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X-RAY FLUORESCENCE (XRF) ANALYSIS OF HANFORD LOW ACTIVITY WASTE SIMULANTS METHOD DEVELOPMENT

ANALYTICAL DEVELOPMENT

August 8, 2007

Washington Savannah River Company
Savannah River Site
Aiken, SC 29808

Prepared for the U.S. Department of Energy Under Contract Number DEAC09-96SR18500

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David M. Missimer, SRNL
Arthur R. Jurgensen, SRNL
Ronny L. Rutherford, SRNL

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Washington Savannah River Company
Savannah River Site
Aiken, SC 29808

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REVIEWS AND APPROVALS

D. M. Missimer, Task Lead, Analytical Development, SRNL Date

A. R. Jurgensen, Co-Author, Analytical Development, SRNL Date

R. L. Rutherford, Co-Author, Analytical Development, SRNL Date

M. E. Summer, Technical Reviewer, Analytical Development, SRNL Date

F. M. Pennebaker, RPP Program Manager, Analytical Development, SRNL Date

R. E. Edwards, RPP Project Manager, Process Science & Engineering, SRNL Date

L. M. Chandler, Director, Analytical Development, SRNL Date

A. V. Arakali, C&T WTP Representative Date

T. A. Lane, C&T WTP Representative Date

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LIST OF ACRONYMS

ADD	Analytical Development Directorate
CRV	Concentrate Receipt Vessel
FP	Fundamental Parameter
GFC	Glass Former Chemicals
HLW	High Level Waste
HRTL	Hydrogen Research Technical Laboratory
IC	Ion Chromatography
ICP-AES	Inductively Coupled Plasma – Atomic Emission Spectroscopy
IUPAC	International Union of Pure and Applied Chemistry
LAW	Low Activity Waste
LDR	Land Disposal Restriction
MFPV	Melter Feed Preparation Vessel
MSDS	Material Data Safety Sheet
NIST	National Institute of Standard Testing
PC	Proportional Counter
PHA	Pulse Height Adjustment
PNNL	Pacific Northwest National Laboratory
QC	Quality Control
RPP	River Protection Project
%RSD	Percent Relative Standard Deviation
SC	Scintillation Counter
SRNL	Savannah River National Laboratory
SRS	Savannah River Site
TRU	Transuranics
WCP	Waste Compliance Program
WTP	Hanford Tank Waste Treatment and Immobilization Plant
WD-XRF	Wavelength Dispersive X-ray Fluorescence
XRF	X-ray Fluorescence

1. Executive Summary

The x-ray fluorescence laboratory (XRF) in the Analytical Development Directorate (ADD) of the Savannah River National Laboratory (SRNL) was requested to develop an x-ray fluorescence spectrometry method for elemental characterization of the Hanford Tank Waste Treatment and Immobilization Plant (WTP) pretreated low activity waste (LAW) stream to the LAW Vitrification Plant. The WTP is evaluating the potential for using XRF as a rapid turnaround technique to support LAW product compliance and glass former batching. The overall objective of this task was to develop an XRF analytical method that provides rapid turnaround time (<8 hours), while providing sufficient accuracy and precision to determine variations in waste.

The Phase 1b restored work scope at SRNL consisted of following activities:

- Re-install the XRF instrument,
- Obtain WTP simulant samples representative of LAW envelopes,
- Perform additional precision testing,
- Procure matrix-match standards to calibrate the XRF instrument,
- Develop an XRF method for the WTP using simulant samples representative of the LAW envelopes.

A small precision study was conducted after re-installing the instrument and procuring new standards. As reported earlier by Jurgensen[1], there were some issues with a few elements precipitating out of the basic solution over time which affected the long term precision of the method. The precision results improved by diluting the simulant sample, but the precipitation issue continued to persist. Simulant precipitation was resolved by converting the basic stimulant to an acid matrix. The precision improved from <10% to <5% for the simulant, AP-101, when the simulant was converted from basic to acidic.

Three XRF fundamental parameters methods were studied, acidic, basic, and drift correcting. A thorough statistical analysis of the results was conducted. The analysis compared Methods 1 and 2 (acidic to basic) to the ICP-AES and IC. Method 3 (drift correcting) was not included in the statistical analysis due to an imperfect drift correction of the x-ray intensities. The results of the statistical evaluation suggest that Method 1 (acidic) is significantly similar to the ICP-AES and IC. The statistically analysis is referenced in the Results section in this report.

[1] A. R. Jurgensen, D. M. Missimer, and R. L. Rutherford, "X-ray Fluorescence (XRF) Analysis of Hanford Low Activity Waste Simulants", WSRC-TR-2006-00137, SRNL-RPP-2006-00019, Savannah River Site, Aiken SC 29808 (May 8, 2006).

2. Introduction and Background

This document addresses the Savannah River National Laboratory (SRNL) Phase 1b method development activities detailed in task plan WSRC-TR-2004-00563, Rev3[2] in support of using x-ray fluorescence (XRF) spectrometry for elemental characterization of the Hanford Waste Treatment and Immobilization Plant (WTP) pretreated low activity waste (LAW) stream to the LAW Vitrification Plant. The WTP is evaluating the potential for using XRF as a rapid turnaround technique to support LAW product compliance and glass former batching. The overall objective of this task was to develop XRF analytical methods that provide the rapid turnaround time (<8 hours) requested by the WTP, while providing sufficient accuracy and precision to determine waste composition variations.

The LAW stream is primarily a liquid supernatant comprised of sodium hydroxide and sodium nitrate. It also contains trace metals, sulfate, aluminum, and potassium and up to 3.8-wt% entrained solids. The waste will be transferred from the tank farms to the Pretreatment Facility in batches up to 5,700,000-L. If required, the LAW stream will be concentrated up to a 5-molar (M) sodium concentration and filtered to remove any entrained solids. As necessary, strontium and transuranics (TRU) will be removed using a permanganate precipitation process and blended with the high level waste (HLW) feed. Cesium will be separated next using an ion exchange resin. The low activity treated waste will then be concentrated along with any LAW vitrification recycle solutions to 5 to 8-M sodium. The target concentration of the treated LAW batch will be defined by the relative concentrations of sodium and sulfate.

The Pretreatment Facility is designed to provide sufficient pretreated LAW feed to produce 90,000-kg of LAW glass per day. For LAW vitrification, 34,000-L batches of treated LAW Feed are transferred from the Pretreatment Process to the LAW- Concentrate Receipt Vessels CRV and then to the Melter Feed Preparation Vessel (MFPV) where the waste is combined with glass former chemicals (GFC). The LAW Concentrate Storage Vessel in the Pretreatment Facility is not well mixed and may change composition as new waste feeds are added during processing. As such, waste and glass formers can not be moved past the LAW - MFPV until selected analytical results are available on the LAW - CRV sample. Each CRV batch is expected to refill an MFPV four times (or twice each for the two LAW MFPVs). The required analysis turnaround time is <15-hr with preferred turnaround time of <8-hr. Based on composition and radionuclide analyses, glass formulation calculations will specify the GFC batch to be added.

This restored work scope focused on developing an XRF method for AN-105, AP-101, AN-107 and AZ-101 simulants that are representative of the LAW envelopes. The simulant samples representing LAW envelopes were obtained from SRNL researchers (AN-105, AP-101, and AN-107) and from Optima Chemical Group (AZ-101). The simulant concentrations are listed in Table 1[3,4]. The hold point elements at the top of Table 1 (Al, Cl, K, Na, and SO_4) were given

[2] A. R. Jurgensen, D. M. Missimer, and F. M. Pennebaker, "Task Technical and Quality Assurance Plan for X-Ray Fluorescence Method Development", WSRC-TR-2004-00563, Rev. 3, Savannah River Site, Aiken SC 29808 (June 13, 2007).

[3] C. A. Nash, M. R. Diugnan, and C. E. Duffey, "Batch, Kinetics, and Column Data from Spherical Resorcinol-Formaldehyde Resin", WSRC-STI-2006-00071, SRNL-RPP-2006-00024, Savannah River National Laboratory, Aiken SC 29808 (December 18, 2006).

primary attention because of their relative importance in glass batching and waste form compliance. Other elements that were given attention were Ca, PO_4, Cr, Ni, and Mo. These ten elements were the main focus of the XRF WTP method development. These elements were proposed by Jurgensen in his report for LAW XRF calibration standards. The only element that is not included in the list compiled by Jurgensen was silicon. The standard vendor had difficulty keeping silicon in solution, so silicon was dropped from the standard matrices. The work scope of the activities did not include the characterization of the glass former feed nor glass product from the melter.

For Phase 1b, SRNL objectives were to:

- Re-install the XRF instrument in SRNL. When the funding was terminated back in April 2006, the instrument was moved to the Hydrogen Research Technical Laboratory (HRTL);
- Obtain LAW simulants from SRNL scientist for the three LAW Envelopes that were tested in phase 1a of this work;
- Perform additional precision testing
- Procure XRF matrix-matched standards; and
- Develop an XRF method.

[4] R. E. Eibling, R. F. Schumacher, and E. K. Hansen , "Development of Simulants to Support Mixing Tests for High Level Waste and Low Activity", WSRC-TR-2003-00220, SRT-RPP-2003-00098, Savannah River Site, Aiken SC 29808 (December 2003).

Table 1. Reported Simulant Compositions.

Component	Envelope A[3] Tank AN-105 Concentration mg/L	Envelope A[3] Tank AP-101 Concentration mg/L	Envelope B/D[4] Tank AZ-101 Concentration mg/L	Envelope C[3] Tank AN-107 Concentration mg/L
Hold Point Elements				
Aluminum	18560	6980	a	248
Chloride	a	1450	121	1520
Potassium	3506	27740	340	1250
Sodium	115000	115000	9252	130000
Sulfate	385	3580	406	5483
Non-Hold Point Elements				
Calcium	a	6.8	a	161
Iron	a	2.2	a	11.4
Magnesium	a	a	a	<1.56
Phosphate	266	1180	2341	883
Silicon	a	122	a	56.5
Zinc	5.6	5	a	20.7
Zirconium	a	a	a	a
LDR Elements			a	
Barium	a	0.29	a	a
Cadmium	a	1.7	a	<0.263
Chromium	631	150	a	0.506
Lead	25	13	a	<2.74
Nickel	a	7	a	320
Selenium	a	a	a	a
Silver	a	a	a	a
Additional XRF Elements				
Cerium	a	a	a	<7.80
Copper	a	1.4	a	14.1
Lanthanum	a	a	a	<1.46
Manganese	a	a	a	0.705
Molybdenum	38	12.9	a	21.7
Neodymium	a	a	a	<6.35
Additional Constituents				
Ammonium	a	a	a	a
Boron	a	14.2	15.7	a
Carbonate	a	26760	3782	a
Fluoride	a	53	68	a
Hydroxide	a	a	2734	a
Nitrate	a	104160	372	156600
Nitrite	a	32500	1242	38500
Acetate	a	1460	85	a
Citric Acid	a	a	a	a
Ethylenediaminetetraacetic acid	a	a	a	a
Formate	a	1110	a	a
Glycolate	a	a	a	a
n-Hydroxyethylenediaminetriacetic acid	a	a	a	a
Iminodiacetic Acid	a	a	a	a
Nitrilotriacetic Acid	a	a	a	a
Oxalate	a	1600	a	<1000
Sodium Gluconate	a	a	a	a

a) These constituents were not reported in references 3 or 4.

3. Experimental

3.1 Instrumentation

3.1.1 Re-Installing the XRF Instrument

The Rigaku Mini II XRF instrument was moved to the HRTL after WTP issued the stop work on Phase 1b activities in April 2006. The instrument was setup except for the P-10 cylinder and helium service. However at the start of the revised work scope, the XRF instrument was moved back to SRNL because of the problems with the in-house gas delivery system. A free software upgrade was available through Rigaku/MSC's FTTP website. Prior to the start of the upgrade, all the information that was stored on the computer was saved and reloaded after the upgrade was complete. This process of saving and reloading information in the software is carried out through a subroutine in the software called DSAVE/DLOAD.

Note: The DSAVE/DLOAD subroutine should be carried out at least once a week and the information stored on floppy disks or on a file server. The new version of the software will allow the user to store data directly to a file server.

3.1.2 Start-Up

After the P-10, 10% methane 90% argon, and helium (ultra pure 99.999%) gas lines were connected to the wavelength dispersive x-ray fluorescence instrument (WD-XRF), the 4-μm instrument film, Ultralene™, was replaced. Initially, the flow rate for the P-10 and helium were set to 50 mL/min and 2 L/min respectively. The P-10 gas was set at a flow rate of 50 mL/min for 30 minutes to purge the gas flow proportional detector then maintained at 25 mL/min. This adjustment to the P-10 gas should be done initially and after changing out the P-10 cylinder. The P-10 and helium gases were allowed to flow through the instrument for one hour before the main power was turned on. The system was initialized and the x-ray tube turned on through the software. The voltage, kV, and current, mA, settings for the x-ray tube were incremented slowly over a 30 minute time period until the x-ray tube was at full power, 40-kV 1.2-mA. The instrument was left at full power with the helium flowing overnight for stability purposes.

3.1.3 Performance Check

Instrument performance was confirmed by running a vendor supplied pulse height adjustment (PHA) sample and a waste compliance program (WCP) Blend glass. Both tests were done at full power in helium. The PHA sample verifies functionality of both detectors, flow proportional counter (PC) and the scintillation detector (SC). The scan of the flow proportional counter, Figure 1, looks at Si Kα on the PET crystal. The scan of the scintillation detector, Figure 2, looks at the Sn Kα on the LIF1 crystal. The peak position for the flow proportional counter and the scintillation detector should be around 200. The resolution for the flow proportional counter and the scintillation detector should be < 35% and < 40 %

respectively. The WCP Blend Glass was run to ensure that the crystals were not bumped in transit from one building to the other and to determine if the x-ray tube is deteriorating. The model for this test looks at three elemental intensities, Al, Na, and Fe, on the three different crystals, PET, RX35, and LIF1. By monitoring these elements intensities, one can determine if the crystals need to be aligned or if the x-ray tube needs to be replaced. Both tests should be done on a monthly basis or earlier if a problem is detected.

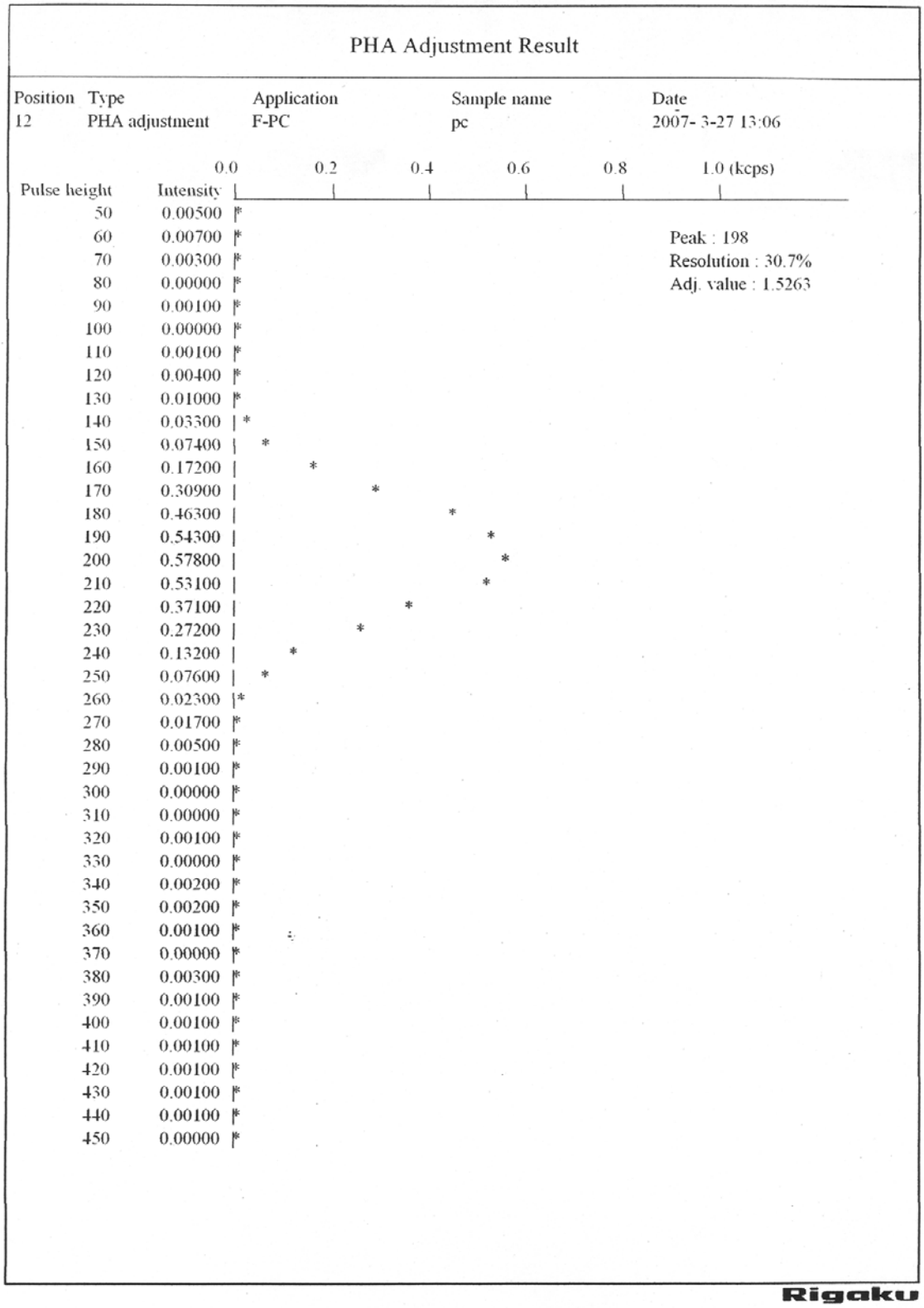

Figure 1. PHA scan for the flow proportional counter.

