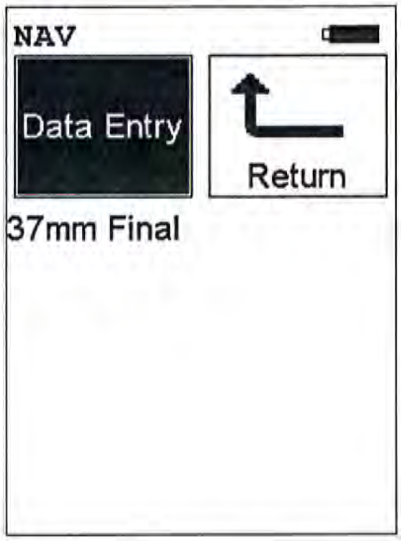


Figure 5-9. 37mm Testing Mode

From the **37mm Testing Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **37mm Testing Menu**to go to the "The Data Entry Screen" on page 5-24, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

Preparing a filter

37 mm filters are often used for monitoring personal exposure. Dust vacuum measures (DVM) use the same size filters and are tested in much the same way. To prepare the filter for testing, remove it from the air-sampling cassette and load it in the correct sized filter sleeve. The filter sleeve consists of a thin piece of cardboard sandwiched between two layers of plastic film. The circular cutout in the cardboard should be slightly larger in diameter than the filter.

Note To avoid contaminating the test results, wear clean surgical gloves when handling the filter. If using tweezers or forceps to handle the filter, make certain they are clean, and never reuse a filter sleeve. ♦

1. **On a clean surface, take a filtersleeve and peel back the top layer of film.**

2. **Remove the bottom plug from the air sampling cassette. Separate the sections of the cassette so you can reach the filter.**

3. **Poke both the filter and filter pad through the outflow plug hole of the cassette, to release them from their seat in the cassette.**

4. **Touching only the edges of the filter and the pad, gently separate one from the other.**

5. **Lift the filter from the cassette and place it on the sleeve in the cutout. Be careful not to have the filter touch the paper mounting of the sleeve, as the light adhesive that holds the plastic in place may rip the filter.**

6. **Fold back the clear plastic of the filter sleeve, covering the filter. A little wrinkling of the plastic is acceptable.**

Note It is advisable to practice mounting several blank filter cassettes prior to mounting your first test sample. ♦

Positioning the 37 mm Filter and Performing a Test.

Note Make certain to place the filter stand on a flat stable surface. Be careful to work on a surface which will not contaminate the test fixtures or samples. ♦

1. **Remove the drawer from the test platform. Verify that the adaptor for holding 37 mm filters is in place. Insert the filter sleeve with the filter to be tested in position A of the adaptor, and slide the drawer back into the test stand.**

Note Testing order is essential: The first reading must be taken from position A. ♦

2. **Select the Thin Sample Mode Select menu, and then choose 37mm CE Filters icon**

3. **Position the instrument in the nose cone adaptor and initiate the first reading.**

You must take three readings, each from a different area of the filter to accurately determine the concentration of elements on the filter. When the desired test time has elapsed, remove the instrument to end the first test. The instrument will beep at 60 nominal (or source) seconds, unless pre set otherwise by the user, which is typically an adequate test time for the precision of the measurement to detect the presence of many contaminants at their action levels.

Note If concentrations of the elements of concern are significantly above the action level, the tests lengths may be shortened at the users discretion. •

4. **Remove the drawer and change the filter to position B.**

5. **Repeat the above sequence to take the second test.**

6. **When the second test is complete, remove the drawer and position the test sleeve in position C and take a third measurement.**

The instrument will automatically calculate the contaminant loading, in µg, after the three readings have been completed.

Reading The Measurement Screen in 37mm Testing Mode

The Measurement screen is continually updated during each test. When each test is terminated, the screen will update one final time, display the phrase Final Results, and store the results for future review or downloading. 37mm Testing Mode reports in units of µg of loading, and using the volumetric flow rate and airsampling time, the results can be converted to $\mu g/m^3$ concentrations.

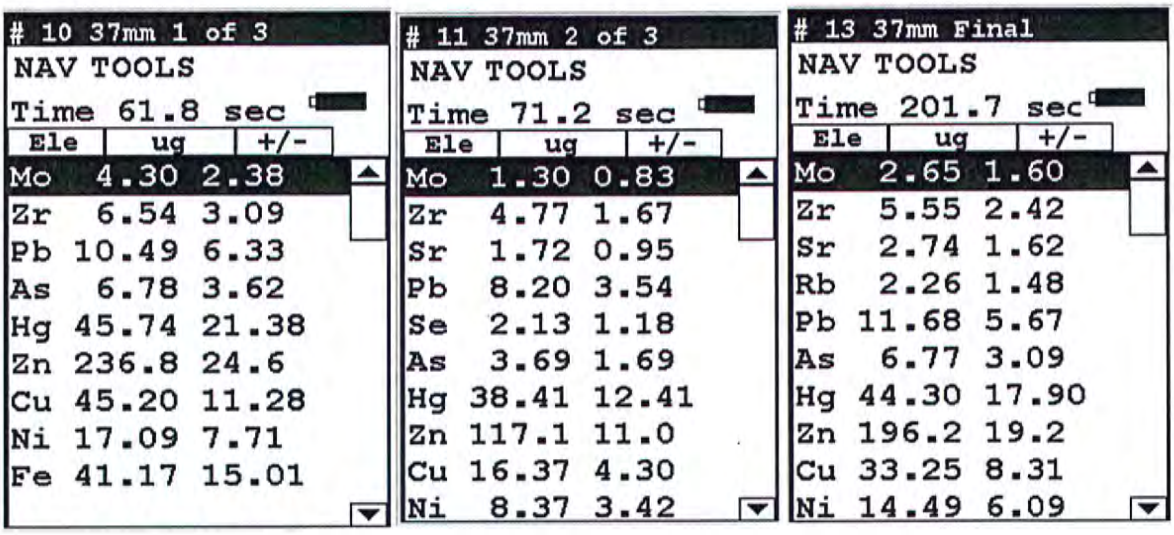


Figure 5-10. All Three 37mm Reading Screens

25mm Testing Mode Menu

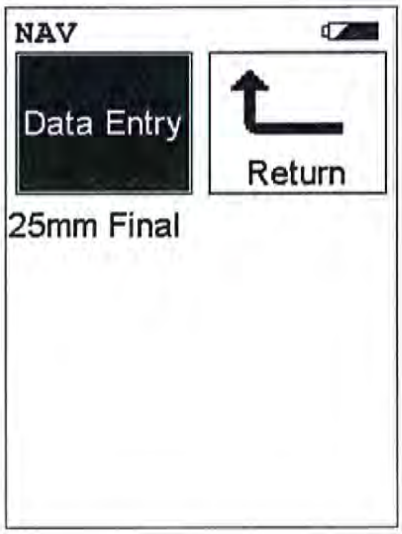


Figure 5-11. 25mm Testing Mode Menu

From the **25mm Testing Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **25mm Testing Menu**to go to the "The Data Entry Screen" on page 5-24, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

Preparing a filter

25 mm filters are often used for monitoring personal exposure. Dust vacuum measures (DVM) use the same size filters and are tested in much the same way. To prepare the filter for testing, remove it from the air-sampling cassette and load it in a the correct sized filter sleeve. The filter sleeve consists of a thin piece of cardboard sandwiched between two layers of plastic film. The circular cutout in the cardboard should be slightly larger in diameter than the filter.

Note To avoid contaminating the test results, wear clean surgical gloves when handling the filter. If using tweezers or forceps to handle the filter, make certain they are clean, and never reuse a filter sleeve. ♦

1. **On a clean surface, take a filtersleeve and peel back the top layer of film.**

2. **Remove the bottom plug from the air sampling cassette. Separate the sections of the cassette so you can reach the filter.**

3. **Poke both the filter and filter pad, through the outflow plug hole of the cassette, to release them from their seat in the cassette.**

4. **Touching only the edges of the filter and the pad, gently separate one from the other.**

5. **Lift the filter from the cassette and place it on the sleeve in the cutout. Be careful not to have the filter touch the paper mounting of the sleeve, as the light adhesive that holds the plastic in place may rip the filter.**

6. **Fold back the clear plastic of the filter sleeve, covering the filter. A little wrinkling of the plastic is acceptable.**

Note It is advisable to practice mounting several blank filter cassettes prior to mounting your first test sample. ♦

Positioning the 25 mm Filter and Performing a Test.

Note Make certain to place the filter stand on a flat stable surface. Be careful to work on a surface which will not contaminate the test fixtures or samples. ♦

1. **Remove the drawer from the test platform. Verify that the adaptor for holding 25 mm filters is in place. Insert the filter sleeve with the filter to be tested in position one of the adaptor, and slide the drawer back into the test stand.**

Note Testing order is essential: The first reading must be taken from the middle of the filter. ♦

2. **Select the Thin Sample Mode Select menu, and then choose 25mm CE Filters icon**

3. **Position the instrument in the nose cone adaptor and initiate the first reading.**

The instrument will automatically calculate the contaminant loading, in mg, after the reading has been completed.

Reading The Measurement Screen in 25mm Testing Mode

The Measurement screen is continually updated during each test. When each test is terminated, the screen will update one final time, display the phrase Final Results, and store the results for future review or downloading. 25mm Testing Mode reports in units of µg of loading in just minutes, and using the volumetric flow rate and airsampling time, the results can be converted to $\mu g/m^3$ concentrations.

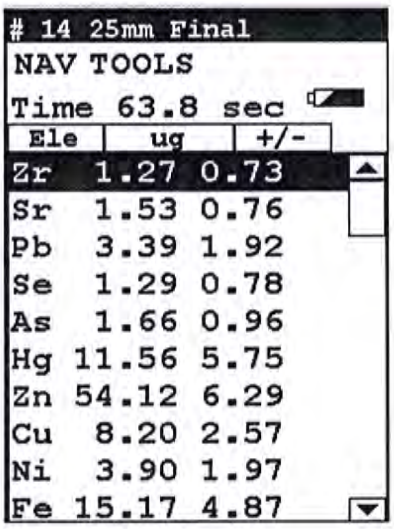


Figure 5-12. 25mm Finalt Results Screen

Standard Testing Mode Menu

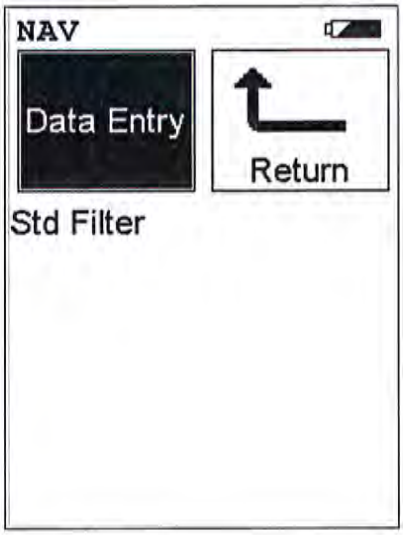


Figure 5-13. Standard Testing Mode Menu

From the **Standard Testing Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **Standard Testing Menu**to go to the "The Data Entry Screen" on page 5-24, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

You should use Standard Thin Sample Mode to test thin samples that have uniform contamination or deposition. These include many filters for liquids and gases, various types of coatings, and the leaves of plants. Operators who want to make a single measurement and obtain a result in units of µg/cm2 should use Standard Thin Sample Mode.

CAUTION Standard Thin Sample Mode should not be used for quantitative lead-paint testing. Use only the three Paint Testing modes to test lead-based paint. ♦

In Standard Thin Sample Mode, each measurement is a separate test. For this reason, there is no Final Result screen in this mode. The results of each test are given in µg/cm2 for up to 14 elements.

Note Testing coatings and paints with Standard Thin Sample Mode may yield lower-than-actual results. The penetration depth of the measurement is highly dependent on the composition of the sample. ♦

Note Standard Thin Sample Mode does not correct for shielding caused by the presence of overlaying coatings. Thus, for coatings testing, the results should be viewed as the minimum amount of contaminants present. If an element is not detected, it may be that the element is present but entirely shielded by overlaying coatings. ♦

Reading The Measurement Screen in Standard Thin Sample Mode

The Measurement screen is continually updated during each test. When each test is terminated, the screen will update one final time, display the phrase Final Results, and store the results for future review or downloading. Standard Thin Sample Mode reports in units of µg/cm²

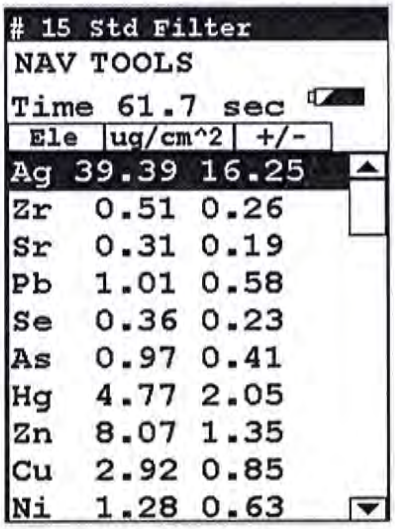


Figure 5-14. The Final Reading Screen for Standard Thin Sample Mode

For more information on this feature, please contact NITON's Customer Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 or your local Authorized NITON Analyzers Service Center.

Analyzing Thin Samples

CAUTION After being powered on, the NITON 300 Series Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your instrument warm up for ten minutes after start up, before testing is begun. •

There are five different methods of operation for taking a sample measurement, and your analyzer will be configured to use one of those methods, depending on the regulatory requirements of your locality. These methods are:

- Trigger-Only method. With the Trigger-Only method, you only need to place the measurement window close to the sample to be analyzed and pull the trigger for sample analysis to be initiated.

- Trigger-and-Proximity-Sensor method. With the Trigger-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the instrument, then pull the trigger for sample analysis to be initiated.

- Momentary-Trigger-Touch-and-Proximity-Sensor method. With the Momentary-Trigger-Touch-and-Proximity-Sensor method, you must place the measurement window against the surface to be analyzed to engage the proximity sensor on the front of the instrument, then pull the trigger.

- The trigger may be released and the reading will continue until you release the proximity button, or other criteria (such as Max Time) are reached. This is the primary mode of operation for the XLp 300A/700A Series Lead-in-Paint Analyzer in the USA.

- Trigger-and-Interlock method. With the Trigger-and-Interlock method, you need to place the measurement window close to the sample to be analyzed, press and keep pressing the interlock button at the rear of the instrument with your free hand, then pull the trigger for sample analysis to be initiated.

- Trigger-Interlock-and-Proximity-Sensor method. With the Trigger-Interlock-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the instrument, press and keep pressing the interlock button at the rear of the instrument with your free hand, then pull the trigger for sample analysis to be initiated.

With any of these methods, analysis will stop if any one of the preconditions are violated. For example, with the Trigger-Interlock-and-Proximity-Sensor method, if the trigger or the Proximity Sensor or the Interlock is released, the reading will stopimmediately, and the shutters will close.

After your NITON XLp analyzer is calibrated, initiate a sample reading using the appropriate method. If you attempt to initiate a sample reading using a different method, the analyzer will inform you that one or more of the preconditions need to be met in order for sample analysis to begin.

WARNING! The three LED warning lights are designed to blink only during a measurement, where one or more of the shutters are open and the trigger depressed. If the LED lights blink at any other time, disconnect the battery pack immediately, place the instrument in its shielded holster, place the holster in the shielded carrying case, and call NITON LLC's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, or your local Authorized NITON Analyzers Service Center. •

WARNING! The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, all the shutters will close, and the measurement will end. The three LED lights will stop blinking, the shutters will close, and the measurement will end. The flashing of the LED lights is not synchronized to minimize power consumption. •

Your NITON Analyzer will display the Results Screen throughout the duration of each reading, The Results Screen is updated regularly throughout the reading. When the reading is complete, a final screen update will appear, and your NITON analyzer will display the final results of the measurement which has just been completed.

The Data Entry Screen

The Data Entry Screen is accessed whenever you select the Data Entry icon from any screen. This screen allows you to input data in several different fields, or categories, concerning your sample, in several different ways:

- By selecting the Virtual Keyboard button and typing the parameter in using the **Virtual Keyboard**.
- By scanning in the parameter name using the integrated bar code scanner.
- By creating a new, or editing your analyzer's existing, '.ndf' file through the NDT program. You can then select from the various custom options you have created using the Drop-down List button.

These fields are saved along with the subsequent reading, and allow you to associate important information about the sample directly with the reading, so that you have a full description of the sample tied into the reading itself.

Once you have input data into a field, that information carries over into the next reading, so that you only have to input the information that has changed since the last reading. For example, if you are analyzing several samples of a particular lot, you only need to input the lot information once during that series of readings, changing only the sample name.

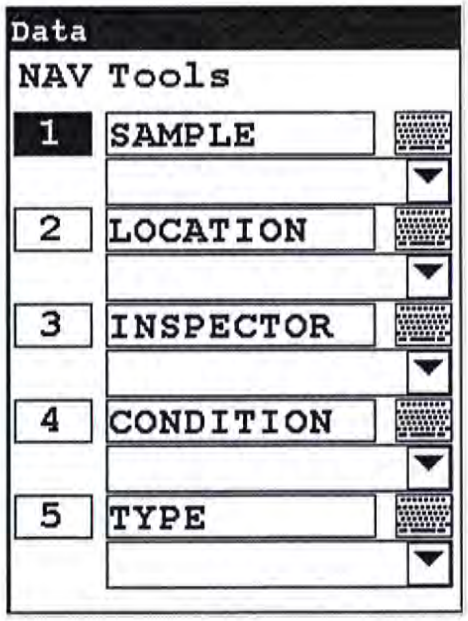


Figure 5-15. Data Entry Screen 1

This is the first section of the Data Entry Screen. There are five parameters in this section.