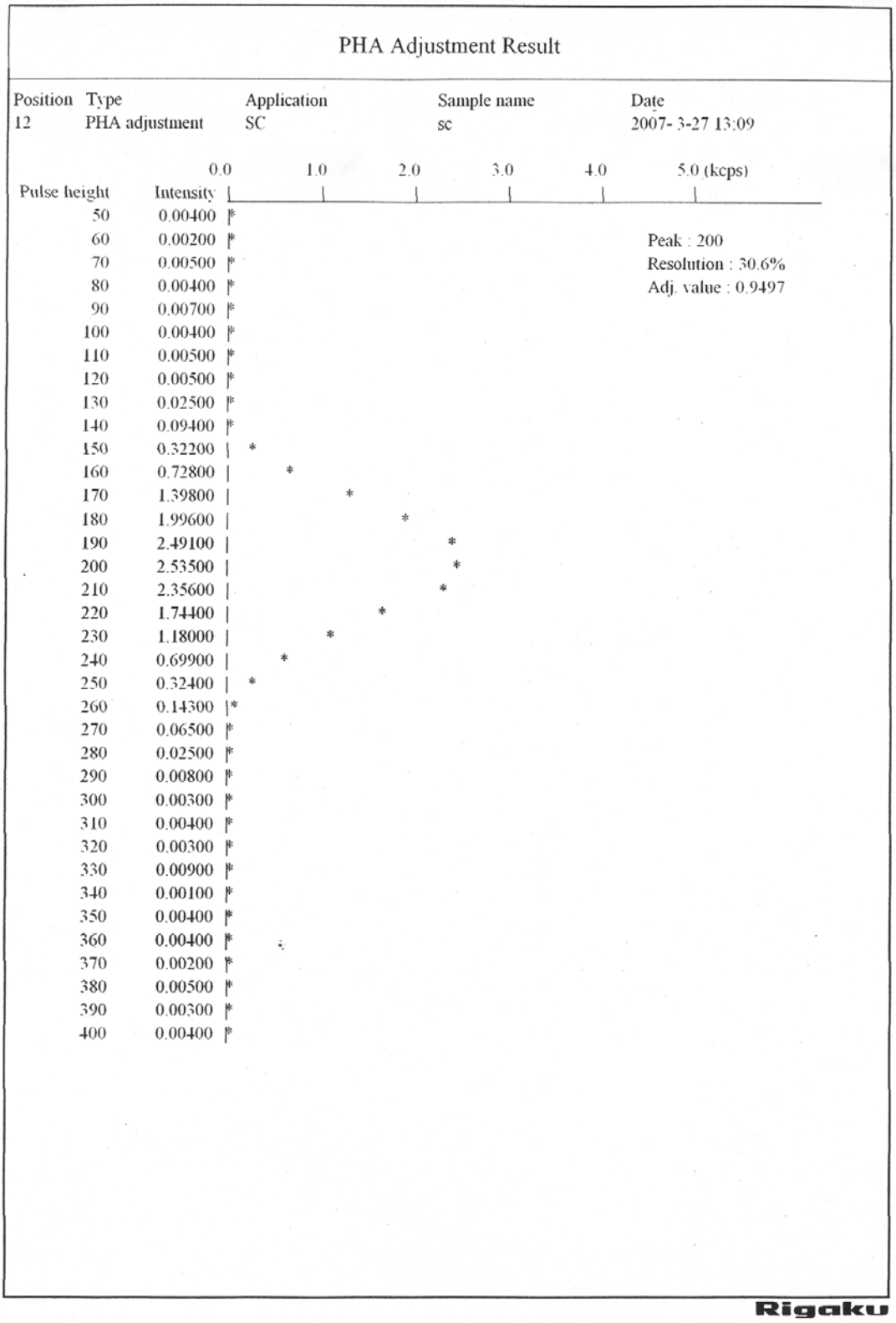

Figure 2. PHA scan for the scintillation detector.

3.1.4 Verification of Standards/Simulants

The five standards and the four simulants were analyzed on a Perkin Elmer Optima 3000 ICP-AES and a Dionex DX-500 IC to independently verify concentrations. The High Purity standards are as follows:

- AY-102 (Standard A)
- AP-101 (Standard B)
- AZ-101 (Standard C)
- AZ-102 (Standard D)
- AP-103 (Standard E).

The simulants are listed in Section 3.2.

3.2 Simulants

The LAW simulants that were prepared and used in Phase 1a and the start of Phase 1b activities of this task were neutralized and discarded when WTP issued a notice to discontinue the remaining work scope in April 2006. Instead of preparing new LAW simulants for this restored WTP XRF work scope, SRNL obtained simulant samples representative of LAW envelopes from Charles Nash of the Separations Science Programs group and Dan Burns of the Process Science and Engineering section of SRNL.

- AN-105 Envelope A simulant,
- AP-101 Envelope A simulant,
- AN-107 Envelope C simulant.
- AZ-101 Envelope B/D simulant.

Envelope A simulants, AN-105 and AP-101, had been prepared using the Russ Eibling recipe[5]. The Envelope A, AN-105, simulant was obtained after the 5M sodium dilution[3]. Envelope C simulant, AN-107, was produced by a strontium/TRU precipitation after caustic adjustment[4]. AZ-101 Envelope B/D simulant was prepared by Optima Chemical Group of Douglas, Georgia for the Gas Release/Holdup Testing currently being performed by SRNL and Pacific Northwest National Laboratory (PNNL). Optima Chemical Group prepared the simulant using the Eibling recipe that was provided to them. The AZ-101 simulant was a combination of sludge and supernate which was centrifuged with a Fisher Scientific Marathon 21K centrifuge set for 2-hr at 3000 rpm to remove the majority of suspended material. Table 2 contains the concentrations for Al, Ca, Cr, Mo, Ni, K, Na, Cl, SO_4, and PO_4 in the simulants representative of the LAW envelopes after any dilutions and/or treatments.

[5] R. E. Eibling and C. A. Nash, "Hanford Waste Simulants Created for Research and Development on the River Protection Project-Waste Treatment Plant", WSRC-TR-2000-00338, Rev. 0, Appendix A, Savannah River Site, Aiken SC 29808 (February, 2001)

Table 2. Major Element Concentrations in RPP Simulants.

Element	Units	AN-105[1]	AP-101	AZ-101[2]	AN-107[3]
Al	mg/L	18560	6980	a	248
Ca	mg/L	a	6.8	a	161
Cr	mg/L	631	150	a	0.506
K	mg/L	3506	27740	340	1250
Mo	mg/L	38	12.9	a	21.7
Na	mg/L	115000	115000	9252	130000
Ni	mg/L	a	7.0	a	320
S (also measured as SO_4)	mg/L	385	3580	406	5483
P (also measured as PO_4)	mg/L	266	1180	2341	883
Cl	mg/L	a	1450	121	1520

[1]Simulant AN-105 was made by the full Eibling recipe and diluted to 5M sodium.

[2] Envelope B/D.

[3]Simulant AN-107 was produced by a stronium/TRU precipitation after caustic adjustment.

a) These elements were not listed in the reports.

All the simulants were filtered through a Corning 0.2-μm cellulose nitrate filter assembly and stored in Teflon bottles. The metals in the filtrates were analyzed by ICP-AES and inorganic anions by IC. Many of the constituents in the solutions, e.g. organics, can not be determined by XRF and were not analyzed. Use of these simulants was approved by WTP on 6/21/07, as defined in SRNL-RPP-2004-00091 Rev 3. (See Appendix A)

3.2.1 Envelope A

Envelope A waste was generated by evaporating the low organic content, waste supernates stored in single shell tanks and the supernate produced by the Hanford B plant. Envelope A can be generally characterized as an alkaline ([OH]>1 M), high sodium (>8 M) supernate. Envelope A tanks contains ^{137}Cs at concentrations that require removal prior to LAW vitrification. The majority of the LAW vitrified product in the initial phase of the RPP-WTP will be Envelope A.

Envelope A simulants were based on the supernates from Tank AN-105 and Tank AP-101, which was decanted from the solids within the waste tank. The simulant concentrations used for AN-105 and AP-101 are listed in the first two columns of Table 1 or 2. These concentrations were reported in Appendix A in WSRC-STI-2006-00071, SRNL-RPP-2006-00024.

Envelope A simulants, AN-105 and AP-101 were analyzed by ICP-AES and IC. The average values for the ICP-AES and IC are listed in Tables 3 and 4. The ICP-AES values are an average of ten runs that were run on different days with different calibrations. The IC values are an average of five runs that were run on different days with different calibrations. IC did not report a phosphate value because of matrix effects. All the detectable components for the ICP-AES and IC can be found in Appendix F. The average values given in Tables 3 and 4 will

be compared to the XRF results.

Table 3. Analytical Results for AN-105 Simulant.

Element	Units	Measured ICP-AES Perkin Elmer	Measured IC Dionex
Al	mg/L	10600	a
Ca	mg/L	<1.29	a
Cr	mg/L	640	a
K	mg/L	3770	a
Mo	mg/L	39.8	a
Na	mg/L	122000	a
Ni	mg/L	<0.35	a
S (also measured as SO_4)	mg/L	398	140
P (also measured as PO_4)	mg/L	289	b
Cl	mg/L	a	4600

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

Table 4. Analytical Results for AP-101 Simulant.

Element	Units	Measured ICP-AES Perkin Elmer	Measured IC Dionex
Al	mg/L	6850	a
Ca	mg/L	<1.29	a
Cr	mg/L	152	a
K	mg/L	28200	a
Mo	mg/L	13.6	a
Na	mg/L	120000	a
Ni	mg/L	<0.35	a
S (also measured as SO_4)	mg/L	3810	3450
P (also measured as PO_4)	mg/L	1210	b
Cl	mg/L	a	1490

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

3.2.2 Envelope C

Envelope C waste was produced from evaporation of wastes derived from high organic content single-shell tank waste and waste generated during the Cs/Sr separation and encapsulation process conducted at the Hanford B plant. The waste is characterized by the high organic carbon content because of the presence of organic complexing agents and their decomposition products. Due to the complexing agents, the concentration of ^{90}Sr and TRU will require

removal using the Sr/TRU precipitation and filtration process. Removal of ^{137}Cs by ion exchange will also be required.

The simulant concentration for AN-107 is listed in the last column of Table 1 and 2. This concentration was reported in Appendix A in WSRC-STI-2006-00071, SRNL-RPP-2006-00024.[3]

Envelope C simulant, AN-107 was analyzed by ICP-AES and IC. The average values for the ICP-AES and IC are listed in Table 5. All the detectable components for the ICP-AES and IC can be found in Appendix F. The average values given in Table 5 will be compared to the XRF results.

Table 5. Analytical Results for AN-107 Simulant.

Element	Units	Measured ICP-AES Perkin Elmer	Measured IC Dionex
Al	mg/L	236	a
Ca	mg/L	145	a
Cr	mg/L	0.643	a
K	mg/L	1200	a
Mo	mg/L	21.8	a
Na	mg/L	141000	a
Ni	mg/L	301	a
S (also measured as SO_4)	mg/L	5200	4710
P (also measured as PO_4)	mg/L	894	b
Cl	mg/L	a	1500

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

3.2.3 Envelope B/D

Envelope B/D is comprised of insoluble compounds referred to as sludge, primarily insoluble transition metal hydroxides with most of the long half- life radionuclides, slurried with the supernate from the same tank. For example, the slurry from tank 241-AZ-101 would be comprised of the B envelope supernate and D envelope sludge. The D envelope waste will require washing using crossflow filtration and possibly caustic leaching to meet glass specifications.

The simulant concentration for AZ-101 is listed in the third column of Table 1 or 2. This concentration was reported on page 46 of WSRC-TR-2003-000220, SRT-RPP-2003-00098. The directions of how Optima Chemical Group prepared the simulant can be found in Appendix F of WSRC-TR-2003-00220, SRT-RPP-2003-00098.

Envelope B/D simulant, AZ-101 was analyzed by ICP-AES and IC. The average values for

the ICP-AES and IC are listed in Table 6. All the detectable components for the ICP-AES and IC can be found in Appendix F. The average values given in Table 6 will be compared to the XRF results.

Table 6. Analytical Results for AZ-101 Simulant.

Element	Units	Measured ICP-AES Perkin Elmer	Measured IC Dionex
Al	mg/L	<2.56	a
Ca	mg/L	<1.29	a
Cr	mg/L	142	a
K	mg/L	781	a
Mo	mg/L	25.5	a
Na	mg/L	11500	a
Ni	mg/L	<0.349	a
S (also measured as SO_4)	mg/L	800	748
P (also measured as PO_4)	mg/L	<60	b
Cl	mg/L	a	259

a) These elements were not measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

3.3 Standards

SRNL consulted with WTP about the standards to calibrate the XRF instrument because no standards existed for the LAW. SRNL and WTP decided to have an offsite vendor prepare and certify a set of standards that covered the anticipated CRV major element concentration from the first five processed LAW tanks: AY-102, AP-101, AZ-101, AZ-102, and AP-103. SRNL selected High Purity Standards from Charleston, South Carolina to prepare the standards. Table 7 lists the elements and concentrations for each of the LAW waste tanks mentioned above. The concentration for each element in the LAW waste tanks came from the most current available Best Basis Inventory obtained from the TWINS website. The standards were labeled A through E for tanks AY-102, AP-101, AZ-101, AZ-102, and AP-103. SRNL requested the Certificates of Analysis and the Material Data Safety Sheets (MSDS) for each of the standards. Standard A, AY-102, was made from a different lot of starting materials from the other four standards. Standards were prepared in 4% HNO_3 at half the targeted concentrations (see Table 7) without silicon due to precipitation problems during preparation. Standard D was prepared at the target concentration. Table 8 lists the final concentration for each element in the standards. (See Appendix B for the Standard Certificates of Analysis and the MSDS information)

Table 7. Target Calibration Standard Concentrations.

Element	Standard A AY-102 µg/mL	Standard B AP-101 µg/mL	Standard C AZ-101 µg/mL	Standard D AZ-102 µg/mL	Standard E AP-103 µg/mL
Aluminum	17700	7300	19000	9600	18300
Calcium	1000	10	250	400	80
Chromium	500	170	800	900	600
Molybdenum	10	20	100	60	60
Nickel	750	10	260	750	80
Potassium	450	34000	4800	3400	6500
Silicon	2800	100	450	560	100
Sodium	73000	129000	124000	65800	178000
Chloride	100	1600	270	70	4300
Phosphate	4900	1300	2100	750	2400
Sulfate	1800	3900	19300	19000	4800

* Standard A, Best Basis Inventory, Tank 241-AY-102, FY06, Q1.
* Standard B, Best Basis Inventory, Tank 241-AP-101, FY05, Q2.
* Standard C, Best Basis Inventory, Tank 241-AZ-101, FY06, Q1.
* Standard D, Best Basis Inventory, Tank 241-AP-103, FY05, Q1.
* Standard E, Best Basis Inventory, Tank 241-AY-102, FY06, Q1.