- Selecting **Sample** allows you to input the sample name parameter
- Selecting **Location** allows you to input the particular location information, if known
- Selecting **Inspector** allows you to specify the inspector.
- Selecting **Condition** allows you to input information on the sample's condition
- Selecting **Type** allows you to specify the sample's type

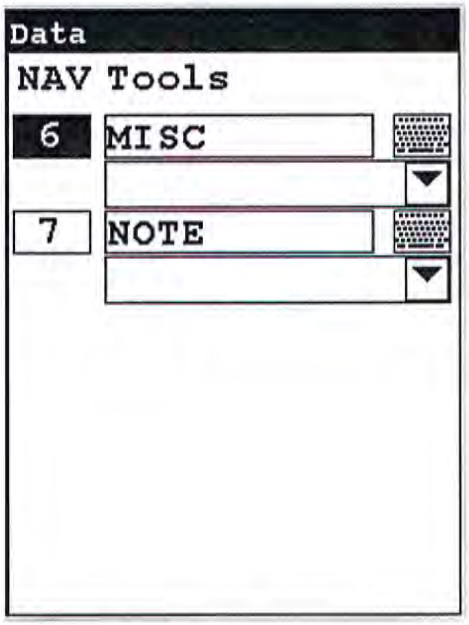


Figure 5-16. Data Entry Screen 2

This is the second section of the Data Entry Screen. There are two parameters in this section.

- Selecting **Misc** allows you to input miscellaneous parameters
- Selecting **Note** allows you to input notes on the sample

Navigating the Data Entry Screen

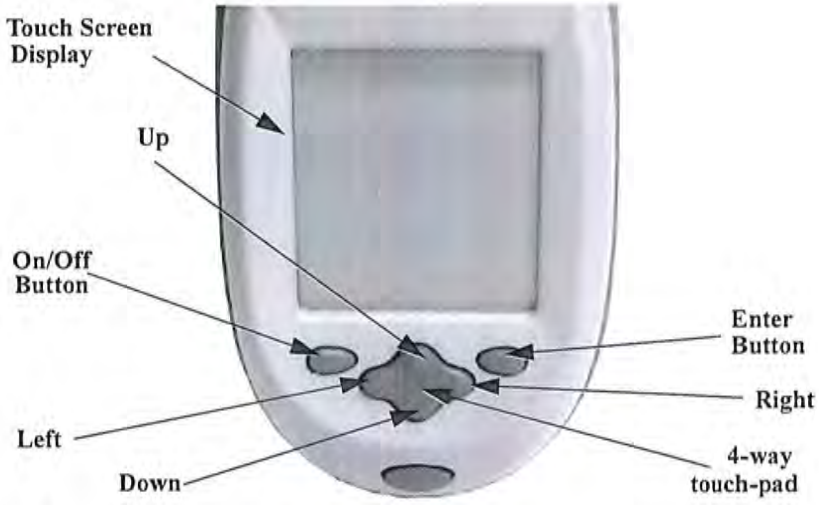


Figure 5-17. The Control Panel

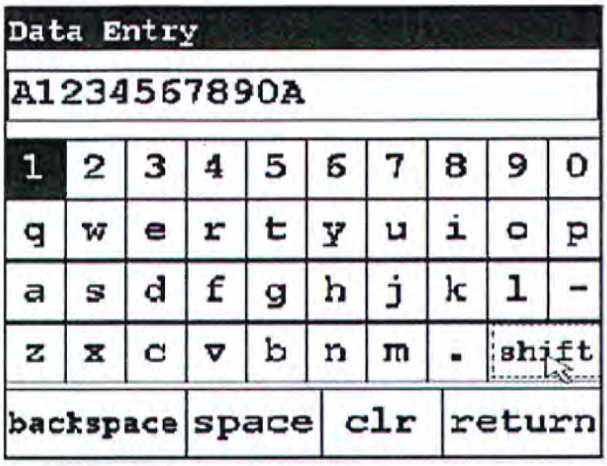
The following description of screen navigation using the control panel assumes that the analyzer is held so that the display is held upright as in Figure 5-17.

- To move from column to column, use the Right and Left portion of the 4-way touch pad.
- To move from row to row, use the Up and Down portions of the 4-way touch pad.
- To select the highlighted option, press the Enter button on the control panel.

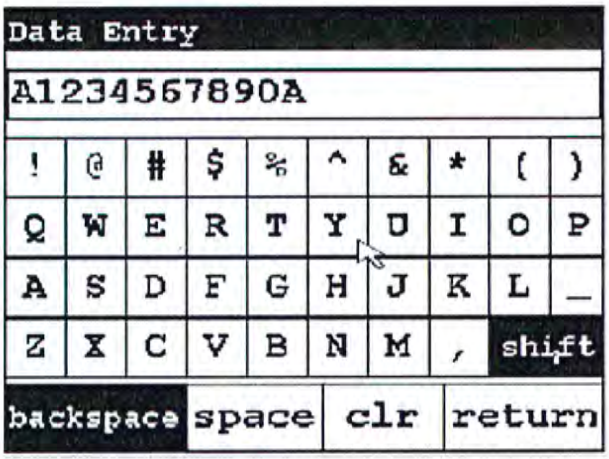
The *Data Entry* Screen is divided into sections of 5 setting parameters. By using the Down portion of the 4-way touch pad when you are on the last row of a section, the display will change to the next section. By using the Up portion of the 4-way touch pad when you are on the first row of a section, the display will change to the previous section.

By selecting the *On/Off* button, you can exit the *Data Entry* Screen.

The Virtual Keyboard



Lower Case Virtual Keyboard



Upper Case Virtual Keyboard

Figure 5-18. The Virtual Keyboard

The Virtual Keyboard is a full alphanumeric keyboard which appears on the LCD Touch Screen Display. You can use the Virtual Keyboard either with the four-way touch pad and control panel buttons, or using the touch screen display directly.

At the top of the screen is the data field you are entering data for, in this case, "Sample", Directly underneath is the data you are entering, in this case "17-4 PH SS", On this line also is the underscore cursor. This graphically shows where the next character will be placed. Up to 25 characters can be stored in the data fields.

Next is the Virtual Keyboard itself, with numbers 0-9, letters A-Z, and special characters *,<,>, and -.

Last is the control button line. This contains the screen buttons for Return and Clear.

When using the four-way touch pad and control buttons, pressing the On/Off button allows you access to the Return and Clear screen buttons. The Return screen button will enter the data and return you to the Data Entry Screen, while the Clear screen button will clear the data you have

All screen areas can be directly accessed using the LCD Touch Screen.

NITON Test Platforms

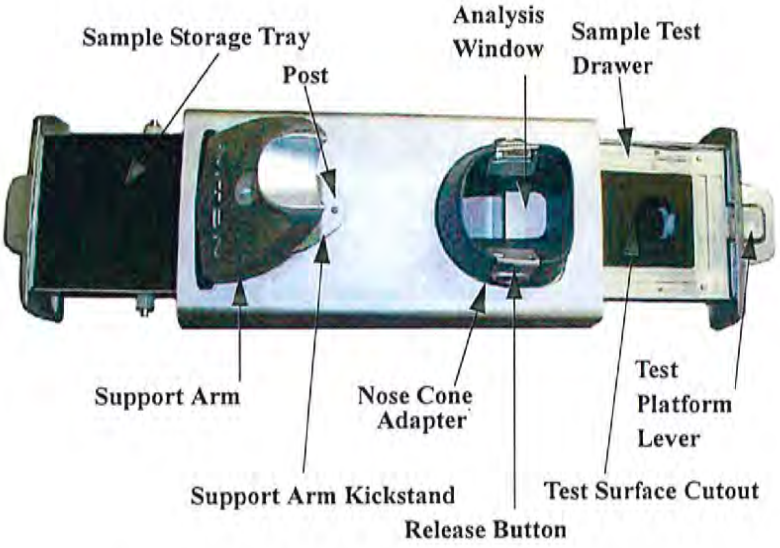


Figure 5-19. NITON Bulk Mode Test Stand

The NITON Bulk Test Stand is designed to facilitate bulk and thin sample testing using NITON Analyzers.The NITON Portable Test Stand is designed for bulk and moderate sized samples, and is portable.

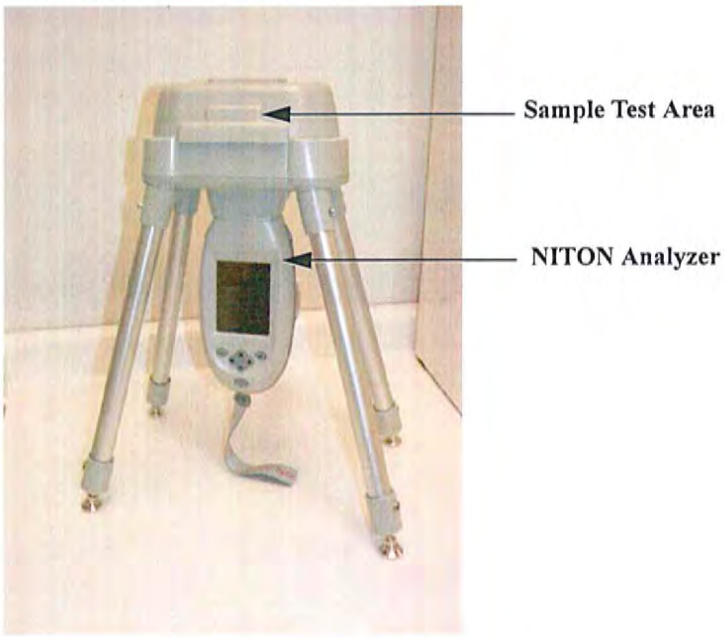


Figure 5-20. NITON Portable Test Stand

Chapter 6 Bulk Sample Test Modes

Using Bulk Sample Test Modes

Bulk Sample Test Modes allow you to test soils and other ground samples for composition and contamination. Your NITON XLp Analyzer may be used for soil and bulk analysis, either *in-situ* or *ex-situ*.

The Tools Menu

The Tools Menu, like the NAV Menu, uses context sensitive menus. The following is the most common menu set.

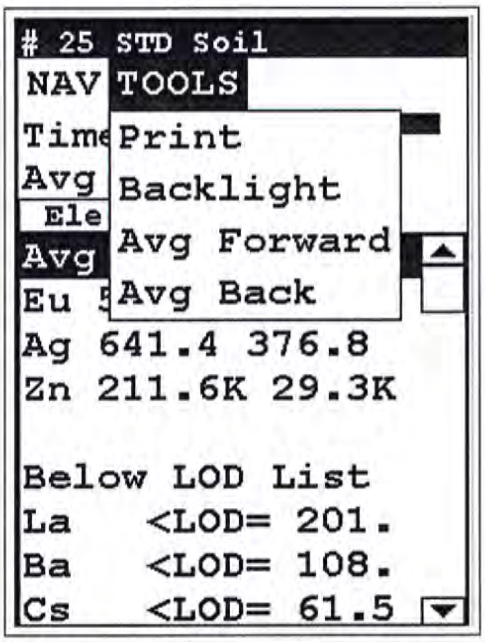


Figure 6-1. The Tools Menu

Print Enables you to print the sample analysis to the attached printer.

Backlight Enables you to turn backlighting on and off.

Avg Forward

Enables you to average different readings together from this analysis forward. Select **Avg Forward** to initiate future sample averaging. **Avg Forward** will set up an automatic personal averaging protocol to be followed until your analyzer is shut down or Averaging is turned off. To begin, select the number of readings you want to average from the virtual numeric keypad. Your analyzer will calculate an average reading after that number of tests, and continue this pattern until stopped. For example, if you select 3 on the virtual keypad, the analyzer will automatically calculate, average, and store a reading for every three tests you take.

Avg Back

Enables you to average different readings together from this analysis backward. Select **Avg Back** to initiate backwards sample averaging. **Avg Back** will take a number of readings you select and average their analytical results. The range is counted from the last reading backward by the number of readings selected. If your last reading was #15, selecting 3 would average readings #13, 14, and 15. The average is calculated, displayed, and stored into memory as the next sequential reading number.

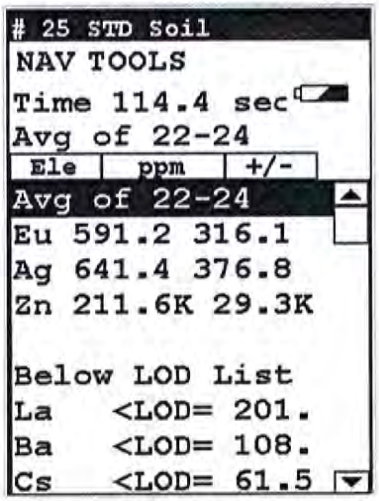


Figure 6-2. Averaging Screen

Note You cannot average readings taken with different modes - or with different sources if they have different element lists - with either Avg Back or Avg Forward. Alloy and Mining modes use the same element lists with the different sources, so averaging works when switching between sources in either of these modes. Thin Film and Bulk modes both use different element lists for different sources, and readings with different sources cannot be averaged when using either of these modes. ♦

Note The Tools Menu is only available when viewing readings, and the menu is only accessible through the touch screen interface or NDTr. ♦

The range number is selected using a virtual keypad on your analyzer similar to the keypad used for login. Select the digits in the range number from the keypad, then select the "E" key to enter the number. "C" will clear all, and "<" will clear the last digit entered. The average will automatically be displayed.

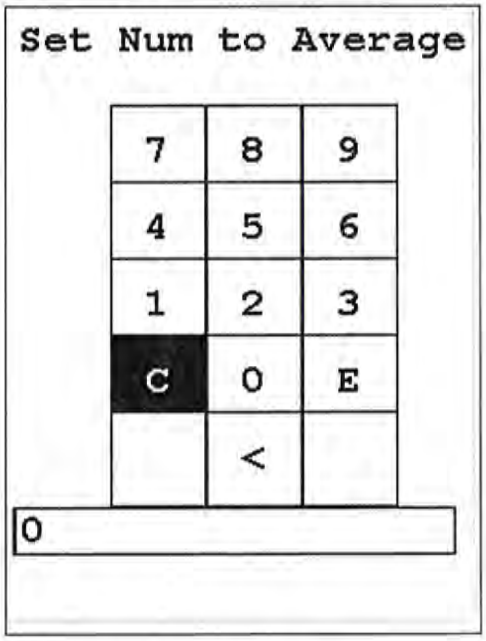


Figure 6-3. Set Range Virtual Keypad

Stop Avg Fwd

Avg Forward is a toggle. The option on the Tools Menu changes to its opposite when selected. To stop averaging, select Stop Avg Fwd from the Tools Menu.

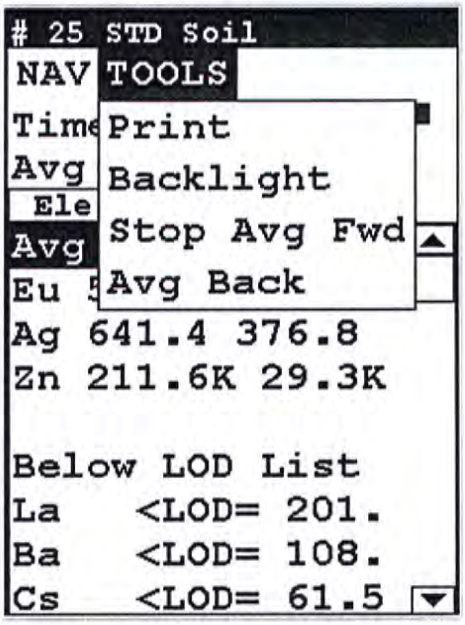


Figure 6-4. Stop Average Tool

Averaging Example

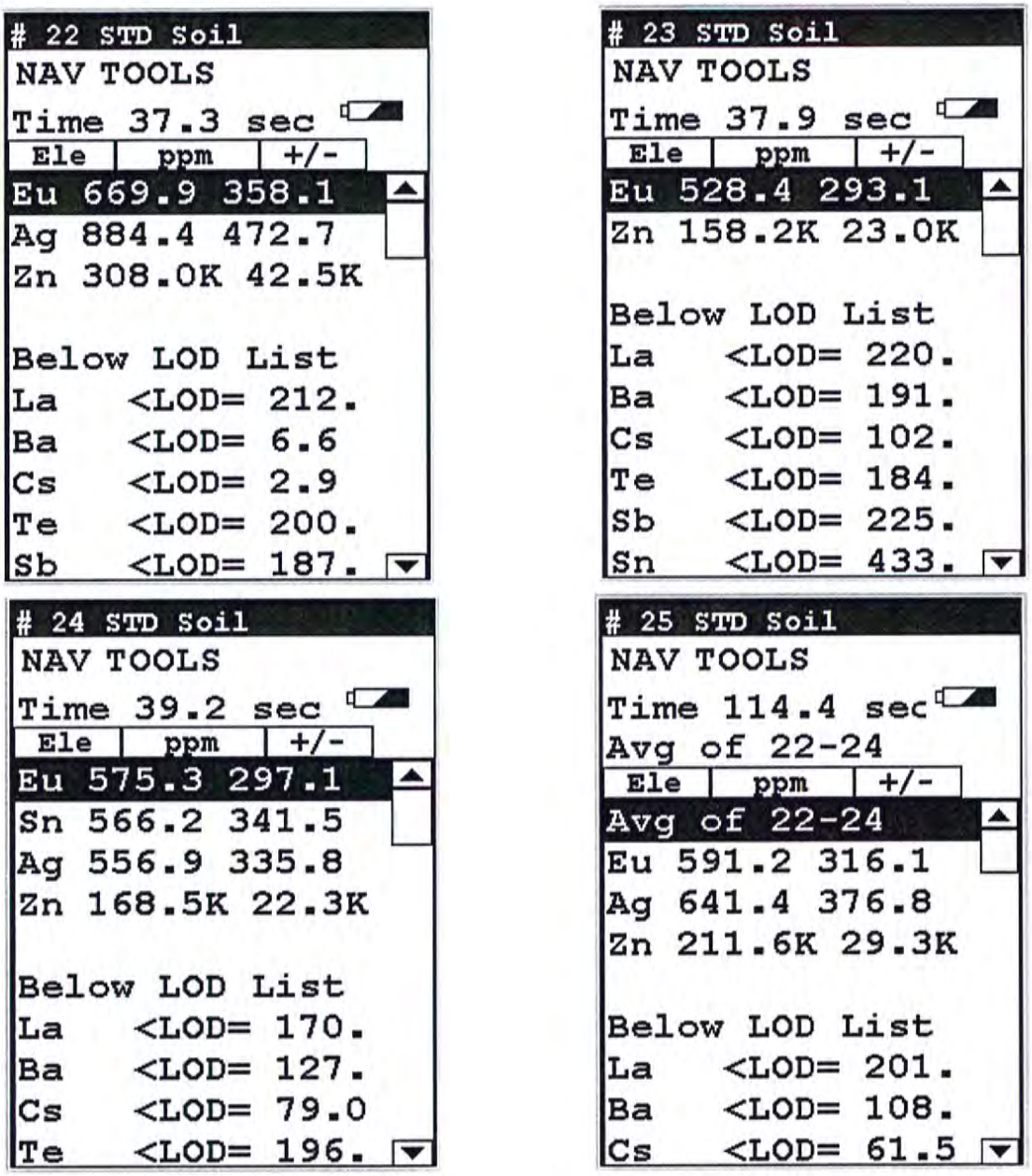


Figure 6-5. Example of Averaging

The Bulk Sample Mode Menu

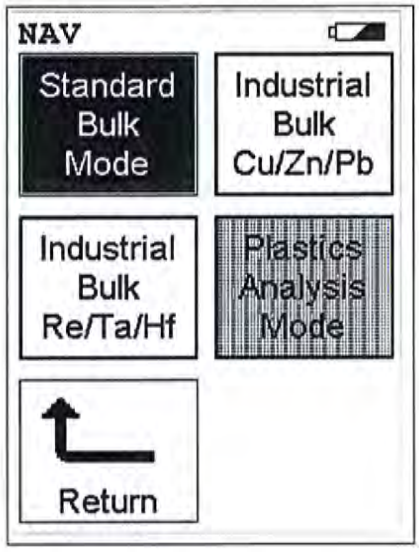
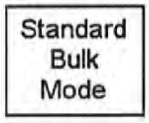