Table 8. High Purity Calibration Standard Concentrations.

Element	Standard A AY-102 µg/mL	Standard B AP-101 µg/mL	Standard C AZ-101 µg/mL	Standard D AZ-102 µg/mL	Standard E AP-103 µg/mL
Aluminum	8850	3650	9500	9600	9150
Calcium	500	5	125	400	40
Chromium	250	85	400	900	300
Molybdenum	5	10	50	60	30
Nickel	375	5	130	750	40
Potassium	225	17000	2400	3400	3250
Sodium	36500	64500	62000	65800	89000
Chloride	50	800	135	70	2150
Phosphate	2450	650	1050	750	1200
Sulfate	900	1950	9650	19000	2400

The ICP-AES analyzed the five High Purity standards on ten different days using a new calibration curve each day, whereas the IC ran the standards on five different days using five different calibrations. Table 9 shows the averages of the runs for each of the different High Purity standards. All the results from the IC and ICP-AES for the standards can be found in Appendix F. Because of matrix effects, the phosphate value was not reported on the IC. The sulfate value was reported, but the value is 2 to 5 times higher than expected. The sulfate could be plagued by the same matrix effect as the phosphate. This matrix effect on sulfate in the IC

data only showed up in the procured High Purity standards, which were made up in 4% HNO_3, but not in the basic simulants.

Table 9. ICP-AES and IC Average Concentrations for the High Purity Standards.

Element	Standard A Certificate µg/mL	Standard A ICP Average[1] µg/mL	Standard A IC Average[2] µg/mL	Standard B Certificate µg/mL	Standard B ICP Average[1] µg/mL	Standard B IC Average[2] µg/mL
Al	8850	8700	a	3650	3560	a
Ca	500	495	a	5	4.8	a
Cr	250	253	a	85	84.2	a
Mo	5	5.5	a	10	10.1	a
Ni	375	374	a	5	4.9	a
K	225	230	a	17000	17000	a
Na	36500	36800	a	64500	64800	a
S (also measured as SO_4)	900	952	3560	1950	2030	7160
P (also measured as PO_4)	2450	2320	b	650	615	b
Cl	50	a	<100	800	a	848

Element	Standard C Certificate µg/mL	Standard C ICP Average[1] µg/mL	Standard C IC Average[2] µg/mL	Standard D Certificate µg/mL	Standard D ICP Average[1] µg/mL	Standard D IC Average[2] µg/mL
Al	9500	9270	a	9600	9490	a
Ca	125	124	a	400	400	a
Cr	400	391	a	900	893	a
Mo	50	49.7	a	60	60.8	a
Ni	130	126	a	750	737	a
K	2400	2440	a	3400	3430	a
Na	62000	62200	a	65800	67300	a
S (also measured as SO_4)	9650	9980	24800	19000	19900	41600
P (also measured as PO_4)	1050	983	b	750	720	b
Cl	135	a	182	70	a	134

Element	Standard E Certificate µg/mL	Standard E ICP Average[1] µg/mL	Standard E IC Average[2] µg/mL
Al	9150	9000	a
Ca	40	38.5	a
Cr	300	290	a
Mo	30	29.6	a
Ni	40	38.1	a
K	3250	3300	a
Na	89000	90000	a
S (also measured as SO_4)	2400	2460	10900
P (also measured as PO_4)	1200	1110	b
Cl	2150	a	2100

[1]An average of 10 runs.

[2]An average of 5 runs.

a) These elements could not be measured by the given method.

b) Because of matrix effects, PO_4^{2-} was not reported.

Note: High bias with IC results for SO4 and PO4 due to matrix interferences.

3.4 Precision

The precision of the simulants representative of LAW envelopes was repeated in this work to confirm that the instrument was operating properly after being moved to and from HRTL, and to resolve the sample stability problem.

For the simulant precision analysis seven separate tests were conducted:

Test 1. Triplicate analysis of ten **filtered undiluted** AN-105 simulant solutions in fixed autosampler positions previously reported in WSRC-TR-2006-00137, SRNL-RPP-2006-00019. (See Appendix C Table C-1)

Test 2. Triplicate analysis of five **filtered diluted** AN-105 simulant (1:1) solutions in fixed autosampler positions. (See Appendix C Table C-2)

Test 3. Fourteen analysis of one **filtered diluted** AN-105 simulant solution (1:1) in fixed autosampler position over 8 hours. (See Appendix C Table C-3)

Test 4. Five analysis of one **filtered diluted** AN-105 simulant solution (1:1) in fixed autosampler position run at different times during the day. (See Appendix C Table C-4)

Test 5. Triplicate analysis of five **filtered diluted** AN-105 simulant (1:1) solutions in fixed autosampler positions with the instrument film was replaced prior to each run. (See Appendix C Table C-5)

Test 6. Triplicate analysis of five **filtered diluted** AP-101 simulant (1:1) solutions in fixed autosampler positions. (See Appendix C Table C-6)

Test 7. Triplicate analysis of five **filtered diluted Acidic** AP-101 simulant solutions in fixed autosampler positions. (See Appendix C Table C-7)

Test 1 mentioned above used ten 5-mL aliquots of filtered undiluted AN-105 simulant. The samples in Tests 2 through 6 were prepared by pipetting 5-mL of the simulant along with 5-mL of DI water into a polyethylene bottle. An 8-mL aliquot was pipetted into a sample cup with an Ultralene™ film on one side of the sample cup and a microporous Telfon™ film on the other side. The samples in Test 7 were prepared by pipetting 5-mL of the listed simulant with 2.5-mL of DI water plus 2.5-mL of HNO_3 into a polyethylene bottle. An 8-mL aliquot was pipetted into a sample cup put together as mentioned above. For Tests 2-7, the sample cups were placed into the five autosampler positions and analyzed in triplicate under a helium atmosphere.

Since the Rigaku WD-XRF system is a sequential instrument, ~ 30 to 40 min was required for each sample analysis depending on the number of elements scanned. The instrument conditions: peak and background positions, peak and background times, crystals, and detectors can be found in Section 3.5 Table 10. The background subtracted peak intensities, averages, standard deviations, and percent relative standard deviations (%RSD) are tabulated in Appendix C.

3.5 Quantitative Application File

Figure 3 shows the operational flow bar to create a quantitative application file. The directions of how to create a quantitative application file, which start on page 3-6 of the ZSXmini II X-ray

Fluorescence Spectrometer Instruction Manual, are well outlined, and therefore will not be documented here.

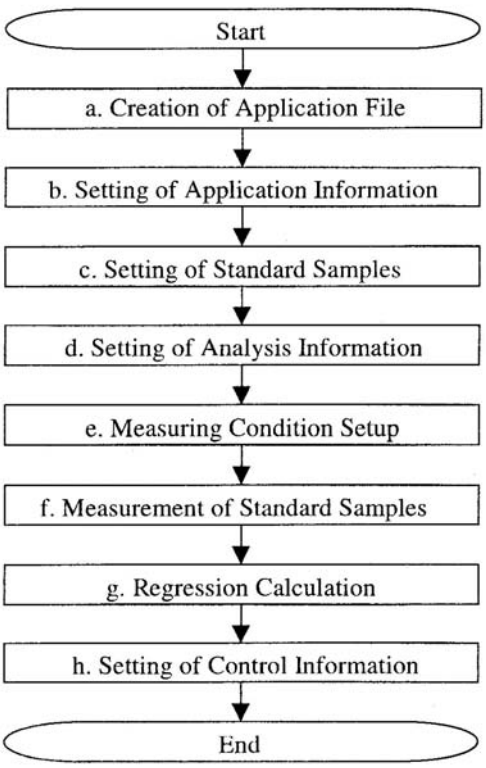

Figure 3. The operational flow bar to create an application file.

The instrument parameters, which where used in the quantification of the simulants representative of the LAW envelopes, are contained in Table 10. The simulants were run using the FP quantification method. There are several restrictions when using the FP approach: (1) The concentration of the standard(s) must equal 99.0 to 101 wt%. Any unmeasured component must be defined as a fixed value, manual input value or a balance component. For example, water (H_2O) was used as a balance component when inputting the standard(s). (2) Elements must be distributed uniformly in the sample. (3) Only matrix effects can be corrected. (4) Standards used to prepare the calibration curve should be similar in concentration to the unknown samples.

Table 10. Instrument Conditions.

Element	Line	Crystal	Detector	Peak (deg)	Time (sec)	BG1 (deg)	Time (sec)	BG2 (deg)	Time (sec)
Na	Kα	RX35[1]	PC[2]	25.202	200	22.500	100	27.500	100
Al	Kα	PET[3]	PC	144.468	200	140.000	100	a	a
P	Kα	PET	PC	89.447	200	91.500	100	a	a
S	Kα	PET	PC	75.687	200	78.000	100	a	a
Cl	Kα	PET	PC	65.401	200	67.500	100	a	a
K	Kα	PET	PC	50.642	200	48.500	100	a	a
Ca	Kα	LIF1[4]	SC[5]	113.004	200	115.000	100	a	a
Cr	Kα	LIF1	SC	69.307	200	70.200	100	a	a
Ni	Kα	LIF1	SC	48.613	200	49.500	100	a	a
Mo	Kα	LIF1	SC	20.228	200	19.720	100	20.920	100

[1] W/Si multilayer diffraction crystal, 2d=55Å.

[2] 10% methane/90% argon flow proportional counter.

[3] Pentaerythritol diffraction crystal (020), 2d=8.808Å.

[4] Lithium fluoride (200) crystal, 2d=4.027Å.

[5] Sodium Iodide scintillation detector.

a Only one background point measured.

3.5 Simulant and Standard Preparations

A direct liquid analysis method was used to prepare the simulant representative of the LAW Envelopes and the standards. Two preparation methods were evaluated on the simulants: basic and acidic. The simulant samples in both methods were diluted to match to the lower elemental concentration in the standards. In the basic method, 5-mL of the simulant was pipetted into a polyethylene bottle along with 5-mL of DI water. In the acid method, 5-mL of simulant was pipetted into a polyethylene bottle along with 2.5-mL of DI water and 2.5-mL of 70% HNO_3. An aliquot of 8-mL of the above mixtures was pipetted out of the polyethylene bottles for each method into a 31-mm diameter circular sample cell, which was covered on the bottom by a piece of 4-μm Ultralene™ (polyethylene). The choice of Ultralene™ over other films was reported by Jurgensen. Also, an aliquot of 8-mL was pipetted directly out of the standard bottles into a circular sample cup. The top of the sample cell was covered with a microporous Teflon™ film that allows for pressure equalization during the analysis. This microporous film prevents the sample surface from distending outwards as the sample is heated, changing the sample to the x-ray source and detector distances. Film preparation was the same for simulants as for the standards. The sample cells were placed in the aluminum holders on the 12-position sample wheel and were analyzed under helium atmosphere, 99.999% He, to minimize light element x-ray absorption.

3.6 Calibration

X-ray spectrometry is essentially a comparative technique. It is absolutely vital that both calibration reference materials and samples to be analyzed are prepared in an identical and reproducible manner and presented and analyzed by the spectrometer under the same

experimental conditions. Table 8 in Section 3.3 lists the concentrations of the ten elements that makeup the five standards. As mentioned earlier, the standards were manufactured by High Purity Standards in Charleston, South Carolina. The Standard Certificates of Analysis and the MSDS's can be found in Appendix B.

The XRF instrument was calibrated using the fundamental parameters algorithm supplied by Rigaku/MSC. The fundamental parameters approach is the derivation of mathematical expressions quantifying fluorescence emissions in terms of fundamental physical parameter and instrumental parameters. Although this method can be standardless, more accurate determinations can be made by analyzing known matrix-matched standards to adjust the primary and secondary absorption and fluorescence factors, jump ratios, and other coefficients in the fundamental parameter algorithm. Relative intensities are calculated from theoretical principles using the fundamental parameter algorithm based on the Sherman equation. These theoretical intensities are compared with the measured intensities, the projected sample composition is adjusted to match these theoretical intensities, and the process is repeated until convergence. Representative calibration curves based on using all five standards and DI water can be found in Appendix D.

Three different methods were tested using this fundamental parameters approach:

Method 1. Calibrating the instrument using the five High Purity standards and then analyzing the filtered diluted (1:1) acidic simulants.

Method 2. Calibrating the instrument using the five High Purity standards and then analyzing the filtered diluted (1:1) basic simulants.

Method 3. Calibrating the instrument using four High Purity standards one time, and then daily using a High Purity standard as a drift monitor on filtered diluted (1:1) basic simulants.

The instrument conditions, peak and background positions, peak and background times, crystals, and detectors can be found in Section 3.5 Table 10. The simulants and standards were prepared as outlined in Section 3.5. The calibration of the instrument was conducted every day the simulants were run for Methods 1 and 2. The total time to calibrate the instrument for Methods 1 and 2 was ~6 hours. To check the calibration of the instrument, one of the standards was used as a Quality Control, QC, check standard. The QC standard was changed from run to run. Appendix E Table E-4 contains the results for the QC.

4. Results and Discussion

4.1 Precision

When comparing the results from the first two tests, the %RSD values for both tests are similar to Jurgensen indicating adequate installation. Table 11 shows the results of this comparison.

Table 11. Test 1 and Test 2 %RSD.

	Test 1[a] % RSD	Test 2[b] % RSD
Na	5.6	3.8
Al	2.7	2.2
P	3.0	5.0
Cl	0.4	2.6
K	3.8	3.3
Cr	0.8	2.0

[a]Triplicate analysis on ten samples.
[b]Triplicate analysis on five samples.

In the earlier precision studies, there was sample degradation, particularly for Al and K. The reason for these intensity drifts is unknown. Likely candidates for intensity drifts are sample heating by the x-ray source and distension of the sample film toward the x-ray tube as it heats or possibly degradation of the solutes and/or complexing agents by the intense x-ray beam. These intensity drifts were observed in Tests 2-6, but not to the extent as in the previous precision study. One reason that these intensity drifts were not as prevalent in Tests 2-6 is that the samples were diluted. Another reason that the intensity drifts were not as pronounced was samples were heated inside the instrument for a shorter time period.

Even with these intensity drifts in Test 2-6, the %RSD is <10% with the exception of molybdenum which has a very low concentration in the AN-105 and AP-101 simulants. The last precision test compared the difference of using a basic solution, Test 6, versus an acidified solution, Test 7. AP-101 simulant was used for this comparison. The results show that the %RSD improved when the simulant was converted from a basic solution to an acidic solution with the exception of molybdenum. The %RSD for all elements with the exception of molybdenum in the acidic test were <5%. The molybdenum precision should be significantly better for both tests since molybdenum is a heavy element, but the samples are not infinitely thick for Mo Kα x-rays. The lower values in the acidic test indicate that the samples are more stable and not hampered by precipitation issues. Table 12 shows the result of this comparison.

Table 12. Basic and Acidic AP-101 Simulant %RSD.

	Basic AP-101	Acidic AP-101
Na	3.7	2.8
Al	7.7	2.0
P	2.1	1.9
Cl	1.7	1.0
K	0.9	0.5
Cr	1.8	1.5
Mo	12.0	19.6
S	5.0	1.1

4.2 XRF Results

Method 3 used a drift monitor after the initial calibration. A drift monitor is a sample or standard that corrects long term drifts of the X-ray intensities. The drift monitor sample must be stable over long periods of time and contain all the analytes at high concentrations. Normally a glass or metal standard with high analyte concentrations is used as the drift monitor. By using a drift monitor, one would calibrate the instrument initially, which takes ~6 hours, and then daily run the drift monitor before any analysis. The drift monitor method would save ~5 hours of calibration time. The instrument was calibrated for this test using Standards B through E. Standard A was used as the QC standard, since this standard was from a different lot of starting material compared to the other standards, and Standard D was used as the drift monitor.

The results of the three methods can be found in Appendix E. Method 1 was run eleven times while Methods 2 and 3 were run only five times. Table 13 compares the average values for the three methods. The results for Methods 2 and 3 are higher than the Method 1. One reason for the higher results in Method 2 is that the simulants and standards matrices were different. The simulants were basic while the standards were prepared in 4% HNO_3. Another reason for the differences in concentrations between these two methods could have come from simulant being degraded by the intense x-ray beam. The drift monitor, Standard D, used in Method 3 to correct the drift of the x-ray intensities on a daily basis under-corrected the intensities, causing the concentration higher than Method 1. This imperfect correction is expected on using a lower concentration liquid standard as the drift monitor. Better results could be obtained using a standard with concentrations between 1-5wt% for corrections to achieve intensities near 1,000,000 counts.

High sodium concentration was observed in one DI water blank for Methods 1 and 2. The potential source of the sodium contamination is the high salt simulants.

Table 13. WD-XRF Average Results for the Three Data Sets.

Element	Acidified[1] AN-105 XRF average µg/mL	Basic[2] AN-105 XRF average µg/mL	Drift Corrected[2] AN-105 XRF average µg/mL	Acidified[1] AP-101 XRF average µg/mL	Basic[2] AP-101 XRF average µg/mL	Drift Corrected[2] AP-101 XRF average µg/mL
Al	11400	12400	12000	7060	7530	7470
Ca	<10	<10	<10	<10	<10	<10
Cr	684	745	690	155	172	157
Mo	41	43	40	14	15	13
Ni	<2	<2	<2	<2	<2	<2
K	3880	4240	3960	28100	30600	28600
Na	129000	142000	137000	123000	134000	130000
S (also measured as SO_4)	389	482	452	3690	4040	3820
P (also measured as PO_4)	356	389	307	1290	1390	1340
Cl	4970	5510	5360	1570	1730	1710

Element	Acidified[1] AN-107 XRF average µg/mL	Basic[2] AN-107 XRF average µg/mL	Drift Corrected[2] AN-107 XRF average µg/mL	Acidified[1] AZ-101 XRF average µg/mL	Basic[2] AZ-101 XRF average µg/mL	Drift Corrected[2] AZ-101 XRF average µg/mL
Al	281	346	414	<80	<80	<80
Ca	147	164	155	<10	<10	<10
Cr	<5	<5	<5	150	169	157
Mo	21	21	19	25	27	24
Ni	305	335	307	<2	<2	<2
K	1180	1320	1200	902	1040	911
Na	145000	160000	152000	9780	12300	13200
S (also measured as SO_4)	4970	5510	4750	829	972	900
P (also measured as PO_4)	935	1050	955	<30	<30	<30
Cl	1350	1530	1470	281	312	314

[1]An average of 11 runs.
[2]An average of 5 runs.

The average results for Method 1 were compared against the average results for the ICP-AES and IC results for the simulants. Table 14 shows this comparison. The average results for the ICP-AES were based on ten runs using ten different calibrations. The average results for the IC were based on 5 runs using five different calibrations. Because of matrix effects, sulfate number was not reported on the IC. The results for all the runs for both the ICP-AES and IC are located in Appendix F. A thorough statistical analysis of this data can be found in SRNL-SCS-2007-00042[6]. "Although measurements obtained by XRF (both acidic, Method 1, and basic, Method 2, preparations) are different from those obtained by ICP-AES and IC, the XRF acidic preparation measurements are sufficiently similar to the measurements made by ICP-AES or IC that either method could be used. The variance analysis suggests that ICP-AES and IC tend to be roughly equal to XRF in terms of uncertainty. Furthermore, the various analyses on the mean measurements indicate that ICP-AES and IC tend to yield smaller measurements than XRF, especially when the XRF samples are prepared using the basic solution. In particular, the XRF method does return smaller mean measurements than ICP-AES or IC for some of the elements of interest when the XRF samples are prepared using the acid solutions. When all ten of the measured elements are considered together, it appears that the overall analysis obtained using the acidic preparation for XRF is statistically equivalent to that obtained by ICP-AES and IC[6]."

[6] M. D. Joner, "A Statistical Comparison of XRF, ICP-AES, and IC Measurements", SRNL-SCS-2007-00042, Washington Savannah River Company, (to be issued).

Table 14. Average Concentrations for the WD-XRF, Method 1, ICP-AES, and IC.

Element	AN-105 XRF average µg/mL	AN-105 ICP Average µg/mL	AN-105 IC Average µg/mL	AP-101 XRF average µg/mL	AP-101 ICP Average µg/mL	AP-101 IC Average µg/mL
Al	11400	10600	a	7060	6850	a
Ca	<10	<3.81	a	<10	<3.81	a
Cr	684	640	a	155	152	a
Mo	41	40	a	14	14	a
Ni	<2	1	a	<2	<0.35	a
K	3880	3770	a	28100	28200	a
Na	129000	122000	a	123000	120000	a
S (also measured as SO_4)	389	393	140	3690	3810	3450
P (also measured as PO_4)	356	289	b	1290	1210	b
Cl	4970	a	4600	1570	a	1490

Element	AN-107 XRF average µg/mL	AN-107 ICP Average µg/mL	AN-107 IC Average µg/mL	AZ-101 XRF average µg/mL	AZ-101 ICP Average µg/mL	AZ-101 IC Average µg/mL
Al	281	236	a	<80	<2.56	a
Ca	147	145	a	<10	<3.81	a
Cr	<5	0.64	a	148	142	a
Mo	21	22	a	26	26	a
Ni	305	301	a	<2	<0.558	a
K	1180	1200	a	932	781	a
Na	145000	141000	a	9780	11500	a
S (also measured as SO_4)	4970	5200	4710	845	800	748
P (also measured as PO_4)	935	894	b	<30	<60	b
Cl	1350	a	1500	306	a	259

a) These elements could not measured by the given method.
b) Because of martix effects, PO_4^{2-} was not reported.

Tables 15 through 18 compare the average results and one sigma values between Method 1 to the ICP-AES and IC results for each element in the four simulants. The less than values reported in Tables 13 through 18 for Al, Ca, Ni, Cr, and PO4 are very good estimates of the detection limits, which were determined by making several dilutions from ICP-AES and IC standards. Each set of dilutions for a particular element was scanned included a DI water blank, and the scans were overlaid in the software. The XRF less than values were estimated to be where analyte peak was determined to be above the DI water blank spectra. A typical method at SRNL to determine the detection limits is the IUPAC calculation, as detailed by Jurgensen.

Table 15. AN-105 Average Concentrations and Standard Deviation for the WD-XRF Method 1, ICP-AES, and IC.

Element	AN-105 XRF average µg/mL	1σ	AN-105 ICP Average µg/mL	1σ	AN-105 IC Average µg/mL	1σ
Al	11400	339	10600	516	a	-
Ca	<10	-	<3.81	-	a	-
Cr	684	11	640	35	a	-
Mo	41	2	40	2.3	a	-
Ni	<2	-	<0.35	-	a	-
K	3880	74	3700	183	a	-
Na	129000	7650	122000	6350	a	-
S (also measured as SO_4)	389	65	398	23	140	13
P (also measured as PO_4)	356	18	289	15	b	-
Cl	4970	115	a	-	4600	82

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

Table 16. AP-101 Average Concentrations and Standard Deviation for WD-XRF Method 1, ICP-AES, and IC.

Element	AP-101 XRF average µg/mL	1σ	AP-101 ICP Average µg/mL	1σ	AP-101 IC Average µg/mL	1σ
Al	7060	178	6850	99	a	-
Ca	<10	-	<3.81	-	a	-
Cr	155	4	152	4	a	-
Mo	14	2	14	1	a	-
Ni	<2	-	<0.35	-	a	-
K	28100	273	28200	673	a	-
Na	123000	4460	120000	3300	a	-
S (also measured as SO_4)	3690	82	3810	89	3450	34
P (also measured as PO_4)	1290	39	1210	40	b	-
Cl	1570	23	a	-	1490	21

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

Table 17. AN-107 Average Concentrations and Standard Deviation for the WD-XRF Method 1, ICP-AES, and IC.

Element	AN-107 XRF average µg/mL	1σ	AN-107 ICP Average µg/mL	1σ	AN-107 IC Average µg/mL	1σ
Al	281	67	236	3	a	-
Ca	147	8	145	3	a	-
Cr	<5	-	1	0.2	a	-
Mo	21	2	22	0.7	a	-
Ni	305	4	301	8	a	-
K	1180	47	1200	67	a	-
Na	145000	5820	141000	4820	a	-
S (also measured as SO_4)	4970	73	5200	131	4710	58
P (also measured as PO_4)	935	40	894	17	b	-
Cl	1350	27	a	-	1500	156

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

Table 18. AZ-101 Average Concentrations and Standard Deviation for the WD-XRF Method 1, ICP-AES, and IC.

Element	AZ-101 XRF average µg/mL	1σ	AZ-101 ICP Average µg/mL	1σ	AZ-101 IC Average µg/mL	1σ
Al	<80	-	<2.56	-	a	-
Ca	<10	-	<3.81	-	a	-
Cr	150	6	142	3	a	-
Mo	25	1	26	1	a	-
Ni	<2	-	<0.558	-	a	-
K	902	56	781	25	a	-
Na	9780	761	11500	247	a	-
S (also measured as SO_4)	829	46	801	21	748	50
P (also measured as PO_4)	<30	-	<60	-	b	-
Cl	281	16	a	-	259	14

a) These elements could not be measured by the given method.

b) Because of martix effects, PO_4^{2-} was not reported.

Silicon concentration in the simulants was not determined by XRF because High Purity had problems with silicon precipitating out of solution. The XRF detection limit for silicon was estimated to be < 30-µg/mL. A High Purity ICP-AES 1000-µg/mL Si in H_2O standard was used to estimate the detection limit. The detection limit for silicon was determined in the same way as mentioned above for Al, Ca, Cr, Ni, and PO_4.

4.3 X-ray Fluorescence (XRF) Counting Statistics

The relative fractional counting uncertainty was determined for the 11 acidified runs and the 5 basic runs using all five High Purity standards in the calibration of the instrument. The relative fractional counting uncertainty was calculated using the following equation:

$$\varepsilon_N = \frac{\sqrt{N}}{N} \times 100$$

where

ε_N is the relative standard deviation of the individual measurement
N is the number of counts.

The results for the relative fractional counting uncertainty for these two tests are in Appendix G. By comparing the average relative standard deviation of the simulants to the average relative fractional counting uncertainty, the average relative fractional counting uncertainties are much smaller than the average relative standard deviations for all the elements in the simulants. Table 19 and 20 show the results of the comparison for the 11 acidified runs and five basic runs.

Table 19. Comparing the Overall Precision to the Counting Uncertainty for the Acidic Filtered Diluted Simulants.

Element	AN-105 %RSD	AN-105 Counting Uncertainty (%)	AP-101 %RSD	AP-101 Counting Uncertainty (%)	AN-107 %RSD	AN-107 Counting Uncertainty (%)	AZ-101 %RSD	AZ-101 Counting Uncertainty (%)
Al	3	0.6	3	0.7	24	3.0	-	-
Ca	-	-	-	-	6	2.4	-	-
Cr	2	0.7	2	1.3	-	-	4	1.2
Mo	5	0.2	12	0.2	9	0.2	4	0.2
Ni	-	-	-	-	1	0.5	-	-
K	2	0.4	1	0.2	4	0.5	6	0.6
Na	6	0.9	4	0.9	4	0.9	8	2.2
S (also measured as SO_4)	17	1.9	2	0.8	1	0.7	6	1.4
P (also measured as PO_4)	5	3.4	3	2.0	4	2.3	-	-
Cl	2	0.3	1	0.5	2	0.6	6	1.0

Table 20. Comparing the Overall Precision to the Counting Uncertainty for the Basic Filtered Diluted Simulants.

Element	AN-105 %RSD	AN-105 Counting Uncertainty (%)	AP-101 %RSD	AP-101 Counting Uncertainty (%)	AN-107 %RSD	AN-107 Counting Uncertainty (%)	AZ-101 %RSD	AZ-101 Counting Uncertainty (%)
Al	6	0.6	2	0.7	30	2.9	-	-
Ca	-	-	-	-	7	2.3	-	-
Cr	2	0.7	4	1.2	-	-	3	1.1
Mo	4	0.2	15	0.2	11	0.2	7	0.2
Ni	-	-	-	-	1	0.4	-	-
K	2	0.4	1	0.2	2	0.5	5	0.5
Na	3	0.9	4	0.9	4	0.8	23	2.1
S (also measured as SO_4)	22	1.8	3	0.7	2	0.6	11	1.3
P (also measured as PO_4)	6	3.3	4	1.9	3	2.2	-	-
Cl	2	0.3	2	0.5	2	0.5	2	1.0

The results indicate that the major source of error is not the instrumental measurement uncertainty, but rather is associated with other preparation and analytical factors, such as preparing the sample cups, distension of the sample film towards the x-ray tube as the sample film heats, or possible degradation of the solutions by the x-ray beam. An additional note regarding sample cups preparation. The 4-μm Ultralene™ film needs to be as flat as possible to avoid any ripples that will distend the film closer to the x-ray source affecting the final results of the measurement.

5. Quality Assurance

The Quality Assurance measures identified in the Task Technical and Quality Assurance Plan[2] were followed in the performance of these activities. The WSRC program applied the appropriate quality assurance requirements from 10 CFR 830 Subpart A, NQA-1-1989 (Part I, Basic and Supplementary Requirements), and NQA-2a-1990, Part 2.7, as indicated by the QA Plan Checklist in Section VIII of the Task Technical and Quality Assurance Plan. A surveillance of the activities was performed by SRNL QA to verify conformance to the QA Plan Checklist. All items that were checked "Yes" in this list were followed with the exception of procedures related to stop work, non-conformance, and the corrective action system. These were not necessary since no issues were identified. SRNL was also requested to perform work in accordance with the requirements established in Quality Assurance Project Plan for Testing Programs Generating Environmental Regulatory Data (PL-24590-QA00001[7]) since the activity supports regulatory and environmental testing for the RPP-WTP.

5.1 Significant Figures

The number of significant figures used in this document was based on the following criteria:

- XRF raw data in kcps was input as listed in the instrument printout.
- The number of significant figures for the data set averages, %RSDs, etc. was based on an estimate of what was appropriate for that particular method at that concentration level.

[7] D. Blumenkranz, "Quality Assurance Project for Testing Programs Generating Environmental Regulatory Data", PL-24590-QA00001, Revision 0, June 2001.

6. Conclusions/ Recommendations

In the precision studies where the basic simulant was run, there was some sample degradation. The reason for these intensity drifts is unknown, but could have been from sample heating by the x-ray source and distension of the sample film toward the x-ray tube as it heats or possibly degradation of the solutes and/or complexing agents by the intense x-ray beam. The precision improved slightly when the basic simulant was diluted 1:1 with DI water, but the problem did not disappear. The results for the precision study where the basic stimulant, AP-101, was converted to an acidic solution were not hampered by the intensity drifts resolving the long term stability that was indicated in Phase 1a.

Three XRF fundamental parameter methods were studied. The results of each XRF method were compared against the results from the ICP-AES and IC methods. The results from the Method 2 and Method 3 were different than the results obtained from the ICP-AES and IC methods. The variance analysis suggests that ICP-AES and IC tend to be roughly equal to Method 1 in terms of uncertainty[5]. The results for Method 1 are statistically comparable to the results of ICP-AES and IC.

SRNL developed an XRF method for WTP LAW vitrification processing support after procuring valid calibration and verification standards that represent LAW compositional ranges. SRNL recommends that when using the XRF fundamental parameters method that the matrix matched standards closely match the waste composition. If the matrix matched standards for the major elements, Al and Na, are 20% away from the actual composition, the accuracy and precision of the results will suffer.

Information provided in the Riguka's User manual is sufficient for WTP implementation of this XRF method. The user manual is easy to follow with step by step instructions and pictures of the various steps.