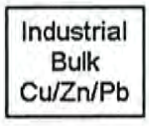
Figure 6-6. Bulk Sample Mode Menu

All NITON XLp Bulk Mode functions are accessible from the **Bulk Mode Menu** and subsidiary menus. Each of the instrument functions represented by an icon on the **Bulk Mode Menu** screen may be selected by choosing the appropriate icon. When one of these **Bulk Mode Menu** icons is selected, the function specific sub-menu appropriate to that icon will be displayed.



To use the **Standard Bulk Mode,** simply select the **Standard Bulk Mode** icon from the **Bulk Mode Menu** to place your instrument into **Standard Bulk Mode**. Use the **Standard Bulk Mode if**:

- The percentage of the elements of interest are <1.0%
- The material is of a light matrix, for example aluminum silicate
- Elements with atomic number greater than iron do not exceed several percent



To use the **Industrial Bulk Cu/Zn/Pb Testing Mode**, simply select the **Industrial Bulk Cu/Zn/Pb** icon from the **Bulk Mode Menu** to place your instrument into **Industrial Bulk Cu/Zn/Pb Testing Mode**. In **Industrial Bulk Cu/Zn/Pb Testing Mode**, all other programmed elements are active in addition to Cu/Zn/Pb (e.g. Ti, Cr, Mn, Fe, Co, Ni, Zr, Nb, Mo, W, etc.). **Industrial Bulk Cu/Zn/Pb Testing Mode** is recommended if you know, *without a doubt,* that your sample contains Cu, Zn, and/or Pb and not Ta, Hf, or Re. When your NITON XLp analyzer is

operating in **Industrial Bulk Cu/Zn/Pb Testing Mode**, the NITON XLp analyzer will not measure concentrations of Hf, Ta, or Re in a sample. Once **Industrial Bulk Cu/Zn/Pb Testing Mode** is selected, you may begin testing samples.

Use Industrial Bulk Cu/Zn/Pb Testing Mode if:

- There is more Cu/Pb/Zn than Ta/Hf/Re in the sample
- The percentage of the elements of interest are >1.0%
- The matrix material is **not** a light matrix
- Elements with atomic number greater than iron (Z = 26) exceed several percent

To use the **Industrial Bulk Re/Ta/Hf Testing Mode**, simply select the **Industrial Bulk Re/Ta/Hf** icon from the **Bulk Mode Menu** to place your instrument into **Industrial Bulk Re/Ta/Hf Testing Mode.** In **Industrial Bulk Re/Ta/Hf Testing Mode**, all other programmed elements are active in addition to Ta, Hf, or Re (e.g. Ti, Cr, Mn, Fe, Co, Ni, Zr, Nb, Mo, W, etc.). **Industrial Bulk Re/Ta/Hf Testing Mode** is recommended if you know, *without a doubt,* that your sample contains Ta, Hf, and/or Re and not Cu, Zn, and/or Pb. When your NITON XLp analyzer is operating in **Industrial Bulk Re/Ta/Hf Testing Mode**, the NITON XLp analyzer will not measure concentrations of Cu, Zn, or Pb in a sample. Once **Industrial Bulk Re/Ta/Hf Testing Mode** is selected, you may begin testing samples.

Use **Industrial Bulk Re/Ta/Hf Testing Mode** if:

- There is more Ta/Hf/Re than Cu/Pb/Zn in the sample
- The percentage of the elements of interest are >1.0%
- The matrix material is **not** a light matrix
- Elements with atomic number greater than iron (Z = 26) exceed several percent

Select the **Plastics Analysis** icon from the **Bulk Mode Menu** to place your instrument into **Plastics Analysis Mode.** Use **Plastics Analysis Mode** to find elements of interest in a plastic matrix. **Plastics Analysis Mode** is not available on this analyzer. For further information on **Plastics Analysis Mode**, contact Thermo or your local NITON representative.

Standard Bulk Mode

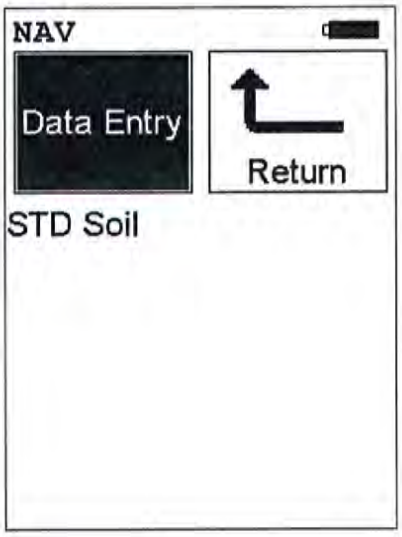


Figure 6-7. Standard Bulk Mode Menu

The **Standard Bulk Mode** is available from the **Bulk Mode Menu**. The **Standard Bulk Mode** allows you to perform tests on soil and other bulk samples without adjusting for a particular matrix. The **Standard Bulk Mode** is ideal for finding contaminants in soil. The **Standard Bulk Mode** uses Compton Normalization to automatically adjust for the effects of the matrix. From the **Standard Bulk Mode Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **Standard Bulk Mode Menu** to go to the "The Data Entry Screen" on page 6-29, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

This mode of operation is optimum for any sample whose elements of interest are present at less than 1%. **Standard Bulk Mode** utilizes the Compton Scatter (Inelastic Collisions) of a particular sample. Compton scatter occurs when primary X-rays do not cause fluorescence but instead collide with the atoms of the sample. The Compton Scatter that occurs is directly proportional to the density (average atomic number (Z)) of the sample. A light matrix material, such as an oil or sand, will have a much greater scatter than that of a heavy matrix, such as ore. The analyzer measures this scatter peak and automatically adjusts the concentration based

on the matrix of the material and allows for the analysis of a bulk sample without the use of site specific calibration standards. This mode is used chiefly for the analysis of contaminants in soils.

Industrial Bulk Cu/Zn/Pb Testing Mode

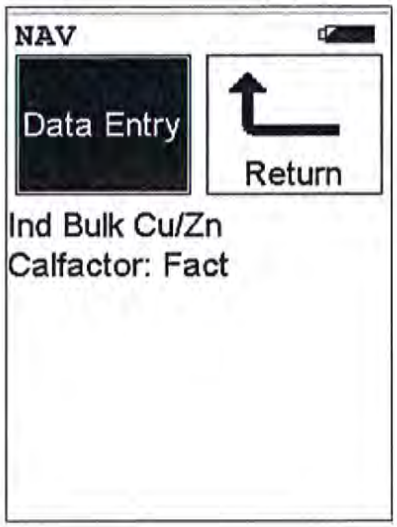


Figure 6-8. Industrial Bulk Cu/Zn/Pb Testing Mode Menu

The **Industrial Bulk Cu/Zn/Pb Testing Mode** is available from the **Bulk Mode Menu.** The **Industrial Bulk Cu/Zn/Pb Testing Mode** allows you to perform tests on soil and other bulk samples without adjusting for a particular matrix. **Industrial Bulk Cu/Zn/Pb Testing Mode** is ideal for finding concentrations of analytes in rock or soil. This mode of operation is optimum for any sample whose elements of interest are present at 1% or greater. **Industrial Bulk Cu/Zn/Pb** Testing Mode utilizes Fundamental Parameters to analyze the sample. From the **Industrial Bulk Cu/Zn/Pb Testing Mode Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **Industrial Bulk Cu/Zn/Pb Testing Mode Menu** to go to the "The Data Entry Screen" on page 6-29, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

Industrial Bulk Re/Ta/Hf Testing Mode

Figure 6-9. Industrial Bulk Re/Ta/Hf Testing Mode Menu

The **Industrial Bulk Re/Ta/Hf Testing Mode** is available from the **Bulk Mode Menu**. The **Industrial Bulk Re/Ta/Hf Testing Mode** allows you to perform tests on soil and other bulk samples without adjusting for a particular matrix. **Industrial Bulk Re/Ta/Hf Testing Mode** is ideal for finding concentrations of analytes in rock or soil. This mode of operation is optimum for any sample whose elements of interest are present at 1% or greater. **Industrial Bulk Re/Ta/Hf Testing Mode** utilizes Fundamental Parameters to analyze the sample.From the **Industrial Bulk Re/Ta/Hf Testing Mode Menu**, you can immediately initiate a sample test using the proper preconditions for operation, enter data about your sample using the **Data Entry** icon, or return to the **Main Menu**.

Select the **Data Entry** icon from the **Industrial Bulk Re/ta/Hf Testing Mode Menu** to go to the "The Data Entry Screen" on page 6-29, to input data about the sample which you are testing. The data you enter will be associated with the next sample you test.

Analyzing Bulk Samples

CAUTION After being powered on, the NITON 300 Series Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your instrument warm up for ten minutes after start up, before testing is begun. ♦

There are five different methods of operation for taking a sample measurement, and your analyzer will be configured to use one of those methods for soil samples, depending on the regulatory requirements of your locality. These methods are:

- Trigger-Only method. With the Trigger-Only method, you only need to place the measurement window close to the sample to be analyzed and pull the trigger for sample analysis to be initiated.

- Trigger-and-Proximity-Sensor method. With the Trigger-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the instrument, then pull the trigger for sample analysis to be initiated.

- Momentary-Trigger-Touch-and-Proximity-Sensor method. With the Momentary-Trigger-Touch-and-Proximity-Sensor method, you must place the measurement window against the surface to be analyzed to engage the proximity sensor on the front of the instrument, then pull the trigger.

- The trigger may be released and the reading will continue until you release the proximity button, or other criteria (such as Max Time) are reached. This is the primary mode of operation for the XLp 300A/700A Series Lead-in-Paint Analyzer in the USA.

- Trigger-and-Interlock method. With the Trigger-and-Interlock method, you need to place the measurement window close to the sample to be analyzed, press and keep pressing the interlock button at the rear of the instrument with your free hand, then pull the trigger for sample analysis to be initiated.

- Trigger-Interlock-and-Proximity-Sensor method. With the Trigger-Interlock-and-Proximity-Sensor method, you must place the measurement window against the sample to be analyzed to engage the proximity sensor on the front of the instrument, press and keep pressing the interlock button at the rear of the instrument with your free hand, then pull the trigger for sample analysis to be initiated.

- With any of these methods, analysis will stop if any one of the preconditions are violated. For example, with the Trigger-Interlock-and-Proximity-Sensor method, if the trigger or the Proximity Sensor or the Interlock is released, the reading will stop immediately, and the shutters will close.

After your NITON XLp analyzer is calibrated, initiate a sample reading using the appropriate method. If you attempt to initiate a sample reading using a different method, the analyzer will inform you that one or more of the preconditions need to be met in order for sample analysis to begin.

Note The three LED lights will blink during calibration or whenever there is a shutter open. ♦

WARNING! The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, all the shutters will close, and the measurement will end. The three LED lights will stop blinking, the shutters will close, and the measurement will end. The flashing of the LED lights is not synchronized to minimize power consumption. ♦

WARNING! The three LED warning lights are designed to blink only during a measurement, where one or more of the shutters are open and the trigger depressed. If the LED lights blink at any other time, disconnect the battery pack immediately, place the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, or your local Authorized NITON Analyzers Service Center. ♦

Your NITON Analyzer will display the Results Screen throughout the duration of each reading, The Results Screen is updated regularly throughout the reading. When the reading is complete, a final screen update will appear, and your NITON analyzer will display the final results of the measurement which has just been completed.

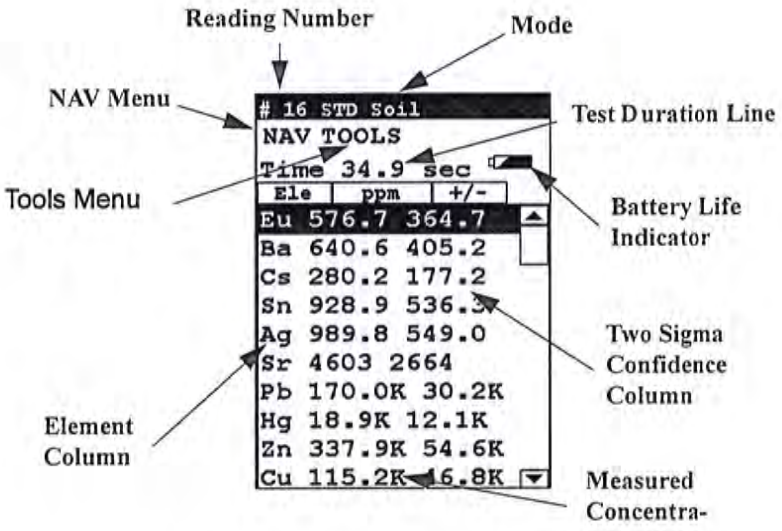


Figure 6-10. The Results Screen

The Results Screen displays the following information:

- The Reading Number line shows a number automatically assigned by your NITON analyzer in order to uniquely identify each reading. The reading number automatically increments up by one with each successive reading.
- The Nominal Seconds Test Duration line shows the number of nominal (source) seconds elapsing since the initiation of the reading. Nominal seconds are true, clock seconds slowed down to compensate for the electronic dead-time that occurs when the NITON XLp is taking a measurement.
- The Mode line displays the test mode in use during the measurement.
- The **Element** (left) column shows the elements that have been detected in the sample.
- The **Concentration Level** (central) column shows the concentration levels of the corresponding elements in percentages.
- The **Confidence** (right) column displays the 2 sigma (95%) confidence interval for the corresponding elements.

To Prepare or Not to Prepare - In Situ vs. Ex Situ

Bulk media are generally tested either on-site (*in situ*) for screening purposes, or removed and prepared (*ex situ*) to enhance the accuracy of the measurement. The degree of preparation may vary depending on the accuracy desired, the characteristics of the sample, and the characteristics of the site.

Understanding the advantages of *in situ* testing and of various degrees of preparation is crucial to obtaining useful data. *In situ* measurements should be used to profile an area, locate sources of contamination, determine the boundaries of contaminated areas, or gather data that will subsequently be used to design a sampling plan.

In situ measurements are usually only approximations, though they may correlate very well with lab analysis if the site tested is highly homogeneous. If the site is non-homogeneous, as is often the case, then in situ measurement results may differ greatly from laboratory obtained results. Both sets of results may be correct. The difference arises from the fact that actual samples tested were different.

Analysis of Unprepared Samples – In Situ

Figure 6-11. In-Situ Soil Testing

Screening Techniques

Use direct measurement when you need to determine whether an element is present (rather than in accurately measuring how much is present). Use preliminary direct measurements to survey a site quickly even if you intend to take samples

Testing In Situ

WARNING! When taking samples from a site where toxic chemicals may be present, always use gloves and respiration equipment for your own protection. ♦

1. **Select a measurement site, and clear away any surface debris and vegetation.**

Note Valid results will depend on a sufficient and appropriate selection of sites to sample. Lead-in-soil from paint, for instance, will usually be concentrated within a few feet of the painted structure. ♦

2. **Choose an area to test where the measurement window of the analyzer will be flush with the test media. Position the nose against the surface to be analyzed and initiate a reading by squeezing the shutter release, and firmly pressing the instrument flat against the surface.**

3. **After the test, inspect the nose of the instrument for contamination, which may affect future analysis. If the nose appears to be soiled, clean it with a soft cloth or tissue.**

WARNING! Always treat radiation with respect. Do not hold your instrument near the Kapton window during testing. Never point your instrument at yourself or anyone else when the shutter is open. ♦

Note Never use in situ testing with field portable XRF when comparing field results with laboratory results to justify XRF usage. Always collect samples and prepare them before testing. Refer to the instructions on sample collection and preparation in *Ex Situ* Testing. ♦

On-site vs. Lab Analysis

When comparing field screening to laboratory analysis, it is preferable to compare results obtained from the same samples. Start by collecting a sample large enough to be divided into two parts, with one portion stored for future reference and the other submitted to a laboratory for independent analysis. For best results, follow the complete protocol for sample preparation, including drying and grinding of the sample. Grinding is essential for homogenizing the sample, ensuring that the portion sent to the lab is the sample as that analyzed on-site.