7. Appendices

7.1 Appendix A: Simulants Hold Point Approval

"Arakali, Aruna"
<avarakal@bechtel.com>
06/21/2007 02:08 PM

To frank.pennebaker@srnl.doe.gov
cc david.missimer@srnl.doe.gov, arthur.jurgensen@srnl.doe.gov, "Lane, Thomas A" <talane@bechtel.com>, "Lau, Barbara"
bcc
Subject WTP Hold Point Approval

Frank,
Reviewed the compositional information on selection of LAW simulants. The listed simulants are approved for using as test samples. Proceed with completion of XRF Phase 1b task.
Thanks

Aruna V. Arakali
WTP Lab Team
River Protection Project-Waste Treatment Plant
2435 Stevens Center Place MS12-B,
Richland, WA 99354

From: frank.pennebaker@srnl.doe.gov [mailto:frank.pennebaker@srnl.doe.gov]
Sent: Thursday, June 21, 2007 10:31 AM
To: Arakali, Aruna
Cc: david.missimer@srnl.doe.gov; arthur.jurgensen@srnl.doe.gov
Subject: WTP Hold Point

Aruna,

SRNL requests WTP approval for the use of simulants as defined in SRNL-RPP-2004-000091Rev3 Table 6. The task plan designates a hold point for Simulant Sample Selection. SRNL requests use of the following simulants.

- Envelope A - Simulant AN-105 was made by the Full Eibling recipe and diluted to 5M Sodium.
- Envelope A - Simulant AP-101 full recipe
- Envelope C - Simulant AN-107 was produced by a stronium/TRU precipitation after caustic adjustment.
- Envelope B/D simulant **with some unknown elemental concentrations** was prepared by Optima for Gas Release/ Holdup Tesing. The slurry was centrifuged to separate the solids from the supernate, and then filtered. **Some of the values measured below detection limit will be measured by XRF to evaluate performance.**

I have included supplemental documents, which give the elemental concentration of each of these simulants, as measured by SRNL. I have also included documentation for the Envelope B/D slurry, prior to filtration. We must complete selection of simulants by June 21 to maintain schedule. If you have any questions, please let me know. Thanks.

Frank Pennebaker
Mgr, Materials Characterization and Nuclear Measurements Group
Savannah River National Laboratory
(803)725-6749

7.2 Appendix B: Standard Certificates of Analysis and Material Safety Data Sheets

P.O. Box 41727
Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-744-042
(Standard A)
Lot # 0711714

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.995+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES and IC against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for three months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions. Expiration date may be extended as stability is determined. This standard was prepared from different master solutions than the other standards in this series.

Theodore C. Rains, Ph.D.
President

Exp Date: AUG 0 5 2007
MSDS ATTACHED

Figure B - 1. Standard A Certificate of Analysis.

SM-744-042
(Standard A)
Element List
(µg/mL)

Aluminum	8850
Calcium	500
Chromium	250
Molybdenum from $(NH_4)_2MoO_4$	5
Nickel	375
Potassium	225
Sodium	36,500
Chloride	50
Phosphate	2450
Sulfate	900

Figure B - 1. Continued.

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Phone: (843) 767-7900
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MATERIAL SAFETY DATA SHEET

SM-744-042
(Standard A)

Issue Date: 04/27/07

SECTION I - Product Identification/Hazardous Ingredients

Formula: N/A Concentration: N/A Molecular Weight: N/A

TSCA: YES CAS NO: 7697-37-2

Component: 5.01% Multielements including(0.04% Ni) in 4% HNO_3 + balance H_2O

TLV/TWA: 8 h Not Estab.: 5 mg/m^3 STEL: N/A PEL: N/A Toxicity: N/A

SECTION II - Physical/Chemical Characteristics

Boiling Point: 100°C Vapor Pressure (mm): N/A Vapor Density (air+1): N/A

Freezing Point: N/A Specific Gravity (H_2O = 1): N/A Solubility in H_2O: Complete

SECTION III - Fire and Explosion Hazard Data

Flash Point: N/A Auto Ignition Temperature: N/A Lower Explosion Level: N/A

NFPA - Rating: N/A Extinguishing Media: Use appropriate

Special Fire-Fighting Procedures: Firefighters should wear proper protective equipment and self-contained breathing apparatus with full face piece operated in positive pressure mode.

Unusual Fires Explosion Hazards: N/A

Toxic Gases Produced: NO_X

Figure B - 2. Standard A Material Data Safety Sheet (MSDS).

SECTION IV - Reacting Data

Unstable : ( ) Stable: (X)

Conditions to Avoid: Metals, hydroxides, carbonates, cyanides

Incompatibles: Strong reducing agents

Hazardous Decomposition: NO_X

SECTION V - Health Hazard Data

Routes of Entry: Inhalation, eye contact, skin contact

Signs and Symptoms of Exposure: Liquid may cause burns to skin and eyes

Medical Conditions Generally Aggravated by Exposure: None identified

Carcinogenicity: NTP: Yes(Ni) IARC: Yes(Ni) OSHA reg.: Yes(Ni)

Emergency First Aid Procedures: CALL A PHYSICIAN; If swallowed, do not induce vomiting, if conscious give water, milk. In case of contact, flush eyes or skin with plenty of water.

SECTION VI - Precautions for Safe Handling and Use

Special Precautions: Keep container tightly closed

In Case of Spill or Discharge: Remove source of ignition if hydrogen is a hazard. Provide optimum ventilation. Flush to holding area for neutralization.

Disposal Procedures: Follow Federal, State and Local regulations for waste.

SECTION VII - Protective Equipment

Respiratory Protection: NIOSH approved respirator

Ventilation: Local Exhaust(X) Mechanical ( )

Protective Gloves: Proper gloves

Eye Protection: Safety glasses with side shields

Other: Lab coat/apron; vent hood

NOTICE

The data and information as stated was furnished by the manufacturers/vendors & or suppliers of this products. High-Purity Standards, Inc. cannot warrant the accuracy of this information and shall not be responsible or liable for any damage that may result, should any of the information be erroneous.
Prepared by: Theodore C Rains, Ph.D 04/27/07

Figure B - 2. Continued

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HIGH-PURITY STANDARDS

Certificate of Analysis

SM-744-043
(Standard B)
Lot # 0709521

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.995+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC).

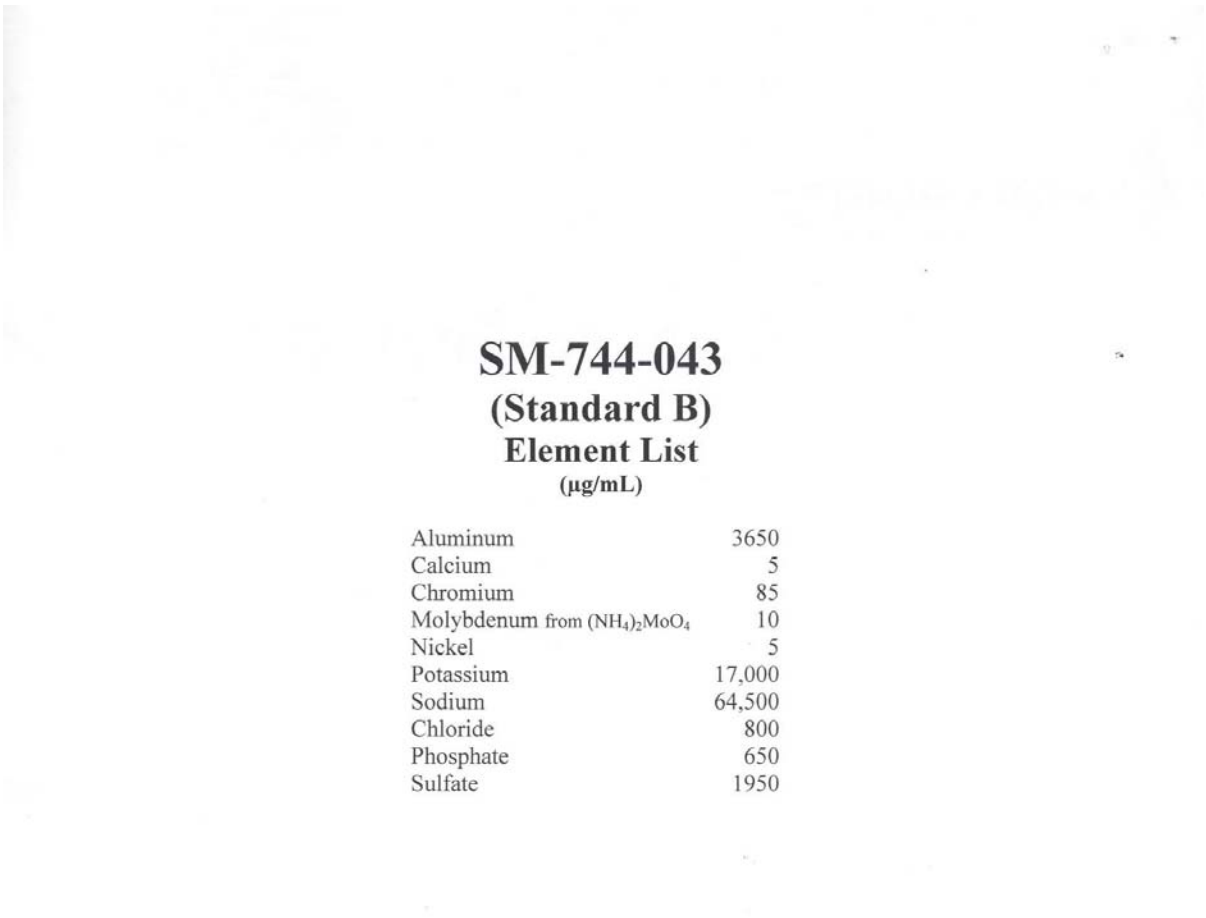
The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES and IC against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for three months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions. Expiration date may be extended as stability is determined.

Theodore C. Rains, Ph.D.
President

Exp Date:
MSDS ATTACHED
ISSUE DATE: MAY 0 8 2007

Figure B - 3. Standard B Certificate of Analysis



SM-744-043
(Standard B)
Element List
(µg/mL)

Aluminum	3650
Calcium	5
Chromium	85
Molybdenum from $(NH_4)_2MoO_4$	10
Nickel	5
Potassium	17,000
Sodium	64,500
Chloride	800
Phosphate	650
Sulfate	1950

Figure B - 3. Continued.

HIGH-PURITY STANDARDS
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Phone: (843) 767-7900
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MATERIAL SAFETY DATA SHEET

SM-744-043
(Standard B)

Issue Date: 4/6/07

SECTION I - Product Identification/Hazardous Ingredients

Formula: N/A Concentration: N/A Molecular Weight: N/A

TSCA: YES CAS NO: 7697-37-2

Component: 8.87% Multielements including(0.001% Ni) in 4% HNO_3 + balance H_2O

TLV/TWA: 8 h Not Estab.; 5 mg/m^3 STEL: N/A PEL: N/A Toxicity: N/A

SECTION II - Physical/Chemical Characteristics

Boiling Point: 100°C Vapor Pressure (mm): N/A Vapor Density (air+1): N/A

Freezing Point: N/A Specific Gravity (H_2O = 1): N/A Solubility in H_2O: Complete

SECTION III - Fire and Explosion Hazard Data

Flash Point: N/A Auto Ignition Temperature: N/A Lower Explosion Level: N/A

NFPA - Rating: N/A Extinguishing Media: Use appropriate

Special Fire-Fighting Procedures: Firefighters should wear proper protective equipment and self-contained breathing apparatus with full face piece operated in positive pressure mode.

Unusual Fires Explosion Hazards: N/A

Toxic Gases Produced: NO_X

Figure B - 4. Standard B Material Data Safety Sheet (MSDS).

SECTION IV - Reacting Data

Unstable : ( ) Stable: (X)

Conditions to Avoid: Metals, hydroxides, carbonates, cyanides

Incompatibles: Strong reducing agents

Hazardous Decomposition: NO_X

SECTION V - Health Hazard Data

Routes of Entry: Inhalation, eye contact, skin contact

Signs and Symptoms of Exposure: Liquid may cause burns to skin and eyes

Medical Conditions Generally Aggravated by Exposure: None identified

Carcinogenicity: NTP: Yes(Ni) IARC: Yes(Ni) OSHA reg.: Yes(Ni)

Emergency First Aid Procedures: CALL A PHYSICIAN; If swallowed, do not induce vomiting, if conscious give water, milk. In case of contact, flush eyes or skin with plenty of water.

SECTION VI - Precautions for Safe Handling and Use

Special Precautions: Keep container tightly closed

In Case of Spill or Discharge: Remove source of ignition if hydrogen is a hazard. Provide optimum ventilation. Flush to holding area for neutralization.

Disposal Procedures: Follow Federal, State and Local regulations for waste.

SECTION VII - Protective Equipment

Respiratory Protection: NIOSH approved respirator

Ventilation: Local Exhaust(X) Mechanical ( )

Protective Gloves: Proper gloves

Eye Protection: Safety glasses with side shields

Other: Lab coat/apron; vent hood

NOTICE

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Prepared by: Theodore C Rains, Ph.D 4/6/07

Figure B - 4. Continued.

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TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-744-044
(Standard C)
Lot # 0710007

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.997+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES and IC against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

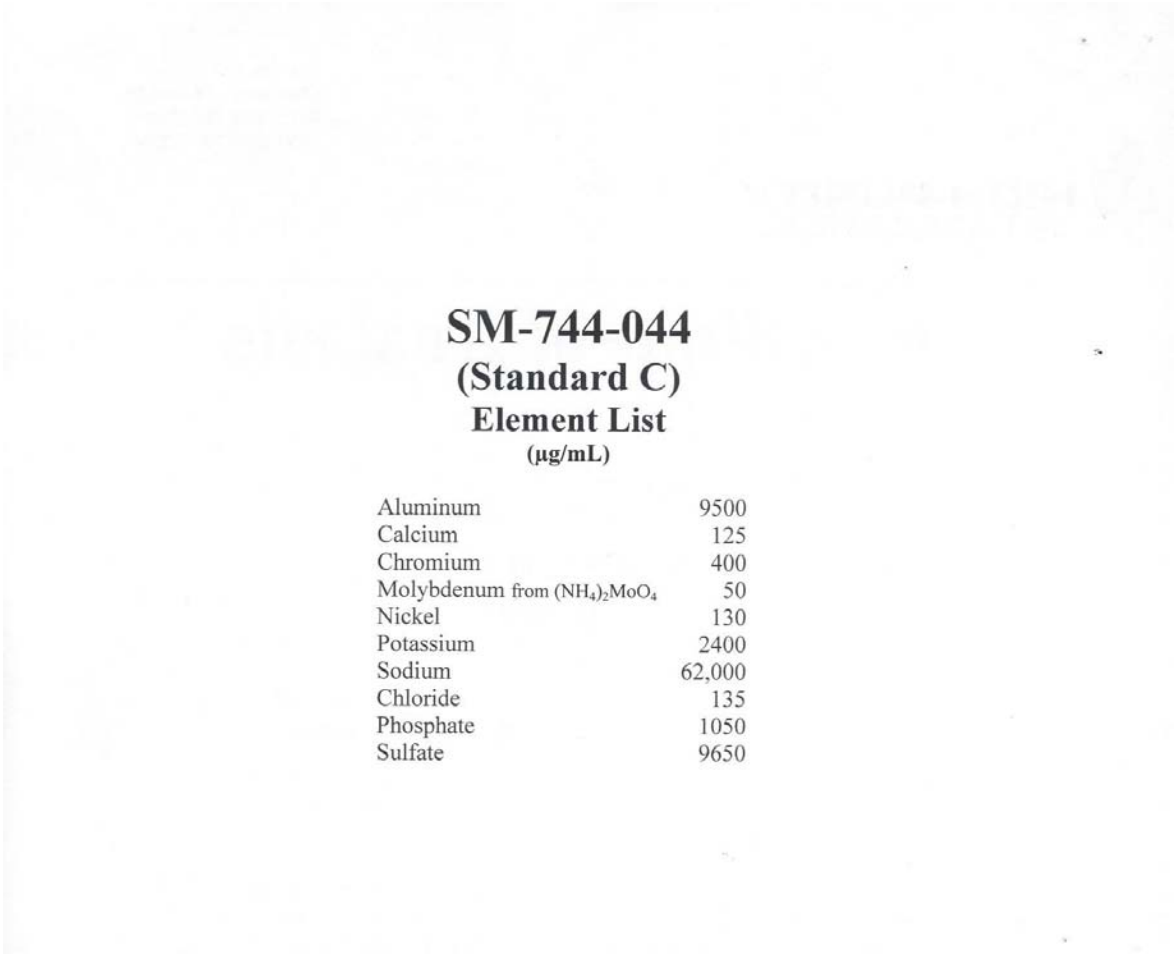
This standard is valid for three months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions. Expiration date may be extended as stability is determined.