If you must test for *in situ* performance evaluation, take several XRF readings bracketing a spot. Then take a sample for laboratory testing from that spot. For further discussion of field screening, please refer to EPA Method 6200, "Field Screening Using A Field-Portable XRF," which can be obtained from NITON Corporation's Customer Service Department. EPA accepts field screening results using portable XRF provided that Method 6200 protocol is followed.

In Situ Depth Profiling

XRF analysis for soil is a surface technique. To perform a depth profile, remove a vertical slice of soil and test several samples taken from different depths. This procedure will yield information, rapidly, about the depth of contamination.

Figure 6-12. In-Situ Depth Profiling

Analysis of Prepared Samples – Ex Situ

Figure 6-13. Ex-Situ Analysis of Prepared Samples

Analysis of bagged bulk samples

Sometimes it is convenient to measure samples in plastic bags. Without further preparation of the sample, you can screen the site by testing each bag. Because you are testing through a bag, test results will tend to be lower than test results obtained from direct analysis. This effect will vary depending on the element analyzed and the thickness of the plastic through which the sample is tested. Bagged samples can be retested and/or be further prepared and then retested, allowing samples of particular interest to be more accurately analyzed.

Sample Collection

Examine the site for differences in soil characteristics before sampling. Valid results depend on a sufficient and appropriate selection of sites to sample. Incorrect sample collection may give rise to misleading or meaningless results, regardless of the analysis method. Delineate sections with different characteristics and treat them as different areas. It may be desirable to subdivide larger areas even if they have the same characteristics to ensure a thorough examination.

Note When testing for lead-in-soil in a residential setting, it is standard practice to sample the top 4 to 6 inches of soil. ♦

Lead is usually concentrated near a building with lead paint (within 4–6 feet), and appropriate sample areas should be defined. Samples from the following areas should also be collected and combined separately:

- near painted structures
- near roads and driveways
- near to where various types of waste have been stored, or
- near pressure-treated wood

The soil probe or sampling tube is a very convenient sampling tool. It quickly takes accurate and consistent samples, and may be inserted to a marked depth to removes the same amount of soil at each insertion. In addition, there also are core-sampling devices that can remove an intact cylinder of sample material. If no sample tool is available, a shovel, spade, narrow (1-1/2 inch) garden trowel, or other sampling tool is adequate to take a soil slice of the required depth.

WARNING! When taking samples from a site where toxic chemicals may be present, always use gloves and respiration equipment for your own protection. ♦

Individual samples may require reduction in size if they were taken with a spade, trowel, or other large sampling tools. Reduce the samples by taking a vertical slice (so it is representative of the entire sample) about one inch wide.

Prepare a composite sample from each predetermined area. A composite sample of an area consists of many smaller samples from different sections of that area. These samples should consist of vertical columns of material approximately 1 inch in diameter. The length of each column should be about 6 inches. The elements you wish to measure and the local history will determine the depth at which you need to sample. For example, lead from paint is usually concentrated within the top 1–4 inches.

Do not combine samples from different areas. A composite sample taken from two distinctly different areas is not representative of either area.

Place the samples in a clean pail. Then mix the sample thoroughly by stirring and by rotating the pail at an angle of 45 degrees. Don't shake the sample—you do not want to stratify the sample.

A sample should contain at least 50–100 grams of soil. Once the sample has been thoroughly mixed, place it in a clean bag that can be closed securely (with a twist tie for example). Zip Lock style bags are very convenient, though extra care must be taken to ensure the bags do not unseal, which could ruin the samples.

Make certain to label each bag thoroughly. Common information included on each bag includes the person and/or the company who collected the sample, the location and area where the sample was taken, and the date the sample was collected.

Testing Samples in Bags

Flatten the bag of soil to form a continuous uniform layer of at least 1 cm. (0.4 inch) thickness. Place the measurement window of the NITON XLp Lead Paint Analyzer on the bag. Then follow *In Situ* testing instructions from page 5.

1. **Choose a convenient work surface. This surface should be free of material containing elements that may be detected by the analyzer. When uncertain of the work surface, test the surface with the analyzer.**

2. **Flatten the bag of soil to form a continuous uniform layer of at least 1 cm. (0.4 inch) thickness. Place the nose of the NITON analyzer on the bag. Be careful of bags with manufacturers printing or labeling surfaces. This printing often contains detectable elements, most notable Ti, and should be avoided when possible.**

3. **Position the instrument against the surface of the bagged sample, and initiate a reading by squeezing the shutter release, and firmly pressing the instrument flat against the sample. The trigger and the proximity sensor must both be engaged before the shutter will open and the measurement initiated.**

WARNING! Do not hold bagged samples while testing. ♦

Prepared sample analysis is the most accurate method for determining the concentration of elements in a bulk medium using the instrument. Sample preparation will minimize the effects of moisture, large particle size, variations in particle size and sample non-homogeneity.

Note: More sample preparation (drying, milling and sifting) will yield greater accuracy. The drier, finer, and more homogeneous the particles, the better the measurements.

Preparing Bulk Soil Samples

Thermo NITON recommends a specific sample protocol. Following this protocol for preparing and testing samples is vital for achieving a level of accuracy comparable with laboratory results.

The equipment you need to prepare samples is included in your kit. Among these are a mortar and pestle, several different sized metal sieves, cups to hold the samples, and the soil test platform.

CAUTION All test equipment must be kept clean to prevent contamination of samples. ♦

Cleaning Your Equipment:

The mortar, pestle, and grinding mill may be cleaned with dry paper towels. You can also clean the mortar, pestle, and the mill's container with water, but be sure each is absolutely dry before using them on another sample. The mortar and pestle may be cleaned by grinding clean, dry sand in the mortar. Use the short bristle brushes (included in your Soil Testing Kit) to clean the sieves. If you have an electric soil grinder in your kit, when the soil grinder blades wear out, unbolt the worn blades and replace them. Call the Thermo Sales Department at 1-800-875-1578 for replacement blades.

Note Using the soil grinder may artificially increase the amount of Fe in soil samples. ♦

Sample Preparation

Prior to analysis, the material should be dry and well homogenized. Ideally, the entire sample should be dried to constant weight, sifted to remove gravel and debris, and ground or milled to a fine powder.

Dry the sample if it is moist and cohesive. The sample can be dried in any of several ways. Choose one of the following:

- Oven dry the sample for approximately 2 hours at 150° C, until the sample reaches a constant weight. Note: Oven drying is inappropriate when volatile compounds may be present in the sample. For example, lead present as tetraethyl lead would be driven off by the heat of drying. Some forms of mercury and arsenic are volatile. Air drying will preserve more of these volatile substances.
- Air dry the sample overnight at room temperature in a shallow pan.
- Stir gently and warm the sample in a pan over a hot plate or burner.

Coning and Quartering

You may need to divide your sample at various times during preparation. Coning and quartering is a method for dividing the sample into homogenous quarters.

- Pour the dry material slowly and carefully onto a flat sheet or pan, forming a symmetrical cone. Divide the cone into equal piles using a flat thin-bladed tool, such as a knife or ruler. Divide these in half again.
- Now you have four samples, each one-quarter the size of the original and each more homogenous than the original.
- Grind the sample to break up dirt clods and/or paint chips.

WARNING! Grinding and sifting dried samples produces dust. Even clean soil contains silica, which may be hazardous when airborne. Prepare all samples in a ventilated area; wear a mask, gloves, and an apron; and spread a drop cloth. ♦

Sift using the #10 (2mm) mesh and separate out the larger pieces (stones, organic matter, metallic objects, etc. Examine the larger particles by eye (look for paint chips), but do not include in the sample. Grind the sample again so its particles will be finer and more homogenous. Use mortar and pestle, or an electrically powered grinding mill. Sift at least 10 grams of the sample through #60 (250 µm) and #120 (125 µm) mesh. Re-grind the un-passed material until the entire fraction is able to pass. Mix the resulting sample.

Placing the Sample in an XRF Sample Cup

The container used to hold the sample will affect the accuracy of the measurement. Use a container with as thin-walled a window as is convenient and use the same kind of container and window for each sample. Consistency and careful attention to detail are keys to accurate measurement.

Note The sample container should be a sample cup of a type that can be filled from the rear; that is, the side opposite the window (e.g. Thermo NITON Part Number 187-466). Thermo recommends using a 1/4 mil Mylar film (e.g. Thermo NITON Part Number 187-492). A supply of cups and films are included.

Place a circle of Mylar film on top of an XRF sample cup. This film goes on the end of the cup with the indented ring. Thermo recommends preparing the cup ahead of time, if possible.

Secure the film with the collar. The flange inside the collar faces down and snaps into the indented ring of the cup. Inspect the installed film window for continuity and smooth, taut appearance.

Set the cup on a flat surface film-window-side down. Fill it with at least five grams of the prepared sample, making sure that no voids or uneven layers.

Lightly tamp the sample into the cup. The end of the pestle makes a convenient tamper.

Place a filter-paper disk on the sample after tamping it.

Fill the rest of the cup with polyester fiber stuffing to prevent sample movement. Use aquarium filter or pillow filling as stuffing. A small supply of stuffing comes with your bulk sample kit.

Cap the cup.

Place a label on the cup. Using a pen with indelible ink, write identifying information on the cup. Keep a record of the sample designation, the site and location, the date of the sample, and any other relevant comments.

Cup is ready for testing.

Preparing Liquids, Sludge or Dust

Liquids Fill an XRF sample cup with the liquid to be tested (do not pad the sample with cotton). The cup must be full so it is best if some liquid is allowed to overflow when the cap is put on.

Sludge Sludge can be placed directly into an XRF cup for screening. This is considered in-situ testing because no attempt has been made to prepare the sample. For more accuracy, the sludge can be dried, sieved, and ground.

Dust Vacuum dust with a household vacuum cleaner and use large dust samples taken from the vacuum cleaner bag. Remove fibers, hairs, and debris. At least three grams of dust are needed to assure accurate analysis. Samples as small as one or two grams may be measured with less accuracy. Even smaller samples (0.3 to 1.0 grams) can be analyzed by applying a weight correction factor and by using a funnel to place the sample in the center of the sample cup.

Prepare in an XRF sample cup and test the same way you would with a soil sample. For risk analysis, it is advisable to use a 60-mesh sieve to isolate and test only fine particles.

Standard Soil Mode

Thermo provides a set of three soil standards containing either NIST high, NIST medium, and NIST low, or containing NIST high, TILL-4, and NCS DC 73308. Use these standards to check the calibration of the instrument when testing in **Standard Soil Mode**. For the compositions of these standards, see Chapter 2 - page 66.

Note Although the standards do not contain every element that the Lead Paint Analyzer is capable of testing, and although the standards do contain elements that the analyzer is not capable of testing, it can be assumed that the XLp Environmental Analyzer will measure all elements correctly if it correctly measures those elements contained in the standards supplied. ♦

Test the standards regularly. Thermo recommends testing immediately after the instrument finishes self-calibration. Test the standard samples appropriate to the type of tests you are conducting, and once every 1–2 hours thereafter.

Note For defensible Quality Control, keep a record of the time and precision of every calibration, using the bar code system when possible. ♦

WARNING! Tampering with the 5,500 ppm (lead high) lead-in-soil standard may cause exposure to lead dust. Keep all standards out of the reach of children. ♦

CAUTION Never tamper with Test Standards. They should not be used unless they are completely intact. ♦

During each test, the instrument looks at the full range of x-ray spectrum and continuously corrects for cross-element interference.

Testing Prepared Samples

Set the test platform on a flat, solid surface. Slide out the drawer and place the sample cup in the holder and slide the drawer shut. Insert the instrument into the nose cone adaptor so that the LCD screen is facing the side that the test platform drawer is on, and follow in-situ bulk sample instructions

FP Soil Testing Modes

These modes of the operating software are intended primarily for the detection of metal concentrations greater than 1% in light or heavy matrices. The full fundamentals parameter (FP) algorithm accurately measures elemental concentrations from minor levels to 100%, and automatically corrects for inter-element effects. However, elements lighter than calcium cannot be detected by XRF and light element combinations, such as oxides, carbonates, and silicates are common matrix components. To fine-tune results, you may enter calibration factors for individual elements to adjust for effects of light element interference. These calibration factors are linear corrections, which adjust the FP calculation. Calibrations only need to be entered once per matrix. However, as matrices can vary considerably from one sampling area to another, it is recommended that new calibrations be done for each change in matrix.

Using both the ^{109}Cd and ^{241}Am sources, concentrations for the following analytes can be determined:

Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, W, Pb, Bi, Zr, Ta, Nb, Mo, Sn, and Ag.

All concentrations are displayed in units of wt.%.

Note For defensible Quality Control, keep a record of the time and precision of every calibration, using the bar code system wherever possible. ♦

WARNING! Tampering with the 5,500 ppm (lead high) lead-in-soil standard may cause exposure to lead dust. Keep all standards out of the reach of children. ♦

CAUTION Never tamper with Test Standards. They should not be used unless they are completely intact. ♦

During each test, the instrument looks at the full range of x-ray spectrum and continuously corrects for cross-element interference.

Testing Prepared Samples

Set the NITON test platform on a flat, solid surface. Slide out the drawer and place the sample cup mylar side facing up in the holder and slide the drawer shut. Insert the instrument into the nose cone adaptor so that the LCD screen is facing in the same side as the test platform drawer and follow ex-situ bulk sample instructions

The Data Entry Screen

The **Data Entry Screen** is accessed whenever you select the **Data Entry** icon from any screen. This screen allows you to input data in several different fields, or categories, concerning your sample, in several different ways:

- By selecting the Virtual Keyboard button and typing the parameter in using the **Virtual Keyboard**.
- By scanning in the parameter name using the integrated bar code scanner.
- By creating a new, or editing your analyzer's existing, '.ndf' file through the NDT program. You can then select from the various custom options you have created using the Drop-down List button.

These fields are saved along with the subsequent reading, and allow you to associate important information about the sample directly with the reading, so that you have a full description of the sample tied into the reading itself.

Once you have input data into a field, that information carries over into the next reading, so that you only have to input the information that has changed since the last reading. For example, if you are analyzing several samples of a particular lot, you only need to input the lot information once during that series of readings, changing only the sample name.

Standard Soil Mode Data Entry Screens

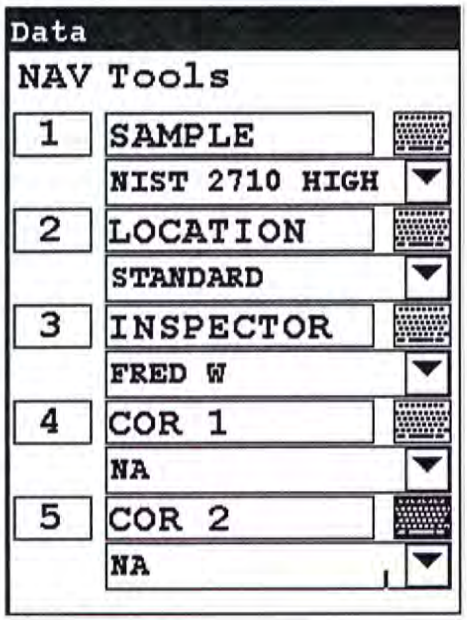


Figure 6-14. Data Entry Screens Standard Soil Mode - First Page

This is the first section of the **Standard Soil Data Entry Screen**. There are five parameters in this section.

- Selecting **Sample** allows you to input the sample name parameter
- Selecting **Location** allows you to input the particular location information, if known
- Selecting **Inspector** allows you to specify the inspector.
- Selecting **COR 1** allows you to specify the first coordinate.
- Selecting **COR 2** allows you to specify the second coordinate.

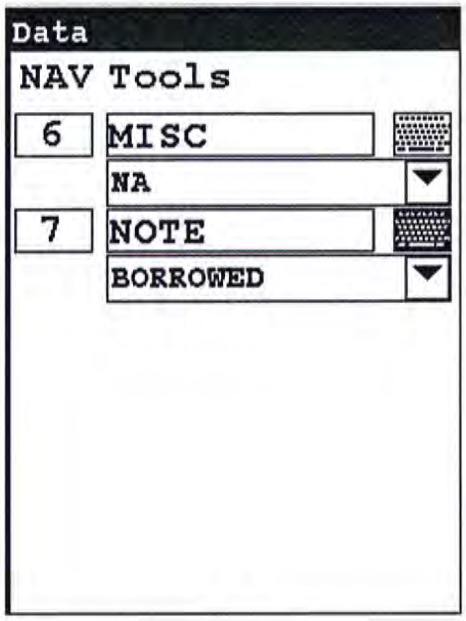


Figure 6-15. Data Entry Screens Standard Soil More - Second Screen

his is the second section of the Standard Soil Data Entry Screen. There are two parameters in this section.

Selecting **Misc** allows you to input miscellaneous parameters

Selecting **Note** allows you to input notes on the sample

Data Entry Screens for all other Bulk Modes

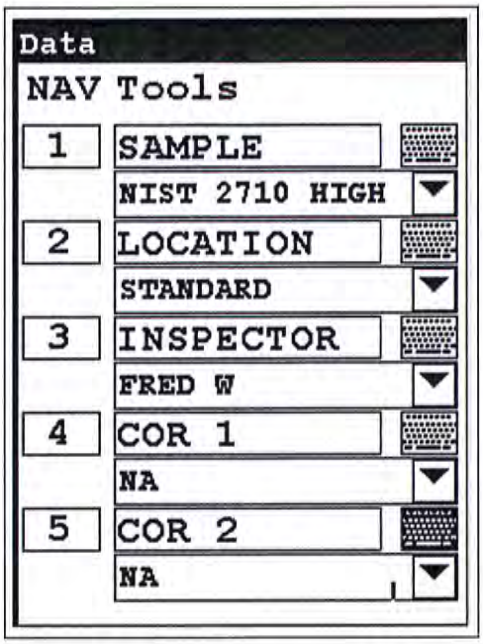


Figure 6-16

Figure 6-16. Data Entry Screen - Other Bulk Mode

This is the only section of the **Data Entry Screen**. There are five parameters in this section.

- Selecting **Sample** allows you to input the sample name parameter
- Selecting **Location** allows you to input the particular location information, if known
- Selecting **Inspector** allows you to specify the inspector.
- Selecting **Misc** allows you to input miscellaneous parameters
- Selecting **Note** allows you to input notes on the sample

Navigating the Data Entry Screen

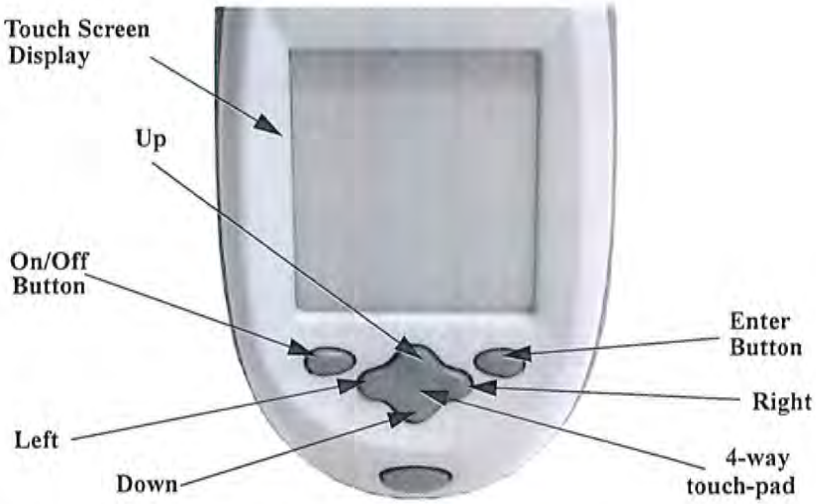


Figure 6-17. The Control Panel

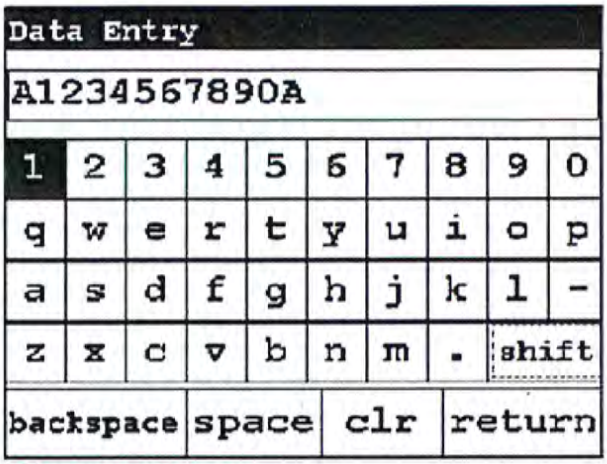
The following description of screen navigation using the control panel assumes that the analyzer is held so that the display is held upright as in Figure 6-17.

- To move from column to column, use the Right and Left portion of the 4-way touch pad.
- To move from row to row, use the Up and Down portions of the 4-way touch pad.
- To select the highlighted option, press the Enter button on the control panel.

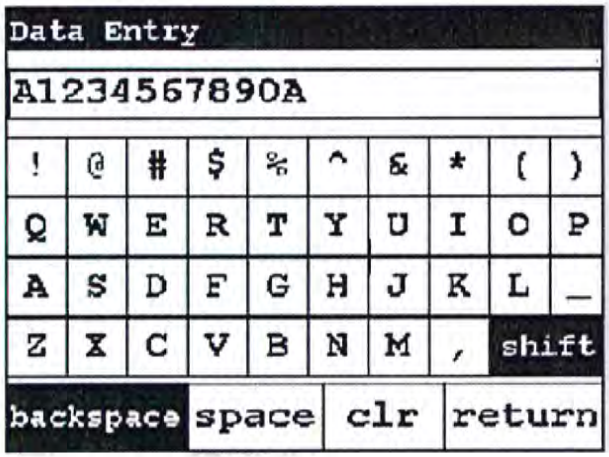
The *Data Entry* Screen is divided into sections of 5 setting parameters. By using the Down portion of the 4-way touch pad when you are on the last row of a section, the display will change to the next section. By using the Up portion of the 4-way touch pad when you are on the first row of a section, the display will change to the previous section.

By selecting the *On/Off* button, you can exit the *Data Entry* Screen.

The Virtual Keyboard



Lower Case Virtual Keyboard



Upper Case Virtual Keyboard

Figure 6-18. The Virtual Keyboard

The Virtual Keyboard is a full alphanumeric keyboard which appears on the LCD Touch Screen Display. You can use the Virtual Keyboard either with the four-way touch pad and control panel buttons, or using the touch screen display directly.

At the top of the screen is the data field you are entering data for, in this case, "Sample", Directly underneath is the data you are entering, in this case "17-4 PH SS", On this line also is the underscore cursor. This graphically shows where the next character will be placed. Up to 25 characters can be stored in the data fields.

Next is the Virtual Keyboard itself, with numbers 0-9, letters A-Z, and special characters *,<,>, and -.

Last is the control button line. This contains the screen buttons for Return and Clear.

When using the four-way touch pad and control buttons, pressing the On/Off button allows you access to the Return and Clear screen buttons. The Return screen button will enter the data and return you to the Data Entry Screen, while the Clear screen button will clear the data you have

All screen areas can be directly accessed using the LCD Touch Screen.

NITON Test Platforms

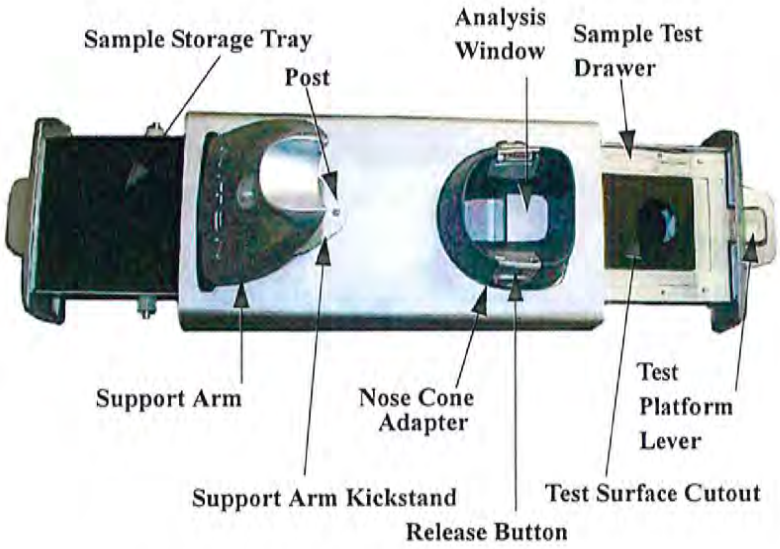


Figure 6-19. NITON Bulk Mode Test Stand

The NITON Bulk Test Stand is designed to facilitate bulk and thin sample testing using NITON Analyzers.The NITON Portable Test Stand is designed for bulk and moderate sized samples, and is portable.

Figure 6-20. NITON Portable Test Stand

NITON RCRA Verification Response Sample

This sample contains 7 of the 8 RCRA metals as follows:

As, Ba, Cd, Cr, Pb, Se, and Ag

NITON may recommend the analysis of this sample if you question the data that the instrument is displaying for the above elements.

Chapter 7 Precious Metals Mode

Using Precious Metals Mode

Precious Metals Mode is not enabled on your Niton XLp analyzer. For more information on this feature please contact Thermo Scientific's Customer Service Department in the United States, Toll free, at (800) 875-1578, or outside the United States at + 1-978-670-7460 or your authorized Niton Analyzer Service Center.

Chapter 8 Bluetooth

Setting up Bluetooth

Bluetooth Wireless Networking enables you to connect to your computer and other Bluetooth-enabled devices such as printers and GPD S devices without the need of cabling, ports, or hubs.

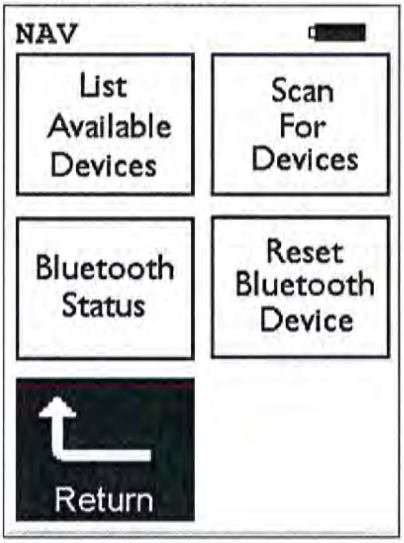
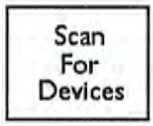


Figure 8-1. The Wireless Setup Menu

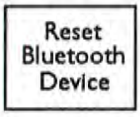
To access the Bluetooth Wireless Networking, select the Wireless Setup icon from the Instrument Setup Menu.

Select the **List Available Devices** icon to show a list of Bluetooth devices previously discovered. The Bluetooth devices listed are only those which were present at the last time you ran a discovery scan for Bluetooth devices, as the list is not automatically updated. Selecting the **List Available Devices** icon brings up the **Available Devices** screen. From the list, you can connect your analyzer to those devices.



Select the **Scan For Devices** icon bring up the **Bluetooth Search Screen**, enabling you to initiate a discovery scan of Bluetooth devices in the operational area. This scan will find all appropriate Bluetooth devices in the operational area, enabling you to connect to those devices.

Select the **Bluetooth Status** icon to view the current status of your Bluetooth connections on the **Bluetooth Status** screen. The Bluetooth Status screen will display your analyzer's serial number, connection status, the transfer rate, and your analyzer's address



Select the **Reset Bluetooth Device** icon to initiate an immediate reset of the Bluetooth Wireless Networking. Selecting the **Reset Bluetooth Device** icon will clear out old settings and data, as well as enabling you to switch between Bluetooth and standard serial cable. While resetting, your analyzer will show the following screen:

Figure 8-2. Bluetooth Reset Alert

Available Devices Screen

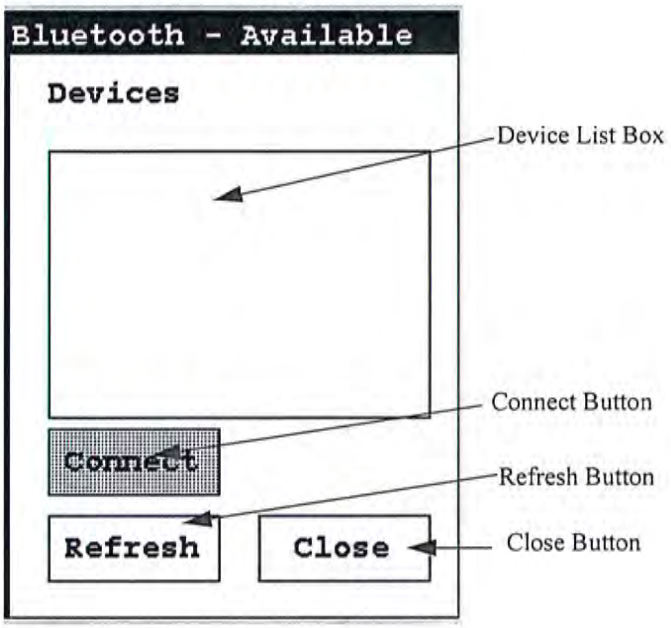


Figure 8-3. Available Devices Screen

Under "Devices," in the Device List Box, the Available Devices Screen lists all known applicable Bluetooth devices in the area found during the last refresh or scan.

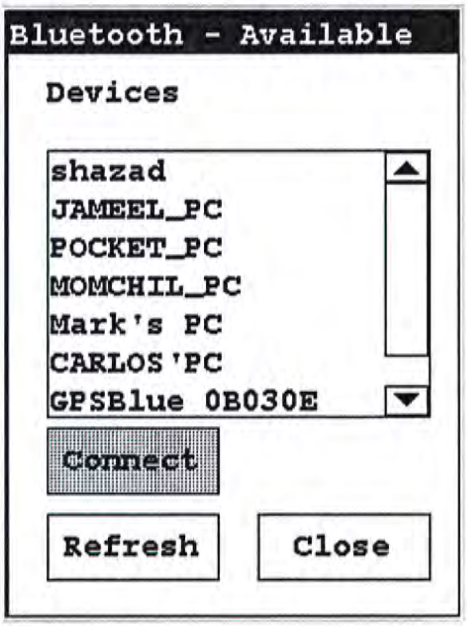


Figure 8-4. Example Device List

- Selecting the Refresh Button initiates a scan of the area for new Bluetooth devices. Devices no longer present are removed.

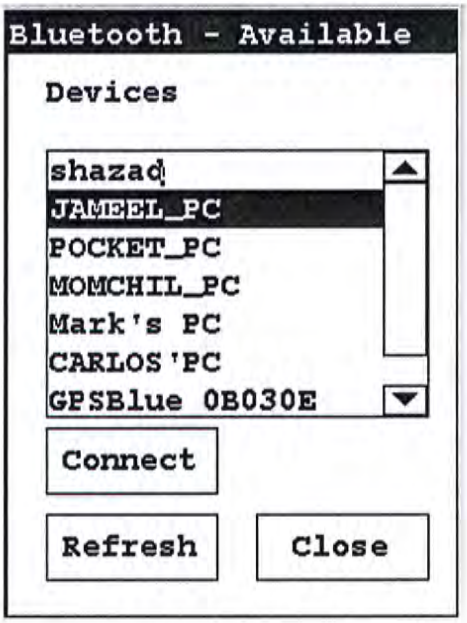


Figure 8-5. Available Device Refresh

- Selecting a listed Bluetooth Device enables the Connect Button.
- Selecting the Connect Button will connect your analyzer to the selected device. See the Connected Screen.

Bluetooth Search Screen

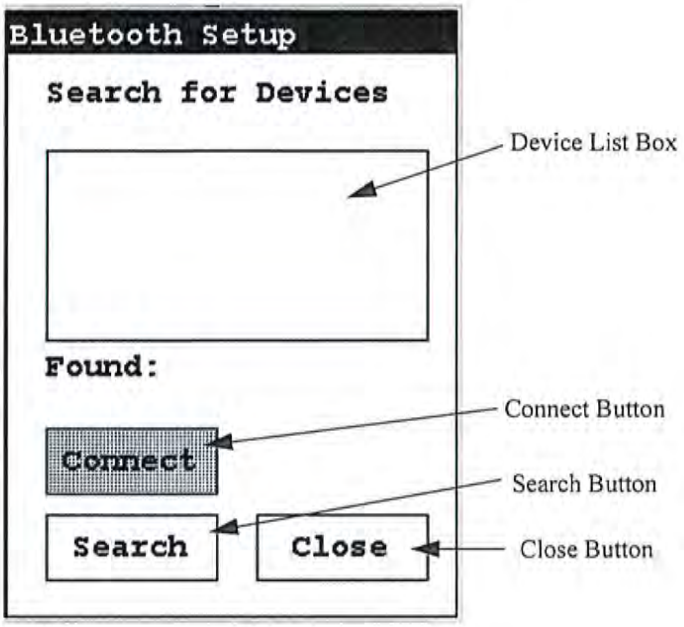


Figure 8-6. Bluetooth Search Screen

The **Bluetooth Search Screen** does not retain information about previously detected Bluetooth Devices. Each time the **Bluetooth Search Screen** is opened, the Device List Box is empty.

- Selecting the Search Button initiates a scan for Bluetooth Devices in the area.

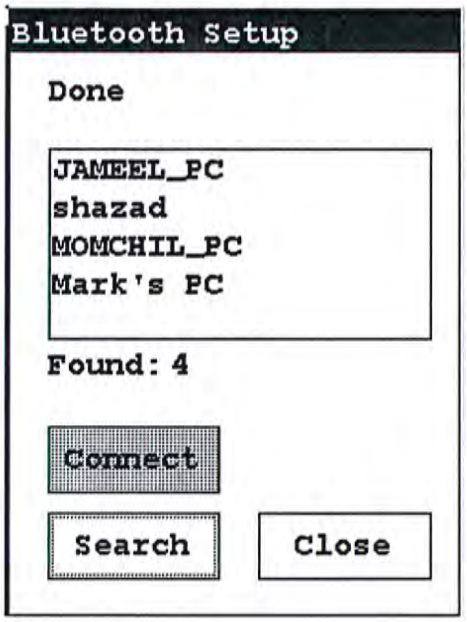


Figure 8-7. Example Search List

Depending on where and when the Search Scan is conducted, certain devices may or may not be detected. You can select a device and connect to that device in exactly the same manner as in the **Available Devices Screen**, once the search is finished.