Theodore C Rains

Theodore C. Rains, Ph.D.
President

Exp Date:
MSDS ATTACHED
Issue date: MAY 0 8 2007

Figure B - 5. Standard C Certificate of Analysis.



SM-744-044
(Standard C)
Element List
(µg/mL)

Aluminum	9500
Calcium	125
Chromium	400
Molybdenum from $(NH_4)_2MoO_4$	50
Nickel	130
Potassium	2400
Sodium	62,000
Chloride	135
Phosphate	1050
Sulfate	9650

Figure B - 5. Continued.

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Fax: (843) 767-7906

MATERIAL SAFETY DATA SHEET

SM-744-044
(Standard C)

Issue Date: 04/27/07

SECTION I - Product Identification/Hazardous Ingredients

Formula: N/A Concentration: N/A Molecular Weight: N/A

TSCA: YES CAS NO: 7697-37-2

Component: 8.54% Multielements including(0.013% Ni) in 4% HNO_3 + balance H_2O

TLV/TWA: 8 h Not Estab.; 5 mg/m^3 STEL: N/A PEL: N/A Toxicity: N/A

SECTION II - Physical/Chemical Characteristics

Boiling Point: 100ºC Vapor Pressure (mm): N/A Vapor Density (air+1): N/A

Freezing Point: N/A Specific Gravity (H_2O = 1): N/A Solubility in H_2O: Complete

SECTION III - Fire and Explosion Hazard Data

Flash Point: N/A Auto Ignition Temperature: N/A Lower Explosion Level: N/A

NFPA - Rating: N/A Extinguishing Media: Use appropriate

Special Fire-Fighting Procedures: Firefighters should wear proper protective equipment and self-contained breathing apparatus with full face piece operated in positive pressure mode.

Unusual Fires Explosion Hazards: N/A

Toxic Gases Produced: NO_X

Figure B - 6. Standard C Material Data Safety Sheet (MSDS).

SECTION IV - Reacting Data

Unstable : ( ) Stable: (X)

Conditions to Avoid: Metals, hydroxides, carbonates, cyanides

Incompatibles: Strong reducing agents

Hazardous Decomposition: NO_X

SECTION V - Health Hazard Data

Routes of Entry: Inhalation, eye contact, skin contact

Signs and Symptoms of Exposure: Liquid may cause burns to skin and eyes

Medical Conditions Generally Aggravated by Exposure: None identified

Carcinogenicity: NTP: Yes(Ni) IARC: Yes(Ni) OSHA reg.: Yes(Ni)

Emergency First Aid Procedures: CALL A PHYSICIAN; If swallowed, do not induce vomiting, if conscious give water, milk. In case of contact, flush eyes or skin with plenty of water.

SECTION VI - Precautions for Safe Handling and Use

Special Precautions: Keep container tightly closed

In Case of Spill or Discharge: Remove source of ignition if hydrogen is a hazard. Provide optimum ventilation. Flush to holding area for neutralization.

Disposal Procedures: Follow Federal, State and Local regulations for waste.

SECTION VII - Protective Equipment

Respiratory Protection: NIOSH approved respirator

Ventilation: Local Exhaust(X) Mechanical ( )

Protective Gloves: Proper gloves

Eye Protection: Safety glasses with side shields

Other: Lab coat/apron; vent hood

NOTICE

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Prepared by: Theodore C Rains, Ph.D 04/27/07

Figure B - 6. Continued.

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Charleston, SC 29423
TEL: (843) 767-7900
FAX: (843) 767-7906

HIGH-PURITY STANDARDS

Certificate of Analysis

SM-744-045
(Standard D)
Lot # 0710008

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.995+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES and IC against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

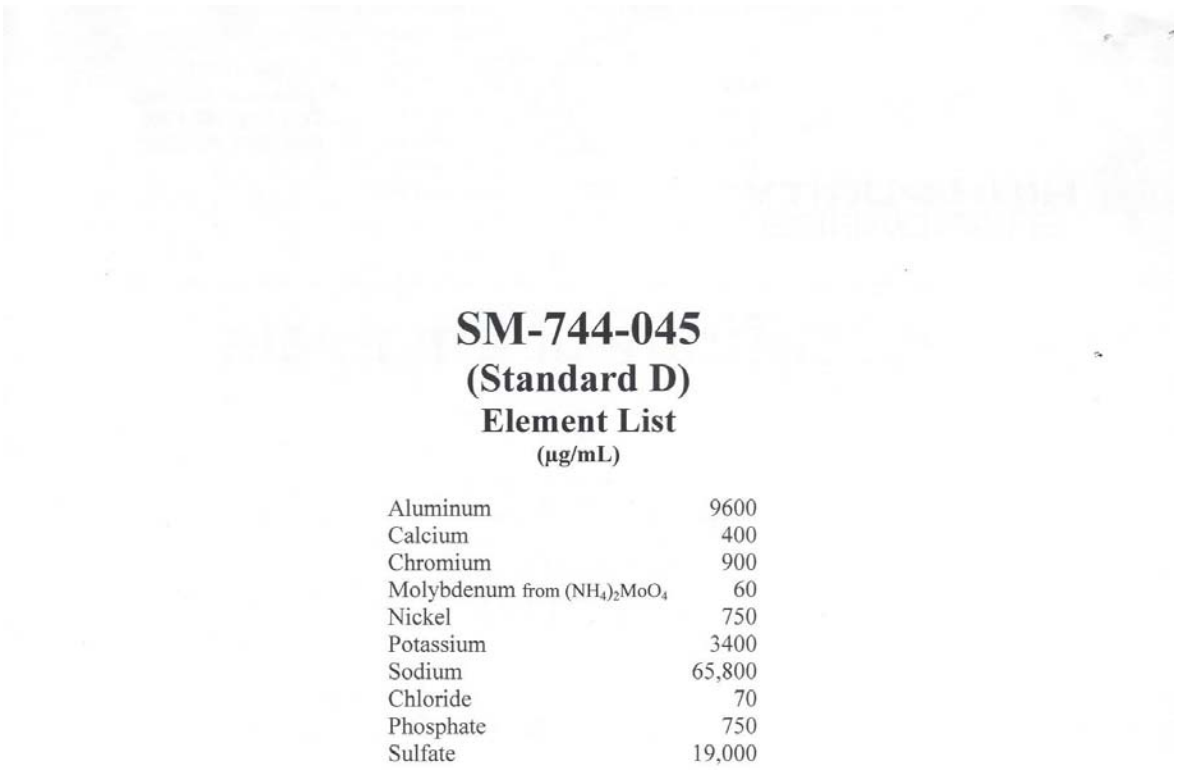
This standard is valid for three months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions. Expiration date may be extended as stability is determined.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date:
MSDS ATTACHED
ISSUE DATE: MAY 0 8 2007

Figure B - 7. Standard D Certificate of Analysis.



SM-744-045
(Standard D)
Element List
(µg/mL)

Aluminum	9600
Calcium	400
Chromium	900
Molybdenum from $(NH_4)_2MoO_4$	60
Nickel	750
Potassium	3400
Sodium	65,800
Chloride	70
Phosphate	750
Sulfate	19,000

Figure B - 7. Continued.

HIGH-PURITY STANDARDS
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MATERIAL SAFETY DATA SHEET

SM-744-045
(Standard D)

Issue Date: 04/27/07

SECTION I - Product Identification/Hazardous Ingredients

Formula: N/A Concentration: N/A Molecular Weight: N/A

TSCA: YES CAS NO: 7697-37-2

Component: 10.07% Multielements including(0.08% Ni) in 4% HNO_3 + balance H_2O

TLV/TWA: 8 h Not Estab.; 5 mg/m^3 STEL: N/A PEL: N/A Toxicity: N/A

SECTION II - Physical/Chemical Characteristics

Boiling Point: 100°C Vapor Pressure (mm): N/A Vapor Density (air+1): N/A

Freezing Point: N/A Specific Gravity (H_2O = 1): N/A Solubility in H_2O: Complete

SECTION III - Fire and Explosion Hazard Data

Flash Point: N/A Auto Ignition Temperature: N/A Lower Explosion Level: N/A

NFPA - Rating: N/A Extinguishing Media: Use appropriate

Special Fire-Fighting Procedures: Firefighters should wear proper protective equipment and self-contained breathing apparatus with full face piece operated in positive pressure mode.

Unusual Fires Explosion Hazards: N/A

Toxic Gases Produced: NO_X

Figure B - 8. Standard D Material Data Safety Sheet (MSDS).

SECTION IV - Reacting Data

Unstable : ( ) Stable: (X)

Conditions to Avoid: Metals, hydroxides, carbonates, cyanides

Incompatibles: Strong reducing agents

Hazardous Decomposition: NO_X

SECTION V - Health Hazard Data

Routes of Entry: Inhalation, eye contact, skin contact

Signs and Symptoms of Exposure: Liquid may cause burns to skin and eyes

Medical Conditions Generally Aggravated by Exposure: None identified

Carcinogenicity: NTP: Yes(Ni) IARC: Yes(Ni) OSHA reg.: Yes(Ni)

Emergency First Aid Procedures: CALL A PHYSICIAN; If swallowed, do not induce vomiting, if conscious give water, milk. In case of contact, flush eyes or skin with plenty of water.

SECTION VI - Precautions for Safe Handling and Use

Special Precautions: Keep container tightly closed

In Case of Spill or Discharge: Remove source of ignition if hydrogen is a hazard. Provide optimum ventilation. Flush to holding area for neutralization.

Disposal Procedures: Follow Federal, State and Local regulations for waste.

SECTION VII - Protective Equipment

Respiratory Protection: NIOSH approved respirator

Ventilation: Local Exhaust(X) Mechanical ( )

Protective Gloves: Proper gloves

Eye Protection: Safety glasses with side shields

Other: Lab coat/apron; vent hood

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Prepared by: Theodore C Rains, Ph.D 04/27/07

Figure B - 8. Continued.

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HIGH-PURITY STANDARDS

Certificate of Analysis

SM-744-046
(Standard E)
Lot # 0709602

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts or Oxides	99.995+%	HNO_3, 4%	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography (IC).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES and IC against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for three months from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions. Expiration date may be extended as stability is determined.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date:
MSDS ATTACHED
ISSUE DATE: MAY 0 8 2007

Figure B - 9. Standard E Certificate of Analysis.

SM-744-046
(Standard E)
Element List
(µg/mL)

Aluminum	9150
Calcium	40
Chromium	300
Molybdenum from $(NH_4)_2MoO_4$	30
Nickel	40
Potassium	3250
Sodium	89000
Chloride	2150
Phosphate	1200
Sulfate	2400

Figure B - 9. Continued.

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Fax: (843) 767-7906

MATERIAL SAFETY DATA SHEET

SM-744-046
(Standard E)

Issue Date: 4/6/07

SECTION I - Product Identification/Hazardous Ingredients

Formula: N/A Concentration: N/A Molecular Weight: N/A

TSCA: YES CAS NO: 7697-37-2

Component: 10.76% Multielements including(0.004% Ni) in 4% HNO_3 + balance H_2O

TLV/TWA: 8 h Not Estab.: 5 mg/m^3 STEL: N/A PEL: N/A Toxicity: N/A

SECTION II - Physical/Chemical Characteristics

Boiling Point: 100°C Vapor Pressure (mm): N/A Vapor Density (air+1): N/A

Freezing Point: N/A Specific Gravity (H_2O = 1): N/A Solubility in H_2O: Complete

SECTION III - Fire and Explosion Hazard Data

Flash Point: N/A Auto Ignition Temperature: N/A Lower Explosion Level: N/A

NFPA - Rating: N/A Extinguishing Media: Use appropriate

Special Fire-Fighting Procedures: Firefighters should wear proper protective equipment and self-contained breathing apparatus with full face piece operated in positive pressure mode.

Unusual Fires Explosion Hazards: N/A

Toxic Gases Produced: NO_X

Figure B - 10. Standard E Material Data Safety Sheet (MSDS).

SECTION IV - Reacting Data

Unstable : ( ) Stable: (X)

Conditions to Avoid: Metals, hydroxides, carbonates, cyanides

Incompatibles: Strong reducing agents

Hazardous Decomposition: NO_X

SECTION V - Health Hazard Data

Routes of Entry: Inhalation, eye contact, skin contact

Signs and Symptoms of Exposure: Liquid may cause burns to skin and eyes

Medical Conditions Generally Aggravated by Exposure: None identified

Carcinogenicity: NTP: Yes(Ni) IARC: Yes(Ni) OSHA reg.: Yes(Ni)

Emergency First Aid Procedures: CALL A PHYSICIAN; If swallowed, do not induce vomiting, if conscious give water, milk. In case of contact, flush eyes or skin with plenty of water.

SECTION VI - Precautions for Safe Handling and Use

Special Precautions: Keep container tightly closed

In Case of Spill or Discharge: Remove source of ignition if hydrogen is a hazard. Provide optimum ventilation. Flush to holding area for neutralization.

Disposal Procedures: Follow Federal, State and Local regulations for waste.

SECTION VII - Protective Equipment

Respiratory Protection: NIOSH approved respirator

Ventilation: Local Exhaust(X) Mechanical ( )

Protective Gloves: Proper gloves

Eye Protection: Safety glasses with side shields

Other: Lab coat/apron; vent hood

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Figure B - 10. Continued.

7.3 Appendix C: Precision

Table C - 1. AN-105 Simulant Solution Precision – Three Runs of Ten Filtered Undiluted Samples in Autosampler from Previous Report[1].

Three consecutive runs with samples in same position on autosampler.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-μm (film was not replaced)
Cell: 31-mm double open end
Sample film Ultralene - 4-μm (same film for each run)
Cover film: Micro-porous Teflon (0.2-μm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 5-mL **filtered** supernate

Background Subtracted Intensities

	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps
Water Blank	2.35	1.53	0.36	5.97	6.70	7.05
A	228.31	713.84	7.73	1345.48	729.79	227.10
B	224.78	715.37	7.91	1342.49	717.28	225.93
C	230.14	720.48	7.75	1345.21	729.07	225.67
D	224.44	713.26	7.87	1343.36	728.17	223.47
E	226.58	709.66	7.73	1338.60	728.07	225.98
F	228.09	717.91	7.95	1346.95	730.99	221.67
G	219.66	707.87	8.02	1337.66	724.79	224.14
H	223.15	713.09	8.00	1340.07	726.84	225.44
I	215.13	706.30	8.26	1336.63	729.60	226.97
J	223.35	710.05	8.07	1336.20	725.27	224.54
A	221.22	712.06	7.98	1338.18	729.03	224.89
B	215.55	702.64	8.12	1336.31	728.54	224.17
C	220.97	704.87	7.89	1337.58	726.95	226.08
D	213.27	698.91	7.83	1334.54	726.99	223.70
E	212.28	696.21	7.30	1336.40	726.35	225.85
F	217.27	707.02	7.81	1339.21	728.88	223.06
G	210.62	691.90	7.43	1330.60	726.53	223.37
H	211.47	698.83	7.75	1335.54	724.27	223.72
I	210.47	696.08	7.60	1331.03	720.71	229.36
J	212.37	696.44	7.40	1337.86	714.17	226.70
A	202.32	689.23	7.95	1334.52	706.14	226.34
B	197.75	678.16	8.07	1334.56	696.64	224.46
C	197.37	679.25	8.16	1330.42	688.78	224.98
D	197.59	669.79	7.94	1336.21	682.16	223.30
E	194.48	667.58	8.04	1333.45	674.70	223.42
F	194.62	667.29	7.57	1333.20	675.12	222.35
G	189.69	658.49	8.17	1331.25	663.52	222.45
H	199.91	673.79	7.74	1326.79	657.33	223.25
I	201.05	667.25	7.77	1331.88	653.06	221.51
J	202.43	669.14	7.69	1333.43	651.09	223.53
Average	212.2	695.1	7.9	1336.5	709.0	224.6
Stdev	11.9	18.5	0.2	4.8	26.9	1.8
%RSD	**5.6**	**2.7**	**3.0**	**0.4**	**3.8**	**0.8**

[1] This Table is from A. R. Jurgensen, D. M. Missimer, and R. L. Rutherford, "X-ray Fluorescence (XRF) Aanalysis of Hanford Low Activity Waste Simulants", WSRC-TR-2006-00137, SRNL-RPP-2006-00019.