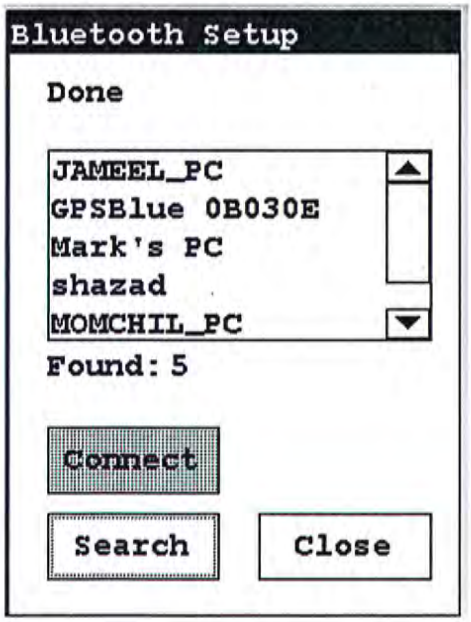


Figure 8-8. Search List with New Device Found

The Connected Screen

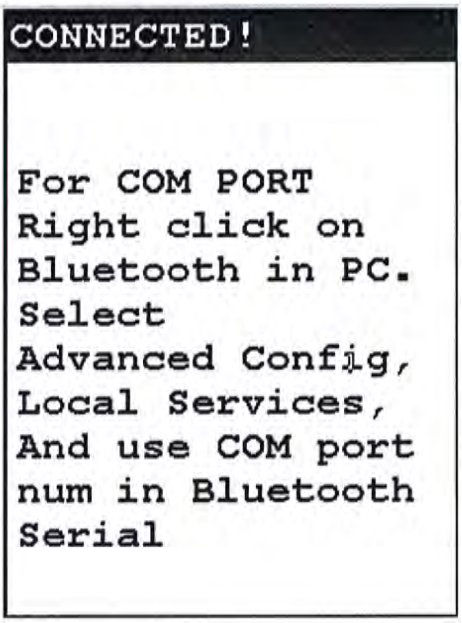


Figure 8-9. The Connected Screen

When you have connected your analyzer to a Bluetooth Device, you get the Connection Screen. The Connection Screen serves as a reminder of what needs to be done to use the connection. With simple devices like GPS devices, a notification that you are connected is given, and everything just works, but working with a PC is a bit more complex.

In order to use a Bluetooth Serial Connection with a PC, you need to know which COM port Bluetooth is connected through. To determine this, right click on the Bluetooth logo in your system tray on your PC. From the popup menu which appears, select Advanced Config., then select Local Services.

Figure 8-10. Advanced Configuration selection on PC

Service Name	Startup	Secure Connection	COM Port
Audio Gateway	Auto...	Not Required	
Headset	Auto...	Not Required	
PIM Synchronization	Manu...	Required	
Fax	Manu...	Required	
File Transfer	Auto...	Required	
PIM Item Transfer	Manu...	Not Required	
Dial-up Networking	Manu...	Required	
Network Access	Auto...	Required	
Bluetooth Serial Port	Auto...	Not Required	COM3

Figure 8-11. Bluetooth Service Listing on PC

In the Bluetooth Serial Port row, the COM port used by Bluetooth is identified. Use this port for any interactions between your analyzer and your computer, such as NDT or NDTr.

Make sure that the Secure Connection setting for the Bluetooth Serial Port is set to "Not Required."

Service Name	Startup	Secure Connection	COM Port
Audio Gateway	Auto...	Not Required	
Headset	Auto...	Not Required	
PIM Synchronization	Manu...	Required	
Fax	Manu...	Required	
File Transfer	Auto...	Required	
PIM Item Transfer	Manu...	Not Required	
Dial-up Networking	Manu...	Required	
Network Access	Auto...	Required	
Bluetooth Serial Port	Auto...	Not Required	COM3

Figure 8-12. Selecting Bluetooth Serial Port on PC

To edit the setting, double click the row.'

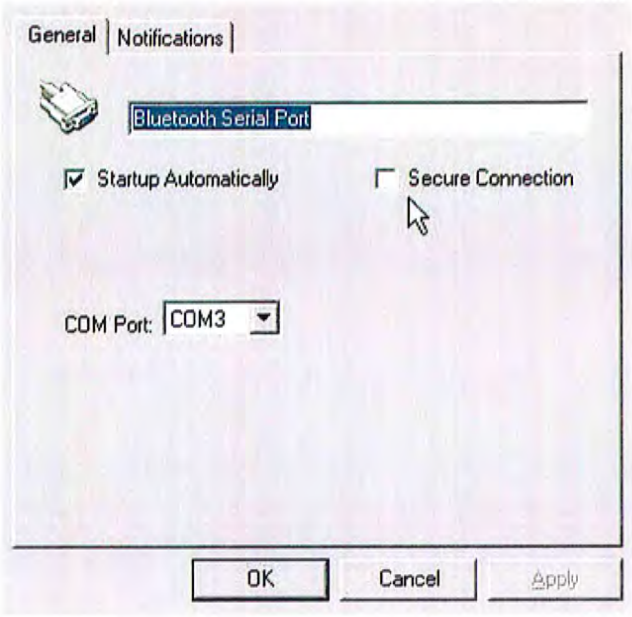


Figure 8-13. Changing the Bluetooth Secure Connection Checkbox on PC

Unselect the Secure Connections checkbox if it is already selected, then select the "OK" button.

Bluetooth Status Screen

The Bluetooth Status Screen enables you to see at a glance if and how your analyzer is connected to your computer.

Figure 8-14. Example Bluetooth Status Screen

The Bluetooth Status Screen shows your analyzer's identification label, its connection state, the speed of the communication port setting, your analyzer's network address, and the COD.

In Figure 8-14, the analyzer "XLt 8789" is not connected to any computer, was last in Slave state - i.e. the last connection was initiated by the computer and not by the analyzer, has a com port set to communicate at 115200 baud, has the unique network (MAC) address of 00A0960CE598, and has a COD (Class Of Device) of 00000000.

The Close screen button will return you to the Wireless Setup Menu.

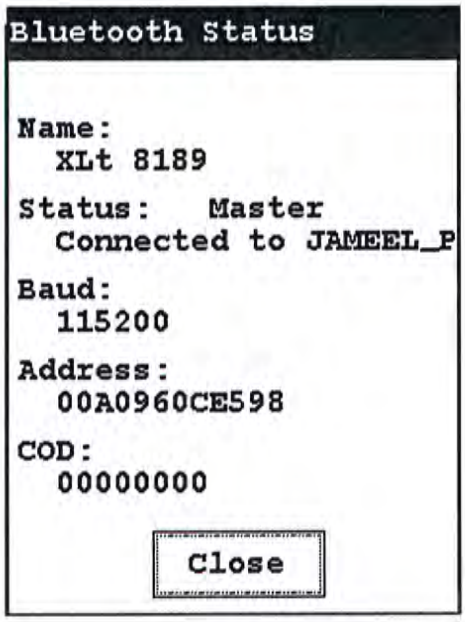


Figure 8-15. Second Example Bluetooth Status Screen

In another example, in Figure 8-15, the analyzer "XLt 8789" is connected to the computer JAMEEL_P, is currently in Master state - i.e. connection was initiated by the analyzer and not by the computer, has a com port set to communicate at 115200 baud, has the unique network (MAC) address of 00A0960CE598, and has a COD (Class Of Device) of 00000000.

GPS Data Tracking

Bluetooth equipped Niton XRF Analyzers are now capable of communicating with GPS modules and saving GPS coordinates with every reading. Follow the Bluetooth connection instructions found in the Users Manual to scan for and connect to a Bluetooth enabled GPS device.

Once connected, the GPS unit sends out a number of signals that can be read. The analyzer will display the relevant information from the GPS after connection, as shown in Figure 8-16

As shown in Figure 8-17, these coordinates can be viewed in the Data screen in entry positions eight, nine, and ten. (Scroll down to reach these fields.) When the results are downloaded using the NDT software the GPS coordinates are also stored and downloaded in data entry fields eight, nine and ten.

Figure 8-16. Example of GPS Data

Example of GPS Data

- 2:52:13 GMT - Greenwich Mean Time obtained from the GPS satellites.
- Lat: 3355.2607 -Latitude coordinate of current location. This should be read as:
  - All digits to the right of the decimal point are seconds.
  - First two digits to the left of the decimal point are minutes.

  - The next two or three digits to the left of the decimal point are degrees.
  - Thus 3355.2607 is read 33 degrees 55 minutes 26.07 seconds.
- N/S: S - Compass direction of Latitude.
- Long: 15111.594 - Longitude coordinate of current location.
  - All digits to the right of the decimal point are seconds.
  - First two digits to the left of the decimal point are minutes.
  - The next two or three digits to the left of the decimal point are degrees.
  - Thus 15111.594 is read 151 degrees 11 minutes 59.4 seconds.
- W/E: E - Compass direction of Longitude.
- Altitude: 17 - Height above sea level in meters.
- Quality: 1 - Quality of signal strength.
- Num Sat: 8 - Number of satellites signals being received by GPS. This number varies depending on your position, the current position of the satellites, and the signal strength.

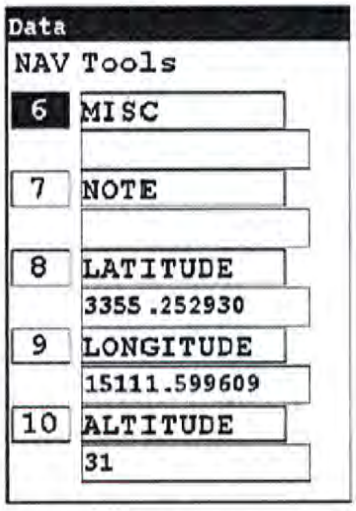


Figure 8-17. GPS Data Integrated Into Reading Data

GPS Options

The communication system standard required for compatibility is NMEA0182 ver. 3.0, using GPGGA, GPSA, GPRMC, and GPGSV formats. This type of GPS is most commonly used for motor and marine directional mapping systems.

Tested Units include:

Copilot BTGPS3

http://www.alk.com/copilot/pocketpc.asp

RoyalTek Star111

http://www.royaltek.com/index.php/content/view/98/80/

IOGEAR Bluetooth GPS

http://www.iogear.com/main.php?loc=product&Item=GBGPS201W6

Note These GPS systems have an accuracy of about 10 meters.

Chapter 9 Routine Maintenance Guidelines

Battery Pack and Battery Charger

Each Niton 300Analyzer is shipped with two lithium ion battery packs. When fully charged, the battery pack provides approximately 6-8-12 hours of use, depending on duty cycle.

There are two types of battery chargers distributed with Niton XRF Analyzers. The Trickle Charger shows a solid red light while charging, then a green light once charging is complete.

The other battery charger has 2 lights in the top of the battery charger. When connected to a wall outlet, the lights will display the following information:

Table 9-1. Battery Charger Status Display

Left Hand Light	Right Hand Light	Battery Status
ON	OFF	CHARGING
ON	ON	80% CHARGED
OFF	ON	COMPLETE
BLINK	BLINK	ERROR
OFF	OFF	NO BATTERY

Replacement battery packs (Niton part number 600-540) may be ordered from Thermo Scientific in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, or from your local Authorized Niton Analyzers Service Center.

Note Before beginning a test, be certain that the battery has sufficient charge. ♦

CAUTION Do not leave the battery pack connected to the charger for excessive periods of time. Overnight recharging is recommended. ♦

CAUTION Store the analyzer and the spare battery packs in a cool place, away from direct sunlight. ♦

Replacing The Battery Pack

1. Rest your Niton analyzer on a clean surface. Avoid damp or dusty environments.
2. Point the front of the analyzer away from you, and press in the battery housing latch.
3. Slide out the battery pack out toward you.
4. Fully insert the new battery pack, making sure that it seats properly.
5. Press in until the latch resets.

Figure 9-1. Location of the Battery Housing Latch

Recharging The Battery Pack

Fully recharging a Niton XLp battery pack takes approximately 2 hours.

1. Remove the battery pack from the analyzer.
2. Lifting the gasket flap, plug one end of the Battery Charger (AC adapter or 12V adapter) into the battery charger port.
3. Plug the other end of the AC adapter into a wall socket.

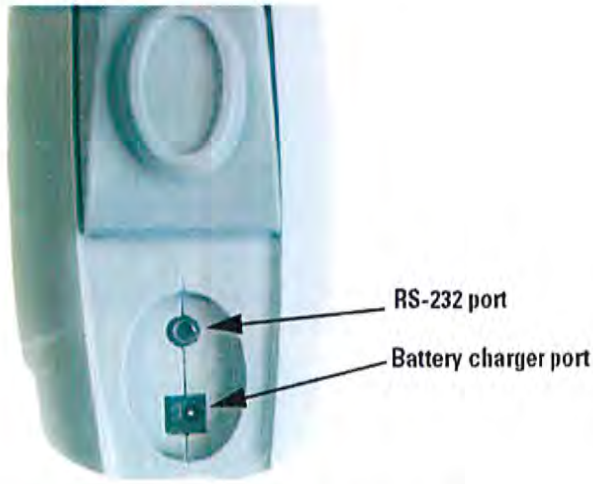


Figure 9-2. Location of the Battery Charger Port

CAUTION Do not force the charger into the RS-232 port! ♦

CAUTION Do not store battery packs or charger in direct sunlight. ♦

The AC adapter/Battery Charger comes with a convenient indicator light to inform you when the battery is fully charged.

CAUTION Do not let the battery pack recharge for excessive periods of time. ♦

Maintenance, Cleaning and Repairs

To ensure the reliability, durability, and performance of your Niton Analyzer, keep it clean—especially the transparent Kapton window covering the analysis window. Clean the Kapton window gently with a cotton swab. Clean the body of the analyzer with a soft cloth. Never use detergents, or solvents on your analyzer, or immerse your analyzer in water. If the Kapton window becomes frayed, ripped, or contaminated with metal particulates, replace it with a new window. Kapton windows (Niton P/N 187-095) may be ordered from Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 or from your local Authorized Niton Analyzers Service Center.

From time to time, your touch screen will need cleaning. Thermo Scientific recommends that you use a lens cleaning solution with a soft cloth. Do not use water to clean your Niton Analyzer.

Note All Service, except exterior cleaning and Kapton window replacement, must be performed by Thermo Scientific or an Authorized Niton Analyzers Service Center. Do not attempt to make repairs yourself. Opening the case of your analyzer will void the analyzer Warranty in its entirety. ♦

CAUTION Always obtain a Return Authorization (RA) number from Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your analyzer to the Niton Service Department or local Authorized Niton Analyzers Service Center. ♦

Replacing the Kapton Window

1. Remove the three Phillips head screws.

Figure 9-3. View of Face Plate and Kapton Window

2. Remove the face plate and place it face down.

Figure 9-4. Face Plate Removed showing Kapton Window on Reverse

3. Remove the old Kapton window.

4. Clean the back surface of the face plate and install the new Window.

5. Turn the face plate over and replace it on the analyzer's front end, fitting the plate carefully over the Proximity Button.

Figure 9-5. Fitting Face Plate over Proximity Button

6. Reinstall the three screws, being careful not to over-tighten them.

Figure 9-6. Replacing the Screws.

Storing and Transporting Your Niton XLp Analyzer

All Niton Analyzers are transported in waterproof, drop-resistant, fully padded carrying cases with padlocks. In most countries, Niton XRF analyzers may be transported by car or plane or shipped as an *ordinary* package. For most courier services, no special labels are required on the outside of the Niton analyzer case or on additional packaging.

Figure 9-7. The Niton Carrying Case

All padlocks are shipped with a default combination of "0-0-0". If you change this combination, please inform Thermo Scientific of the new combination if you return the unit for service.

To change the combination:

1. **Dial the default combination to open the lock, and pull out the shackle.**

2. **Rotate the shackle 180 degrees and push it down as far as it can go.**

3. **While holding the shackle down, rotate it 90 degrees back in either direction and release shackle.**

4. **Change the dial settings to the desired combination, record the combination, and without disturbing the dials, rotate the shackle back 90 degrees to the position it had in step 2.**

5. **Pull shackle out and rotate it 180 degrees and secure it. Your lock now has its own secret combination.**

CAUTION Always transport the unit in its padded carrying case, and store the Niton Analyzer in its case whenever it is not being used. •

CAUTION In most cases, no notification is required if transporting within state boundaries. This may not be the case when entering federal properties. •

CAUTION Within the United States, always keep a copy of the US DOT compliance statement in your Niton analyzer case at all times. A copy is included with your analyzer. •

CAUTION Always follow all pertinent local and national regulations and guidelines, wherever your XLp 300 series analyzer is transported or used. •

CAUTION Always obtain a Return Authorization (RA) number from Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your analyzer to the Service Department or to your local Authorized Niton Analyzers Service Center. •

CAUTION If you return your Niton analyzer without the carrying case, you will void your Niton 300 Series Analyzer's warranty in its entirety. You will be billed for a replacement case plus any repairs resulting from improper shipping. •

CAUTION Always remove the battery pack when transporting or storing your analyzer. •

Note THE Niton SPECTRUM ANALYZER CONFORMS TO THE CONDITIONS AND LIMITATIONS SPECIFIED IN 49 CFR 173.424 FOR EXCEPTED RADIOACTIVE MATERIAL. EXCEPTED-PACKAGE INSTRUMENTS AND ARTICLES, N.O.S. UN-2911. •

Appendices

Appendix A: X-ray Emission Energies Arranged Alphabetically by Element, by Atomic Number

Table A-1. X-ray Emission Energies Arranged Alphabetically by Element, by Atomic Number

Element	Symbol	Atomic Number	Atomic Weight	Ka	Kb	La	Lb	Lg
potassium	K	19	39.10	3.3	3.7			
calcium	Ca	20	40.80	3.7	4.0			
scandium	Sc	21	44.96	4.1	4.5			
titanium	Ti	22	47.90	4.5	4.9			
vanadium	V	23	50.94	4.9	5.4			
chromium	Cr	24	52.00	5.4	5.9			
manganese	Mn	25	54.94	5.9	6.5			
iron	Fe	26	55.85	6.4	7.1			
cobalt	Co	27	58.93	6.9	7.6			
nickel	Ni	28	58.70	7.5	8.3			
copper	Cu	29	63.55	8.0	8.9			
zinc	Zn	30	65.38	8.6	9.6			
gallium	Ga	31	69.72	9.2	10.3			
germanium	Ge	32	72.59	9.9	11.0			
arsenic	As	33	74.92	10.5	11.7			
selenium	Se	34	78.96	11.2	12.5			
bromine	Br	35	79.90	11.9	13.3			
krypton	Kr	36	83.80	12.6	14.1			
rubidium	Rb	37	85.47	13.4	15.0			
strontium	Sr	38	87.62	14.1	15.8			
yttrium	Y	39	88.91	14.9	16.8			
zirconium	Zr	40	91.22	15.7	17.7			
niobium	Nb	41	92.91	16.6	18.6			
molybdenum	Mo	42	95.94	17.4	19.6			
technetium	Tc	43	98.00	18.3	20.6	2.4	2.5	2.8

Appendix A:

Table A-1. X-ray Emission Energies Arranged Alphabetically by Element, by Atomic Number

Element	Symbol	Atomic Number	Atomic Weight	Ka	Kb	La	Lb	Lg
ruthenium	Ru	44	101.07	19.2	21.7	2.6	2.6	3.0
rhodium	Rh	45	102.91	20.2	22.8	2.7	2.8	3.1
palladium	Pd	46	106.42	21.1	23.9	2.8	3.0	3.3
silver	Ag	47	107.87	22.1	25.0	3.0	3.2	3.5
cadmium	Cd	48	112.41	23.1	26.1	3.1	3.3	3.7
indium	In	49	114.82	24.1	27.4	3.3	3.5	3.9
Tin	Sn	50	118.71	25.2	28.6	3.4	3.7	4.1
antimony	Sb	51	121.76	26.3	29.9	3.6	3.8	4.3
tellurium	Te	52	127.60	27.4	31.1	3.8	4.0	4.6
iodine	I	53	126.90	28.5	32.4	3.9	4.2	4.8
xenon	Se	54	131.30	29.7	33.8	4.1	4.4	5.0
cesium	Cs	55	132.91	30.9	35.1	4.3	4.6	5.3
barium	Ba	56	137.33	32.1	36.6	4.5	4.8	5.5
lanthanum	La	57	138.91	33.3	38.0	4.7	5.0	5.8
cerium	Ce	58	140.12	34.6	39.5	4.8	5.3	6.0
praseodymium	Pr	59	140.91	35.9	41.0	5.0	5.5	6.3
neodymium	Nd	60	144.24	37.2	42.5	5.2	5.7	6.6
promethium	Pm	61	145.00	38.5	44.0	5.4	6.0	6.9
samarium	Sm	62	150.35	39.9	45.6	5.6	6.2	7.2
europium	Eu	63	151.96	41.3	47.3	5.8	6.5	7.5
gadolinium	Gd	64	157.25	42.8	48.9	6.1	6.7	7.8
terbium	Tb	65	158.92	44.2	50.7	6.3	7.0	8.1
dysproium	Dy	66	162.50	45.7	52.4	6.5	7.3	8.4
holmium	Ho	67	164.93	47.3	54.2	6.7	7.5	8.7
erbium	Er	68	167.26	48.8	56.0	6.9	7.8	9.1
thulium	Tm	69	168.93	50.4	57.8	7.2	8.1	9.4
ytterbium	Yb	70	173.04	52.0	59.7	7.4	8.4	9.8
lutetium	Lu	71	174.97	53.7	61.6	7.7	8.7	10.1
hafnium	Hf	72	178.49	55.4	63.6	7.9	9.0	10.5
tantalum	Ta	73	180.95	57.1	65.6	8.1	9.3	10.9
tungsten	W	74	183.85	58.9	67.6	8.4	9.7	11.3
rhenium	Re	75	186.20	60.7	69.7	8.7	10.0	11.7
osmium	Os	76	190.20	62.5	71.8	8.9	10.4	12.1

Table A-1. X-ray Emission Energies Arranged Alphabetically by Element, by Atomic Number

Element	Symbol	Atomic Number	Atomic Weight	Ka	Kb	La	Lb	Lg
iridium	Ir	77	192.20	64.3	73.9	9.2	10.7	12.5
platinum	Pt	78	195.09	66.2	76.1	9.4	11.0	12.9
gold	Au	79	196.97	68.2	78.4	9.7	11.4	13.4
mercury	Hg	80	200.59	70.2	80.7	10.0	11.8	13.8
thallium	Tl	81	204.37	72.2	83.0	10.3	12.2	14.3
lead	Pb	82	207.19	74.2	85.4	10.5	12.6	14.8
bismuth	Bi	83	208.98			10.8	13.0	15.2
polonium	Po	84	(209.0)			11.1	13.4	15.7
astatine	At	85	(210.0)			11.4	13.9	16.2
radon	Ra	86	(222.0)			11.7	14.3	16.8
francium	Fr	87	(223.0)			12.0	14.8	17.3
radium	Ra	88	(226.0)			12.3	15.2	17.8
actinium	Ac	89	(226.0)			12.7	15.7	18.4
thorium	Th	90	232.04			13.0	16.2	19.0
protactinium	Pa	91	(231.0)			13.3	16.7	19.6
uranium	U	92	238.03			13.6	17.2	20.2
neptunium	Np	93	237.00			13.9	17.7	20.8
plutonium	Pu	94	242.00			14.3	18.3	21.4

Appendix B: X-ray Emission Energies Arranged Alphabetically by Element, by name

Table A-2. X-ray Emission Energies Arranged Alphabetically by Element, by name

Element	Symbol	Atomic Number	Atomic Weight	Ka	Kb	La	Lb	Lg
actinium	Ac	89	(226.0)			12.7	15.7	18.4
antimony	Sb	51	121.76	26.3	29.9	3.6	3.8	4.3
arsenic	As	33	74.92	10.5	11.7			
astatine	At	85	(210.0)			11.4	13.9	16.2
barium	Ba	56	137.33	32.1	36.6	4.5	4.8	5.5
bismuth	Bi	83	208.98			10.8	13.0	15.2
bromine	Br	35	79.90	11.9	13.3			
cadmium	Cd	48	112.41	23.1	26.1	3.1	3.3	3.7
calcium	Ca	20	40.80	3.7	4.0			
cerium	Ce	58	140.12	34.6	39.5	4.8	5.3	6.0
cesium	Cs	55	132.91	30.9	35.1	4.3	4.6	5.3
chromium	Cr	24	52.00	5.4	5.9			
cobalt	Co	27	58.93	6.9	7.6			
copper	Cu	29	63.55	8.0	8.9			
dysprosium	Dy	66	162.50	45.7	52.4	6.5	7.3	8.4
erbium	Er	68	167.26	48.8	56.0	6.9	7.8	9.1
europium	Eu	63	151.96	41.3	47.3	5.8	6.5	7.5
francium	Fr	87	(223.0)			12.0	14.8	17.3
gadolinium	Gd	64	157.25	42.8	48.9	6.1	6.7	7.8
gallium	Ga	31	69.72	9.2	10.3			
germanium	Ge	32	72.59	9.9	11.0			
gold	Au	79	196.97	68.2	78.4	9.7	11.4	13.4
hafnium	Hf	72	178.49	55.4	63.6	7.9	9.0	10.5
holmium	Ho	67	164.93	47.3	54.2	6.7	7.5	8.7
indium	In	49	114.82	24.1	27.4	3.3	3.5	3.9
iodine	I	53	126.90	28.5	32.4	3.9	4.2	4.8
iridium	Ir	77	192.20	64.3	73.9	9.2	10.7	12.5
iron	Fe	26	55.85	6.4	7.1			
krypton	Kr	36	83.80	12.6	14.1			
lanthanum	La	57	138.91	33.3	38.0	4.7	5.0	5.8
lead	Pb	82	207.19	74.2	85.4	10.5	12.6	14.8

Table A-2. X-ray Emission Energies Arranged Alphabetically by Element, by name

Element	Symbol	Atomic Number	Atomic Weight	Ka	Kb	La	Lb	Lg
lutecium	Lu	71	174.97	53.7	61.6	7.7	8.7	10.1
manganese	Mn	25	54.94	5.9	6.5			
mercury	Hg	80	200.59	70.2	80.7	10.0	11.8	13.8
molybdenum	Mo	42	95.94	17.4	19.6			
neodymium	Nd	60	144.24	37.2	42.5	5.2	5.7	6.6
neptunium	Np	93	237.05			13.9	17.7	20.8
nickel	Ni	28	58.70	7.5	8.3			
niobium	Nb	41	92.91	16.6	18.6			
osmium	Os	76	190.20	62.5	71.8	8.9	10.4	12.1
palladium	Pd	46	106.42	21.1	23.9	2.8	3.0	3.3
platinum	Pt	78	195.09	66.2	76.1	9.4	11.0	12.9
plutonium	Pu	94	244.00			14.3	18.3	21.4
polonium	Po	84	(209.0)			11.1	13.4	15.7
potassium	K	19	39.10	3.3	3.7			
praseodymium	Pr	59	140.91	35.9	41.0	5.0	5.5	6.3
promethium	Pm	61	(145.0)	38.5	44.0	5.4	6.0	6.9
protactinium	Pa	91	231.0			13.3	16.7	19.6
radium	Ra	88	(226.0)			12.3	15.2	17.8
radon	Rn	86	(222.0)			11.7	14.3	16.8
rhenium	Re	75	186.20	60.7	69.7	8.7	10.0	11.7
rhodium	Rh	45	102.91	20.2	22.8	2.7	2.8	3.1
rubidium	Rb	37	85.47	13.4	15.0			
samarium	Sm	62	150.35	39.9	45.6	5.6	6.2	7.2
scandium	Sc	21	44.96	4.1	4.5			
selenium	Se	34	78.96	11.2	12.5			
silver	Ag	47	107.87	22.1	25.0	3.0	3.2	3.5
strontium	Sr	38	87.62	14.1	15.8			
tantalum	Ta	7	180.95	57.1	65.6	8.1	9.3	10.9
tellurium	Te	52	127.60	27.4	31.1	3.8	4.0	4.6
terbium	Tb	65	158.92	44.2	50.7	6.3	7.0	8.1
thallium	Tl	81	204.37	72.2	83.0	10.3	12.2	14.3
thorium	Th	90	232.04			13.0	16.2	19.0
thulium	Tm	69	168.93	50.4	57.8	7.2	8.1	9.4

Table A-2. X-ray Emission Energies Arranged Alphabetically by Element, by name

Element	Symbol	Atomic Number	Atomic Weight	Ka	Kb	La	Lb	Lg
tin	Sn	50	118.71	25.2	28.6	3.4	3.7	4.1
titanium	Ti	22	47.90	4.5	4.9			
tungsten	W	74	183.85	58.9	67.6	8.4	9.7	11.3
uranium	U	92	238.03			13.6	17.2	20.2
vanadium	V	23	50.94	4.9	5.4			
xenon	Se	54	131.30	29.7	33.8	4.1	4.4	5.0
ytterbium	Yb	70	173.04	52.0	59.7	7.4	8.4	9.8
yttrium	Y	39	88.91	14.9	16.8			
zinc	Zn	30	65.38	8.6	9.6			
zirconium	Zr	40	91.22	15.7	17.7			

Appendix C: SpectraView

SpectraView enables you to qualitatively analyze the fluorescent x-rays of most of the elements in the periodic table in a given sample. For a complete list of elements and their fluorescent x-rays see Appendix A. In SpectraView Mode, the spectrum is displayed in a linear scale, autoscaled logarithmically so that the highest peak on the screen reaches the top of the scale.

How to Use SpectraView

You can access the SpectraView screen after taking a measurement in any mode, or while viewing a previous measurement, by selecting Spectra from the NAV Menu. Once you are in SpectraView, you can use the up and down positions of the 4-way touch pad to scroll through the spectrum, or you can tap on the spectrum display with the stylus to place the cursor at the point you tapped. The vertical cursor line indicates the current position along the spectrum.

Viewing the Information in SpectraView Mode

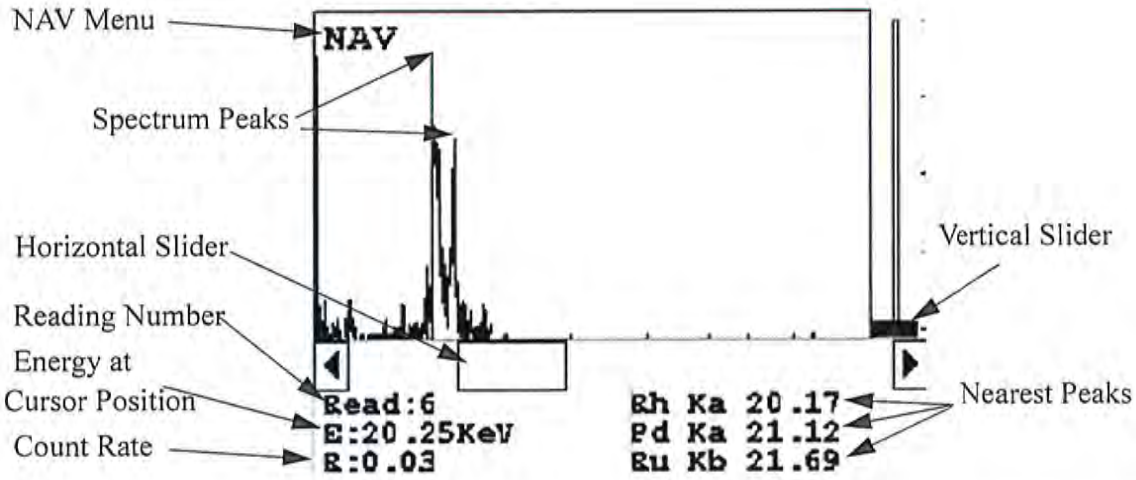


Figure A-1. The SpectraView Screen

By default, the following information is shown along with the spectrum:

The **Reading** number (Bottom Left) in the form "Read:x", where x is the Reading number.

The **position of the cursor** on the energy scale (Bottom Left, under the Reading number), in the form "E: x.xx KeV", where KeV is thousands of electron volts.

The **count rate** (Bottom Left, under the energy position), in the form "R:x.xx".

Ka, Kb, La, Lb, and/or **Lg** peaks of the three elements closest to where your cursor is positioned on the energy scale (Bottom Right). This information is written with the element symbol first, followed by either Ka (K shell alpha peak), Kb (K shell beta peak), La (L shell alpha peak), La (L shell beta peak), or Lg (L shell gamma peak). An example would be "Al Ka 1.5." To determine if a given element is present, look at the count rate at that cursor position.

SpectraView cannot be used to determine exact element percentages in a sample.

Multiple Spectra

SpectraView can display the reading spectra from multiple sources if your analyzer has more than one X-ray source or filter. Use the NAV Menu to select which spectrum to view.

The "Spectra1" choice will display the display the spectrum produced by excitation from the first source.

The "Spectra2" choice will display the display the spectrum produced by excitation from the second source.

The "Spectra3" choice will display the display the spectrum produced by excitation from the third source.

SpectraView Navigation

Use the left button on the 4-way touch pad to expand the spectrum, centered on the position of the cursor.

Use the right button on the 4-way touch pad to contract the spectrum, centered on the position of the cursor.