Table C - 2. AN-105 Simulant Solution[1] Precision – Three Runs of Five Filtered Diluted Samples (1:1) in Autosampler.

Three consecutive runs with samples in same position on autosampler.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-μm (film was not replaced)
Cell: 31-mm double open end
Sample film Ultralene - 4-μm (same film for each run)
Cover film: Micro-porous Teflon (0.2-μm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

Background Subtracted Intensities

	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps	Mo cps	S cps
Water Blank	1.91	1.17	0.20	4.42	64.39	7.53	0.00	1.56
A	67.12	159.30	3.19	540.63	350.17	104.04	85.68	11.08
B	67.60	157.86	3.37	542.99	348.49	101.63	86.74	11.45
C	65.90	160.90	3.54	546.26	347.89	103.41	89.58	11.53
D	66.04	157.07	3.40	541.21	348.94	103.05	87.94	12.33
E	65.13	152.53	3.25	540.94	350.81	104.34	84.48	12.51
A	61.98	156.96	3.05	538.79	351.95	104.31	91.68	12.87
B	61.67	154.79	3.30	546.11	354.16	103.44	81.15	13.22
C	61.59	159.52	3.05	540.72	350.29	104.56	88.03	13.45
D	63.47	157.03	3.25	547.54	348.92	104.84	66.04	13.72
E	63.77	153.97	3.03	552.76	339.16	104.62	92.31	13.10
A	61.18	156.93	3.32	551.69	324.80	103.12	88.18	14.66
B	63.77	157.48	3.11	558.83	327.82	104.44	98.74	15.16
C	67.86	166.02	3.25	568.26	325.59	106.45	100.65	14.92
D	65.10	158.20	3.09	565.06	327.30	107.96	101.61	15.04
E	68.63	163.23	3.50	591.01	331.44	109.91	99.90	14.77
Average	64.7	158.1	3.2	551.5	341.8	104.7	89.5	13.3
Stdev	2.5	3.4	0.2	14.2	11.1	2.1	9.1	1.4
%RSD	**3.8**	**2.2**	**5.0**	**2.6**	**3.3**	**2.0**	**10.1**	**10.3**

[1] Simulant AN-105 was made using the full Eibling recipe and diluted to 5M sodium.

Table C - 3. AN-105 Simulant Solution[1] Precision – Fourteen Runs of One Filtered Diluted Sample (1:1) in Autosampler over 8 Hours.

One sample in same position on autosampler run 14 times over 8 hours.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was not replaced)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm (same film for each run)
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

Background Subtracted Intensities

	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps
	63.27	153.96	3.09	557.87	353.44	111.31
	65.26	155.18	3.33	548.84	348.34	107.34
	64.21	153.18	3.24	546.15	346.77	106.36
	62.39	152.87	3.10	540.24	348.76	103.06
	63.80	151.08	3.26	542.19	348.61	104.68
	61.30	153.08	3.34	545.71	352.47	103.74
	62.07	153.31	3.36	543.72	354.63	103.55
	62.06	153.29	3.56	550.08	357.41	105.63
	61.33	151.48	3.45	549.48	357.92	105.82
	58.90	150.92	3.13	546.09	357.46	106.40
	58.56	151.06	2.89	548.60	359.67	106.20
	57.37	149.82	3.36	550.67	357.58	108.49
	57.50	147.97	3.38	549.93	357.06	106.86
	54.57	147.74	2.76	548.69	361.43	106.65
Average	61.2	151.8	3.2	547.7	354.3	106.2
Stdev	3.2	2.3	0.2	4.5	5.0	2.1
%RSD	**5.2**	**1.5**	**7.2**	**0.8**	**1.4**	**2.0**

[1] Simulant AN-105 was made using the full Eibling recipe and diluted to 5M sodium.

Table C - 4. AN-105 Simulant Solution[1] Precision – One Filtered Diluted Sample (1:1) in Autosampler Run at Different Times During the Day.

One sample in same position on autosampler run at different times during the day.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-mm (film was not replaced)
Cell: 31-mm double open end
Sample film Ultralene - 4-mm (same film for each run)
Cover film: Micro-porous Teflon (0.2-mm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

Background Subtracted Intensities

Time	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps
intial	60.77	150.02	3.53	536.48	345.51	105.08
2-hrs	65.08	157.04	3.36	539.27	348.94	104.27
4-hrs	65.76	157.49	3.42	546.86	350.77	103.65
5-hrs	65.14	158.04	3.17	550.35	354.96	105.84
6-hrs	65.20	158.05	3.39	557.2	357.94	106.18
Average	64.4	156.1	3.4	546.0	351.6	105.0
Stdev	2.0	3.4	0.1	8.4	4.9	1.1
%RSD	**3.2**	**2.2**	**3.9**	**1.5**	**1.4**	**1.0**

* The instrument film and sample film were exposed to the x-ray beam for 2.5 hours.

[1] Simulant AN-105 was made using the full Eibling recipe and diluted to 5M sodium.

Table C - 5. AN-105 Simulant Solution[1] Precision – Three Runs of Five Filtered Diluted Samples (1:1) in Autosampler with the Instrument Film Replaced Prior to Each Run.

Three consecutive runs with samples in same position on autosampler

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced prior to each run)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm (same film for each run)
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

Background Subtracted Intensities

	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps
A	68.38	160.36	3.12	553.35	353.39	106.31
B	69.07	164.94	3.51	556.26	354.55	108.19
C	65.08	154.39	3.16	540.44	347.20	104.48
D	66.05	153.23	3.56	536.50	346.44	104.99
E	65.20	154.51	3.46	545.85	349.36	105.53
A	64.67	151.61	3.63	537.00	349.76	104.28
B	66.02	162.41	3.58	548.19	353.63	105.49
C	65.07	157.57	3.15	542.27	351.84	103.31
D	66.69	157.05	3.22	548.86	348.56	103.41
E	69.25	155.46	3.22	548.17	349.42	104.52
A	65.08	156.05	3.38	552.41	355.02	108.26
B	66.54	162.44	3.23	552.46	357.75	106.40
C	64.66	157.78	3.33	548.55	353.40	104.65
D	63.93	155.48	3.36	552.12	353.99	107.78
E	65.95	155.38	3.86	551.09	355.92	106.57
Average	66.1	157.2	3.4	547.6	352.0	105.6
Stdev	1.6	3.8	0.2	6.0	3.4	1.6
%RSD	**2.5**	**2.4**	**6.3**	**1.1**	**1.0**	**1.5**

[1] Simulant AN-105 was made using the full Eibling recipe and diluted to 5M sodium.

Table C - 6. AP-101 Simulant Solution[1] Precision – Three Runs of Five Filtered Diluted Samples (1:1) in Autosampler.

Three consecutive runs with samples in same position on autosampler

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was not replaced)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

Background Subtracted Intensities

	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps	Mo cps	S cps
Water Blank	0.00	7.32	0.00	3.91	60.84	6.74	0.00	2.34
A	56.21	90.36	12.28	173.88	2104.42	26.03	21.24	83.51
B	58.11	91.58	12.31	176.68	2115.30	26.65	15.25	85.01
C	58.28	94.45	12.36	174.40	2124.12	26.25	20.50	85.63
D	59.22	104.09	12.17	178.56	2129.52	26.51	19.98	93.66
E	57.65	97.66	11.64	176.01	2127.38	26.33	19.51	85.68
A	53.00	98.85	11.86	175.87	2109.87	26.51	23.37	84.57
B	59.14	112.88	12.28	182.55	2121.33	26.65	17.47	85.57
C	58.01	106.19	12.34	179.81	2129.18	26.41	18.43	85.86
D	56.65	116.64	12.04	183.32	2121.90	27.13	24.37	100.66
E	57.61	109.69	11.89	182.25	2123.97	26.28	23.11	88.07
A	56.25	92.93	11.92	176.48	2142.57	26.94	20.97	84.82
B	57.92	97.78	11.81	177.12	2155.36	27.18	22.25	85.29
C	62.95	101.79	12.46	181.44	2159.83	27.43	19.95	87.36
D	57.88	99.21	12.05	178.52	2156.70	27.69	18.67	87.92
E	60.19	101.23	12.44	179.87	2167.90	26.90	22.70	87.51
Average	57.9	101.0	12.1	178.5	2132.6	26.7	20.5	87.4
Stdev	2.2	7.7	0.3	3.0	19.3	0.5	2.5	4.4
%RSD	**3.7**	**7.7**	**2.1**	**1.7**	**0.9**	**1.8**	**12.0**	**5.0**

[1] Simulant AP-101 was made using the Full Eibling recipe

Table C - 7. AP-101 Simulant Solution[1] Precision – Three Runs of Five Filtered Acidic Samples in Autosampler.

Three consecutive runs with samples in same position on autosampler.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-μm (film was not replaced)
Cell: 31-mm double open end
Sample film Ultralene - 4-μm
Cover film: Micro-porous Teflon (0.2-μm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 2-mL DI water + 2-mL HNO_3

Background Subtracted Intensities

	Na cps	Al cps	P cps	Cl cps	K cps	Cr cps	Mo cps	S cps
Water Blank	0.00	3.83	0.20	5.44	62.21	7.54	0.00	20.34
A	48.42	85.89	11.20	162.08	1971.90	25.60	20.40	77.99
B	52.82	91.99	11.58	166.24	1993.70	25.73	18.79	78.74
C	54.23	90.91	11.50	165.12	2000.74	25.62	20.70	80.14
D	51.71	90.82	11.48	167.27	1993.44	25.13	21.99	81.11
E	53.30	91.38	11.36	166.72	1990.64	25.78	12.89	80.16
A	52.28	88.32	11.51	161.67	1980.79	24.82	17.38	78.78
B	52.83	88.19	10.83	163.24	1974.78	26.10	15.53	77.98
C	53.95	87.83	11.39	164.30	1982.02	24.82	23.10	79.27
D	51.43	88.73	11.08	164.61	1976.26	25.36	23.47	80.24
E	51.58	90.49	11.15	163.92	1982.03	25.12	20.12	79.80
A	50.86	87.94	11.48	163.94	1991.78	25.11	21.38	78.80
B	50.82	87.55	11.35	163.48	1989.87	25.75	22.77	79.52
C	50.64	87.83	11.64	164.05	1997.58	25.50	28.53	79.15
D	51.33	87.11	11.36	164.41	1993.81	25.28	15.01	79.38
E	52.47	90.45	11.20	165.46	2000.10	25.12	23.78	79.34
Average	51.9	89.0	11.3	164.4	1988.0	25.4	20.4	79.4
Stdev	1.5	1.8	0.2	1.6	9.3	0.4	4.0	0.9
%RSD	**2.8**	**2.0**	**1.9**	**1.0**	**0.5**	**1.5**	**19.6**	**1.1**

[1] Simulant AP-101 was made using the full Eibling recipe.

7.4 Appendix D: Representative Calibration Curves

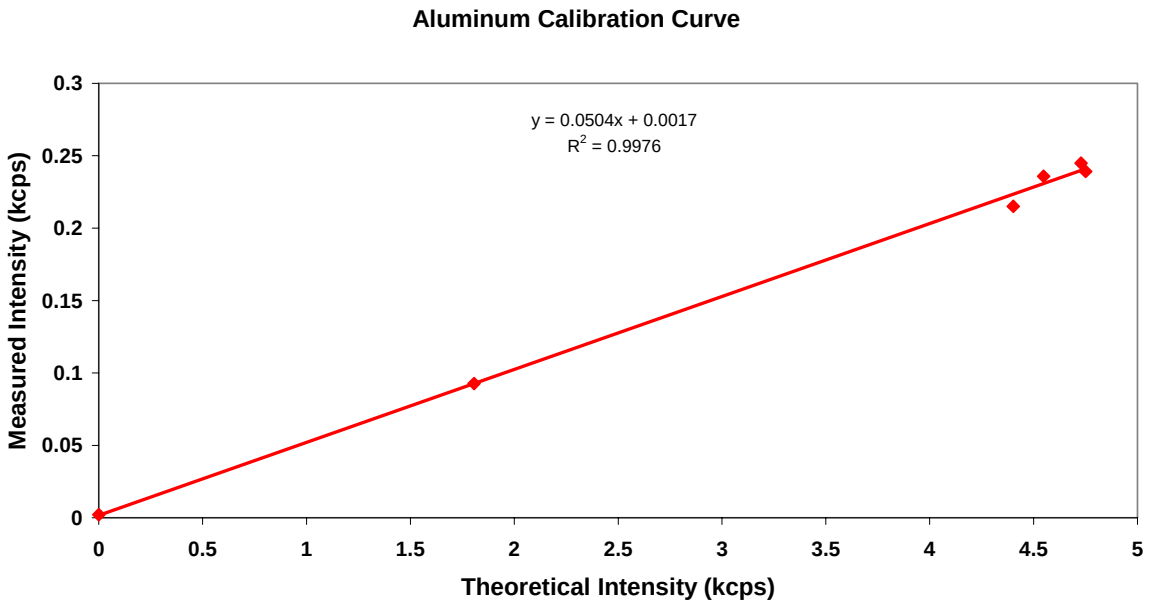

Figure D - 1. Aluminum calibration curve using all five High Purity standards.

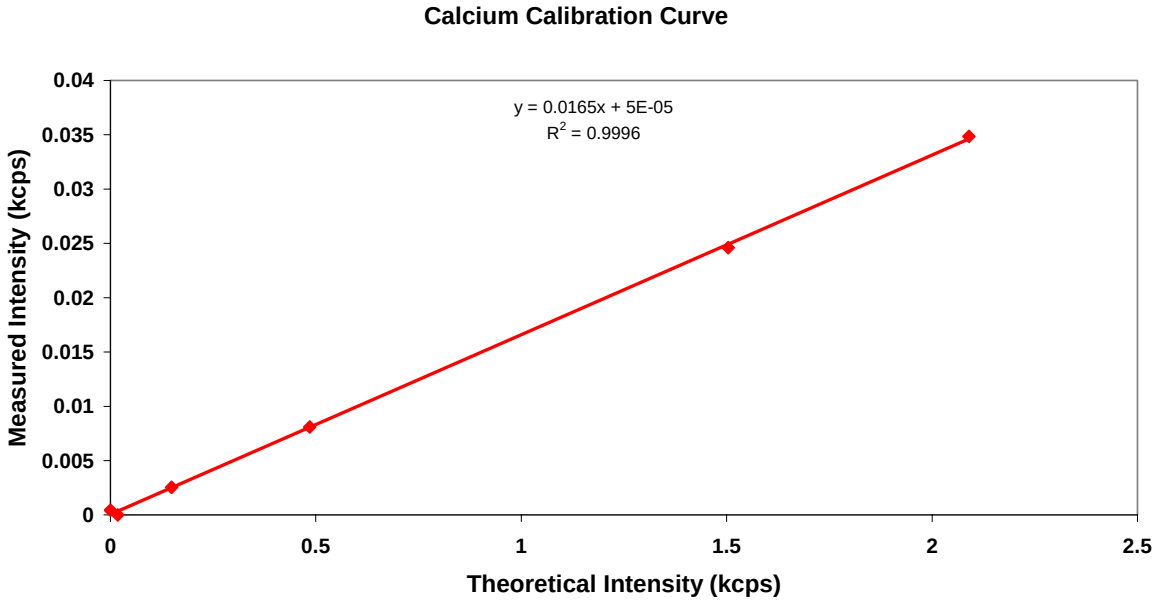

Figure D - 2. Calcium calibration curve using all five High Purity standards.