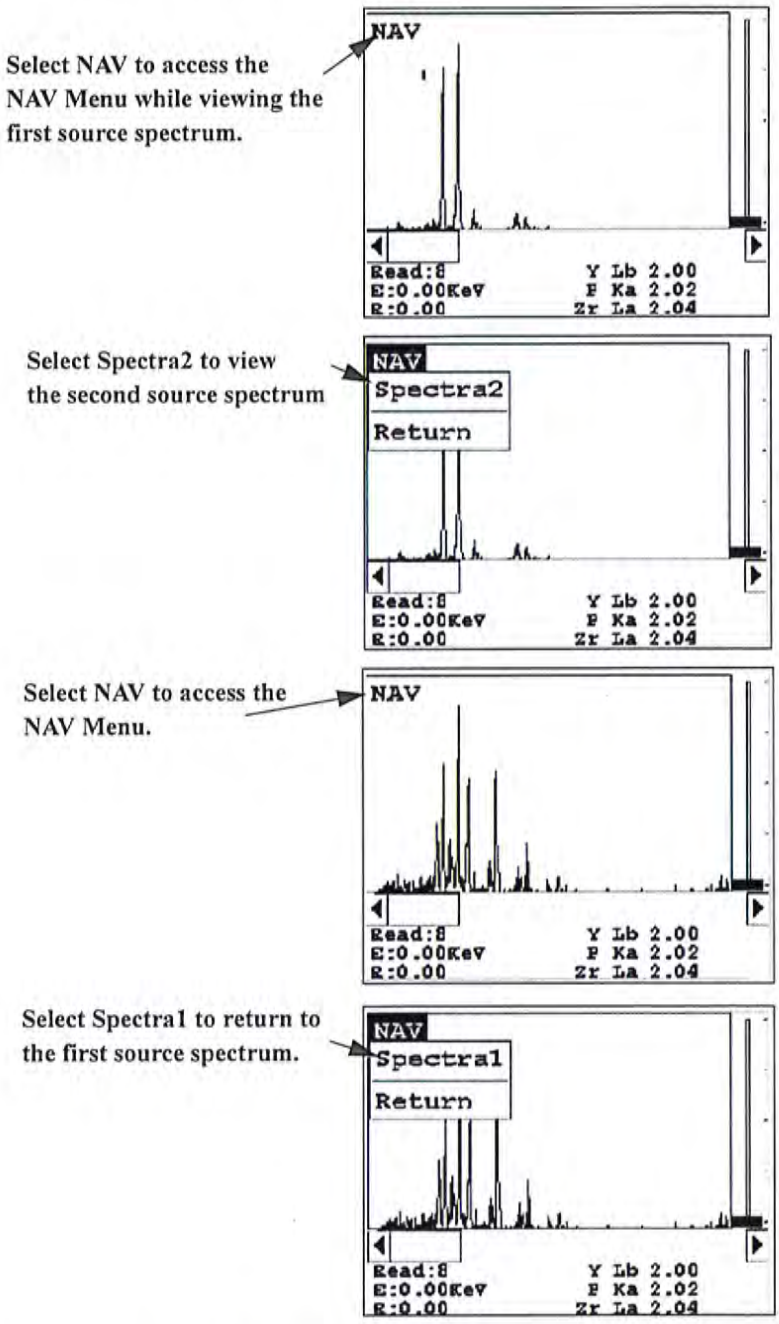


Figure A-2. Viewing Multiple Spectra

Appendix D: Summary of Warnings

Warning! Do not attempt to use this instrument without first reading and understanding the entire User's Guide! •

WARNING! Always be aware of the location of your instrument's radioactive sources and the direction of their beam of X-rays. The location of the sources is at the front end of the instrument. •

WARNING! Always treat radiation with respect. Do not hold your instrument near the Kapton window during testing. Never point your instrument at yourself or anyone else when the shutter is open. •

WARNING! In the unlikely event that the shutter becomes stuck in the open position, remove the battery (see Battery Pack and Battery Charger - Routine Maintenance Guidelines - Note: All shutters should close immediately and remain locked in the closed position when the battery pack is not attached to the instrument), replace the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department at (800) 875-1578 or +1-978-670-7460. •

WARNING! If your LCD Touch Screen displays the message "SHUTTER DOES NOT OPERATE", remove the battery (see Battery Pack and Battery Charger - Routine Maintenance Guidelines - Note: All shutters should close immediately and remain locked in the closed position when the battery pack is not attached to the instrument), replace the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department at (800) 875-1578 or +1-978-670-7460. •

WARNING! The preconditions for operation must be continued for the duration of the reading. If the preconditions are violated, all the shutters will close, and the measurement will end. The three LED lights will stop blinking, the shutters will close, and the measurement will end. The flashing of the LED lights is not synchronized to minimize power consumption. •

WARNING! The three LED warning lights are designed to blink only during a measurement, where one or more of the shutters are open and the trigger depressed. If the LED lights blink at any other time, disconnect the battery pack immediately, place the instrument in its shielded holster, place the holster in the shielded carrying case, and call Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460, or your local Authorized NITON Analyzers Service Center. ♦

WARNING! When taking samples from a site where toxic chemicals may be present, always use gloves and respiration equipment for your own protection. ♦

WARNING! Do not hold bagged samples while testing. ♦

WARNING! Tampering with the 5,500 ppm (lead high) lead-in-soil standard may cause exposure to lead dust. Keep all standards out of the reach of children. ♦

WARNING! It is important that the date and time information displayed on the **Date and Time Screen** is correct. If either the date or time is incorrect, the information stored with your readings will be incorrect. In addition, an incorrect date prevents the instrument from properly compensating for normal source decay - causing erroneous analysis results for instruments equipped with 109Cd. This information must be correct before proceeding with testing. ♦

WARNING! Do not attempt to take measurements while downloading readings! This will generate an error requiring a system reset, and may corrupt your stored readings, requiring all stored readings to be erased. ♦

WARNING! Grinding and sifting dried samples produces dust. Even clean soil contains silica, which may be hazardous when airborne. Prepare all samples in a ventilated area; wear a mask, gloves, and an apron; and spread a drop cloth. ♦

WARNING! All Service, except exterior cleaning and Kapton window replacement, must be performed by Thermo Scientific. Do not attempt to make repairs yourself. Opening the case of your NITON will void the instrument Warranty in its entirety. ♦

Appendix E: Summary of Cautions

CAUTION NITON Analyzers are not intrinsically safe instruments in regard to sparking. All pertinent Hot Work procedures should be followed in areas of concern. •

CAUTION Enabling the backlight will reduce your battery pack operating time, requiring more frequent recharges. •

CAUTION After being powered on, the NITON 300 Series Analyzer will perform an internal re-calibration before an analysis is initiated. It is recommended that you let your instrument warm up for ten minutes after start up, before testing is begun. •

CAUTION Standard Thin Sample Mode should <u>not</u> be used for quantitative lead-paint testing. Use only the three Paint Testing modes to test lead-based paint, which are available only on certain models. •

CAUTION All test equipment must be kept clean to prevent contamination of samples. •

CAUTION Never tamper with Test Standards. They should not be used unless they are completely intact. •

CAUTION Your XLp analyzer <u>must</u> have the correct time and date set in order to test samples correctly using the ^{109}Cd radioisotope source. Please make sure that the time and date are set correctly before testing. •

CAUTION Never turn off the instrument while data is being erased! •

CAUTION Do not leave the battery pack connected to the charger for excessive periods of time. Overnight recharging is recommended. •

CAUTION Store the instrument and the spare battery packs in a cool place, away from direct sunlight. •

CAUTION Always obtain a Return Authorization (RA) number from Thermo Scientific's Service Department in the United States, toll free, at (800) 875-1578, or outside the United States, at +1-978-670-7460 before returning your instrument to the NITON Service Department or local Authorized NITON Analyzer Service Center. •

CAUTION Do not force the charger into the RS-232 port! •

CAUTION Do not store battery packs or charger in direct sunlight. •

CAUTION Do not let the battery pack recharge for excessive periods of time. •

CAUTION Always transport the unit in its padded carrying case, and store the NITON Analyzer in its case whenever it is not being used. •

CAUTION In most cases, no notification is required if transporting within state boundaries. This may not be the case when entering federal properties. •

CAUTION Within the United States, always keep a copy of the US DOT compliance statement in your NITON instrument case at all times. A copy is included with your instrument. •

CAUTION Always follow all pertinent local and national regulations and guidelines, wherever your XLp 300 series analyzer is transported or used. •

CAUTION If you return your NITON instrument without the carrying case, you will void your NITON 300 Series Analyzer's warranty in its entirety. You will be billed for a replacement case plus any repairs resulting from improper shipping. •

CAUTION Always remove the battery pack when transporting or storing your instrument. •

CAUTION Use of controls, adjustments or performance of procedures other than those specified herein may result in hazardous laser light exposure. •

CAUTION Avoid any vibration, loud noise, strong electronice fields, or other possible interference when your analyzer is calibrating its detector. ♦

Appendix F: Laser Device Notice

Thermo Scientific products using lasers comply with US 21CFR1040.10, Subchapter J, and with IEC825/EN 60 825 or IEC825-1/EN 60 825-1, depending on date of manufacture. The laser classification is marked on one of the labels on the product.

Class 1 laser devices are not considered to be hazardous when used for their intended purpose. The following statement is required to comply with IUS and International regulations:

CAUTION Use of controls, adjustments or performance of procedures other than those specified herein may result in hazardous laser light exposure. ♦

Class 2 laser scanners use a low power, visible light diode. As with any very bright light source, such as the sun. the user should avoid staring directly into the light beam. Momentary exposure to a Class 2 laser is not known to be harmful.

Appendix G: Warranty

Thermo Fisher Scientific will warranty parts and labor for any manufacturer's defects for two years (24 months). No precision instrument is warranted if crushed, dropped on the floor or in a bucket of water. All service, including repairs and routine maintenance, and x-ray tube replacement or re-sourcing, must be performed by Thermo Fisher Scientific or by an Authorized NITON Analyzer Service Facility. Any attempt to open the sealed plastic housing of your NITON instrument will nullify the instrument warranty in its entirety.

Limited Warranty Provision for Use with Purchase and License Agreement for Thermo Fisher Scientific XRF Detection instruments:

Except as otherwise agreed in writing, Thermo Fisher Scientific warrants, under normal conditions of operation, each product sold (except for components not of its manufacture) against defects of material and workmanship, provided that such product has been properly utilized. This warranty applies to the original purchaser only and shall commence to run from the date of shipment and shall continue for a period of twenty-four (24) months. In any event, Thermo Fisher's liability for any such defects of material and workmanship shall not exceed the cost of replacement of defective parts upon timely notification of such defect in writing delivered to Thermo Fisher's home office. Thermo Fisher shall not be liable for damage or destruction caused during delivery or caused other than by employees of Thermo Fisher Scientific.

Material, accessories, parts, or items of equipment furnished by suppliers to Thermo Fisher and used in the manufacture of Thermo Fisher products are guaranteed by Thermo Fisher Scientific only to the extent of the original manufacturer's express warranty to Thermo Fisher for a period not to exceed the warranty period described in paragraph (a) above and provided that the purchaser shall have notified Thermo Fisher so as to enable Thermo Fisher to avail itself of its rights under such original manufacturer's express warranty.

Specific warranties of some common accessories:

- Battery Charger 12 months
- Batteries 12 months
- Soil Grinder no warranty
- Single-stage helium tank regulator 12 months
- Two-stage helium tank regulator 12 months

Thermo Fisher Scientific shall, at its option, repair such defects or replace the parts or products found defective. All defective parts are to be returned, freight prepaid, immediately to Thermo Fisher for inspection and credit. Thermo Fisher will make no allowance for repairs or alterations made by the purchaser unless made with the advance written consent of Thermo Fisher.

Thermo Fisher Scientific assumes no liability for costs of disassembly of defective parts and equipment. Shipment by purchaser of all repairs and replacements under this warranty are F.O.B. Thermo Fisher Scientific's factory or authorized service representative and method of shipment will be determined by Thermo Fisher. The purchaser will pay shipping costs and insurance in both directions of products, parts, or components shipped for warranty service hereunder. The purchaser will be responsible for risk of loss in both direction. Replaced parts or components will become the property of Thermo Fisher Scientific. Replacement parts or components may contain recycled, refurbished, or remanufactured parts equivalent to new parts and shall be warranted for the remainder of the original warranty period for the products.

Thermo Fisher Scientific shall not be liable for delays, deprivation of use, or any other damages, direct or indirect, which may result to the purchaser because of defects in the product or because of the purchaser's inability to operate it or use it to his satisfaction. Thermo Fisher Scientific will not be liable to anyone for special or consequential damages of any kind. Thermo Fisher neither assumes nor authorizes any person to assume for it, any other obligation or liability with respect to Thermo Fisher products.

a. Except for the foregoing express warranty, there are no warranties, representations, or guarantees, express or implied, except as are expressly set forth herein. The foregoing warranty is the only warranty made by Thermo Fisher Scientific. Any implied warranty of merchantability or fitness for a particular purpose on this product is limited in duration to the one year (12 month) duration of this written warranty. Some countries, and some states within the United States, do not allow limitations on how long an implied warranty lasts or the exclusion of limitation of incidental or consequential damages so the above limitations or exclusions may not apply to you. This warranty gives you specific legal rights and you may also have other rights which vary from state to state and country to country.

Appendix G:

ATTACHMENT F

BUTTE HILL COVER SOIL

APPROVAL SUBMITTAL FORM

BUTTE HILL COVER SOIL APPROVAL SUBMITTAL

11/13/2021

Source:
Sample #:

Description		Specification		Sample	Specification Met Yes	Specification Met No	Other Information Requested
Chemical (mg/kg)							**Organic Matter (%)**
	As	<	97				WB
	Cd	<	4				
	Cu	<	250				**Soil Nutrients**
	Hg	<	5				NO_3 (ug/g)
	Pb	<	100				P (ug/g)
	Zn	<	250				K (ug/g)
pH (s.u.)							
		>	5.5				
		<	8.5				
SAR							
		<	12				
Saturation (%)							
		<	85				
		>	25				
EC (mmhos/cm)							
		<	4				**Particle Size**
Textural Classification (USDA) <2.0 mm							Sand (%)
							Silt (%)
							Clay (%)
			Loam				
			Sandy loam				
			Sandy clay loam				
			Sandy clay				
			Clay loam				
			Silty clay				
			Silty clay loam				
			Silt loam				
			Silt				
			*Per EPA Approval (Loamy sand)				
Rock Content (%) (by volume)		<	45				

Legend:

# Value	- Criteria met
# Value	- Does not meet Criteria

B-SB Representative _____ Date: _____

EPA Representative: _____ Date: _____

ATTACHMENT G

CORRECTIVE ACTION REPORT

Corrective Action Report/ Corrective Action Plan

Project ID	Project Name	Document ID

Preparer's Signature/Submit Date	Submitted to:

Description of the requirement or specification	
Reason for the Corrective Action	
Location, affected sample, affected equipment, etc. requiring corrective action	
Suggested Corrective Action	(Continue on Back)
Corrective Action Plan	(Continue on Back) ☐ Approval signature/date:_____
	Approval of corrective actions required by EPA? ☐ Yes ☐ No ☐ EPA approval name/date:_____ ☐ Corrective actions completed name/date:_____
Preventative Action Plan	(Continue on Back) ☐ Preventative actions completed name/date:_____

Corrective Action Report/
Corrective Action Plan

Suggested Corrective Action (Continued)	
Corrective Action Plan (Continued)	
Preventative Action Plan (Continued)	

ATTACHMENT H

DATA VALIDATION CHECKLIST

Data Validation Checklist for Metals Sample Analysis

Site: **Case No:** **Laboratory:**

Project: **Sample Matrix:** **Analyses:**

Sample Date(s): **Analysis Date(s):**

Data Validator: **Validation Date(s):**

1. Holding Times

Analyte(s)	Laboratory	Matrix	Method	Holding Times*	Collection Date(s)	Batch	Analysis Date(s)	Holding Time Met (Y/N)	Affected Data Flagged (Y/N)

*Reference for Holding Times –

Were any data flagged because of holding time? Y ☐ N ☐

Were any data flagged because of preservation problems? Y ☐ N ☐

Describe Any Actions Taken:

Comments:

2. Instrument Calibration

Was the Tune analysis performed?	Y	☐	N	☐	N/A	☐
Was the peak widths and resolution of the masses within the required control limits?	Y	☐	N	☐	N/A	☐
Was the percent relative standard deviation ≤ 5% for all analytes in the Tune solutions?	Y	☐	N	☐	N/A	☐
Was Instrument successfully calibrated at the correct frequency?	Y	☐	N	☐		
Was Instrument calibrated with appropriate standards and blanks?	Y	☐	N	☐		
Were Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) samples analyzed?	Y	☐	N	☐		
Were ICV and CCV results within the control window?	Y	☐	N	☐		
Were any data flagged because of calibration problems?	Y	☐	N	☐		

Describe Any Actions Taken:

Comments:

3. Blanks

Were Initial and Continuing Calibration Blanks (ICB and CCBs) analyzed?	Y	☐	N	☐
Were ICBs and CCBs within the control window?	Y	☐	N	☐
Were Method Blanks (MBs) analyzed at the frequency of 1 per analytical batch?	Y	☐	N	☐
Were MBs within the control window?	Y	☐	N	☐
Were any data flagged because of blank problems?	Y	☐	N	☐

Describe Any Actions Taken:

Comments:

4. Interference Check Samples

Were ICP Interference Check Samples (ICS) within the control limits?	Y	☐	N	☐
Were any data flagged because of ICS problems?	Y	☐	N	☐

Describe Any Actions Take:

Comments:

5. Laboratory Control Samples

Were Laboratory Control Samples (LCS) analyzed at the frequency of 1 per batch?	Y		N	
What was the source of the LCS?				
Were LCS results within the control window?	Y		N	
Were any data flagged because of LCS problems?	Y		N	

Describe Any Actions Taken:

Comments:

6. Duplicate Sample Results

Were Laboratory Duplicate Samples (LDS) analyzed at the frequency of 1 per batch?	Y		N	
Were LDS results within the control window?	Y		N	
Were any data flagged because of LDS problems?	Y		N	

Describe Any Actions Taken:

Comments:

7. Matrix Spike Sample Results

Were Laboratory Matrix Spike Samples (LMS) analyzed at the frequency of 1 per batch?	Y		N	
Were LMS percent recovery (%R) results within the control window?	Y		N	
Were any data flagged because of LMS problems?	Y		N	

Describe Any Actions Taken:

Comments:

8. ICP Serial Dilutions

Were ICP Serial Dilutions (SD) analyzed at the frequency of 1 per batch?	Y		N	
Were SD percent differences (%D) results within the control window?	Y		N	
Were any data flagged because of SD problems?	Y		N	

Describe Any Actions Taken:

Comments:

9. Internal Standards

Were internal standards added to each sample in the analytical batch?	Y		N	
Were the percent relative recoveries (%RI) within the control window?	Y		N	
Were any data flagged because of internal standard problems?	Y		N	

Describe Any Actions Taken:

Comments:

10. Field Blanks

Were field blanks submitted as specified in the Sampling Analysis Plan (SAP)?	Y		N		N/A	
Were field blanks within the control window?	Y		N		N/A	
Were any data qualified because of field blank problems?	Y		N		N/A	

Describe Any Actions Taken:

Comments:

11. Field Duplicates

Were field duplicates submitted as specified in the Sampling Analysis Plan (SAP)?	Y		N		N/A	
Were the field duplicates within the control window?	Y		N		N/A	
Were any data qualified because of field duplicate problems?	Y		N		N/A	

Describe Any Actions Taken:

Comments:

12. Overall Assessment

Are there analytical limitations of the data that users should be aware of?	Y		N	

If so, explain:

Comments:

13. Authorization of Data Validation

Data Validator

Name: **Reviewed by:**

Signature: _____ _____

Date: _____ _____

ATTACHMENT I

ANNUAL QAPP REVISION SUMMARIES

Attachment I
Annual QAPP Revision Summary Page

Date	Revision #	Summary of Changes