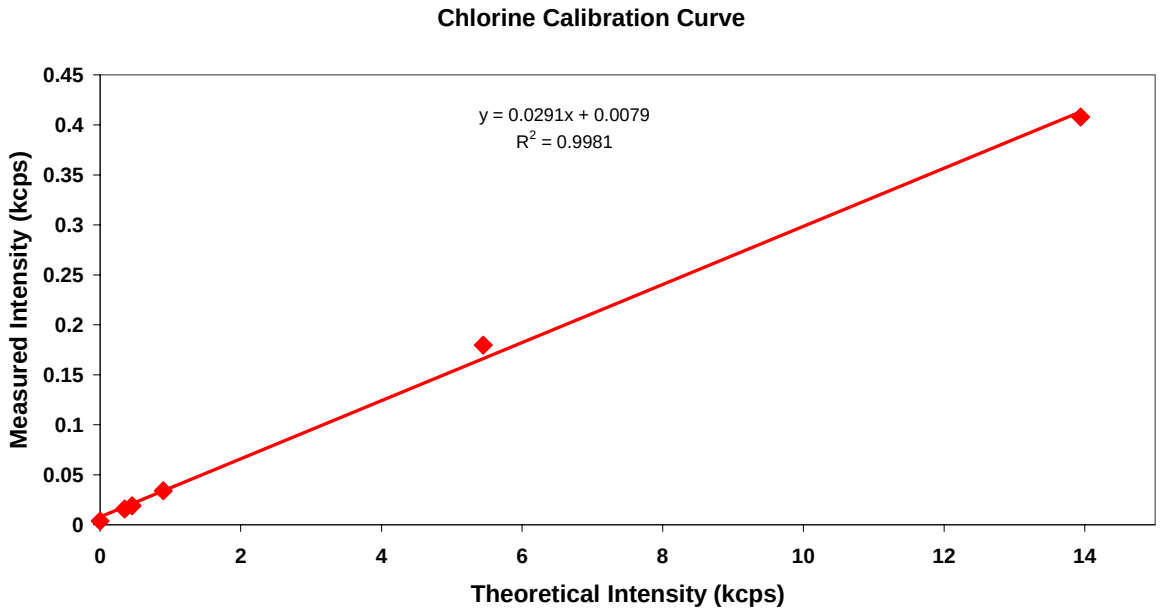

Figure D - 3. Chlorine calibration curve using all five High Purity standards.

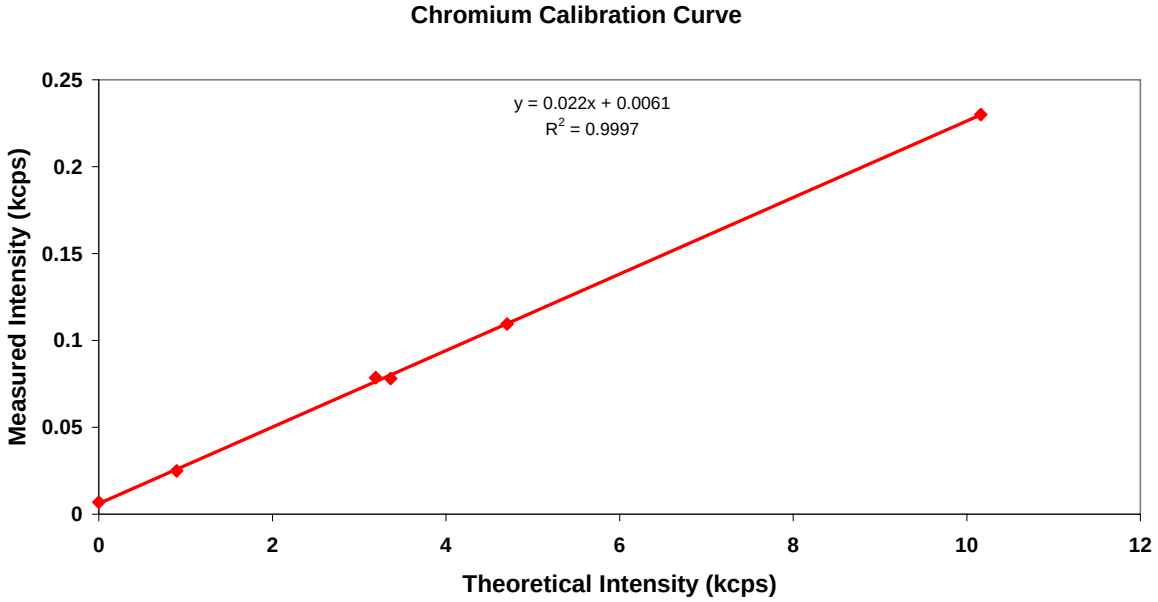

Figure D - 4. Chromium calibration curve using all five High Purity standards.

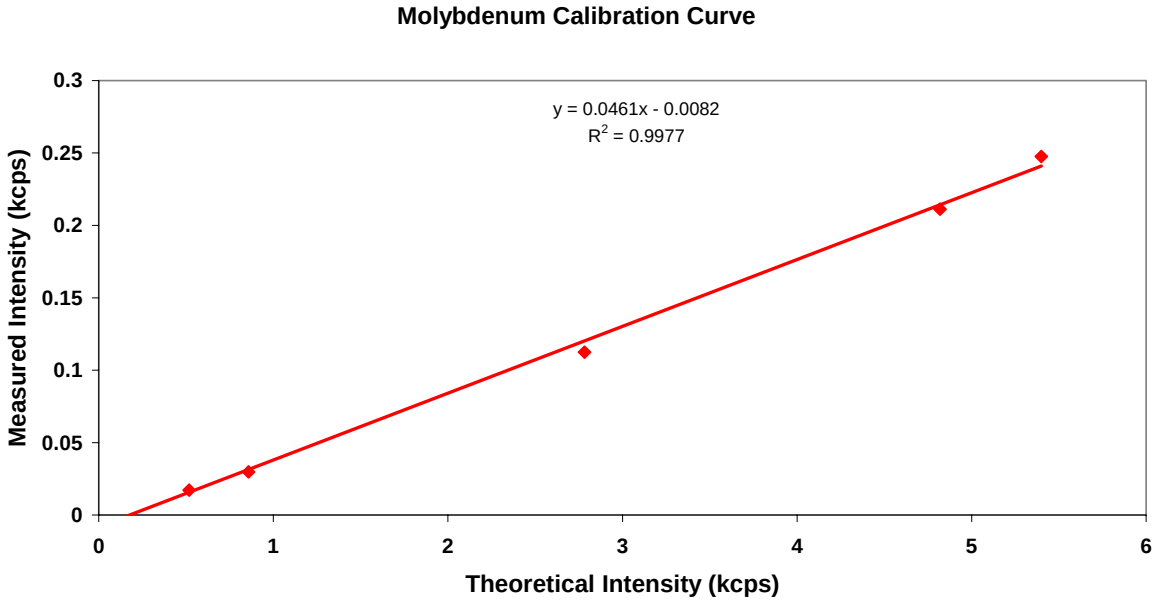

Figure D - 5. Molybdenum calibration curve using all five High Purity standards.

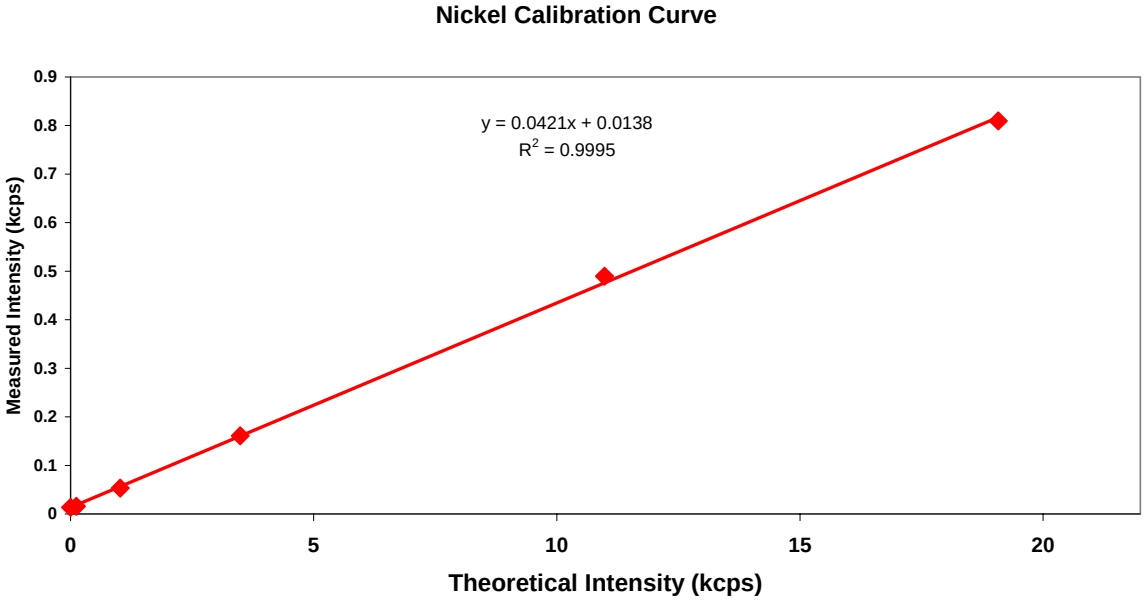

Figure D - 6. Nickel calibration curve using all five High Purity standards.

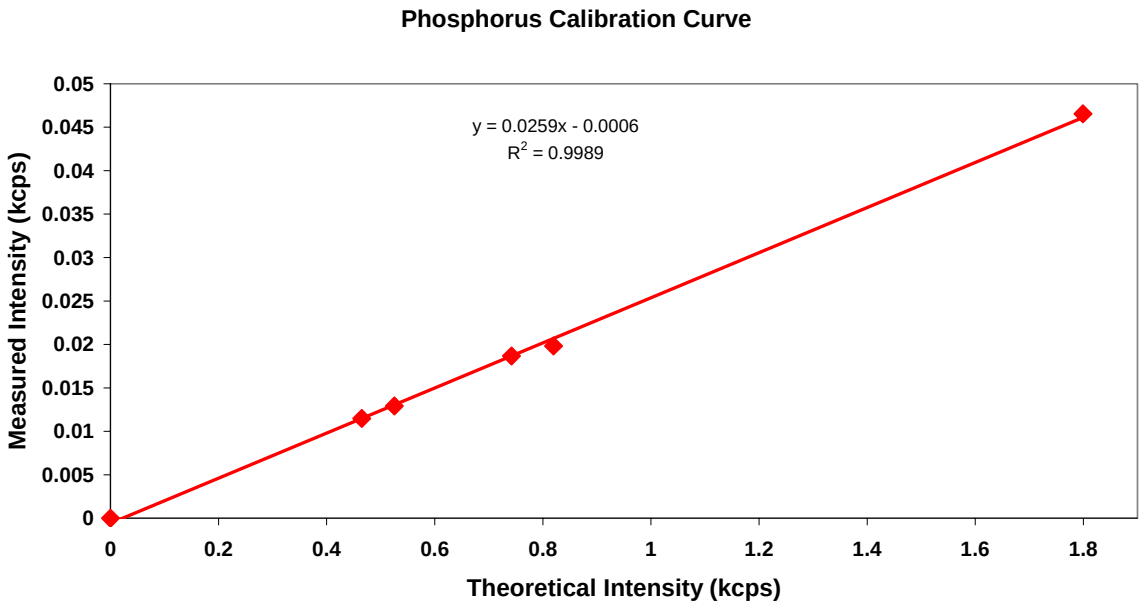

Figure D - 7. Phosphorus calibration curve using all five High Purity standards.

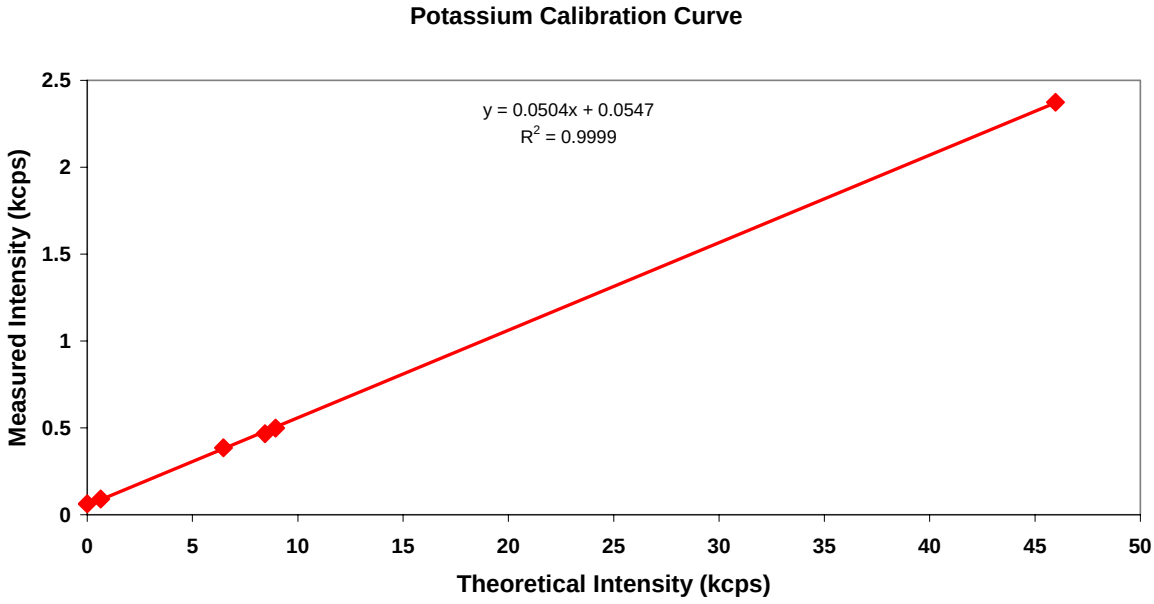

Figure D - 8. Potassium calibration curve using all five High Purity standards.

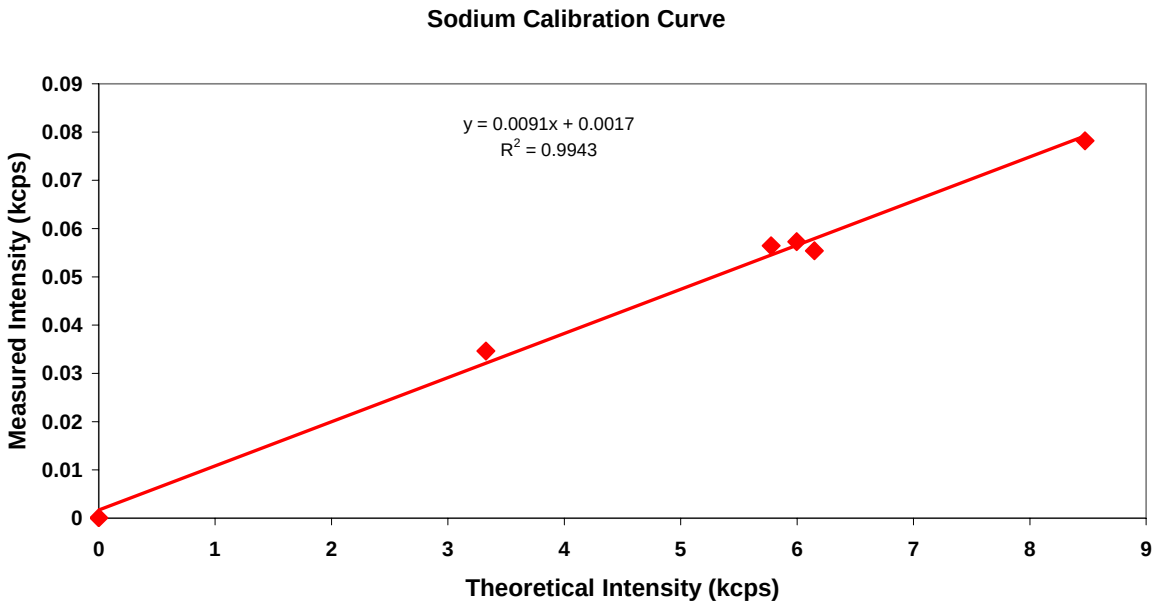

Figure D - 9. Sodium calibration curve using all five High Purity standards.

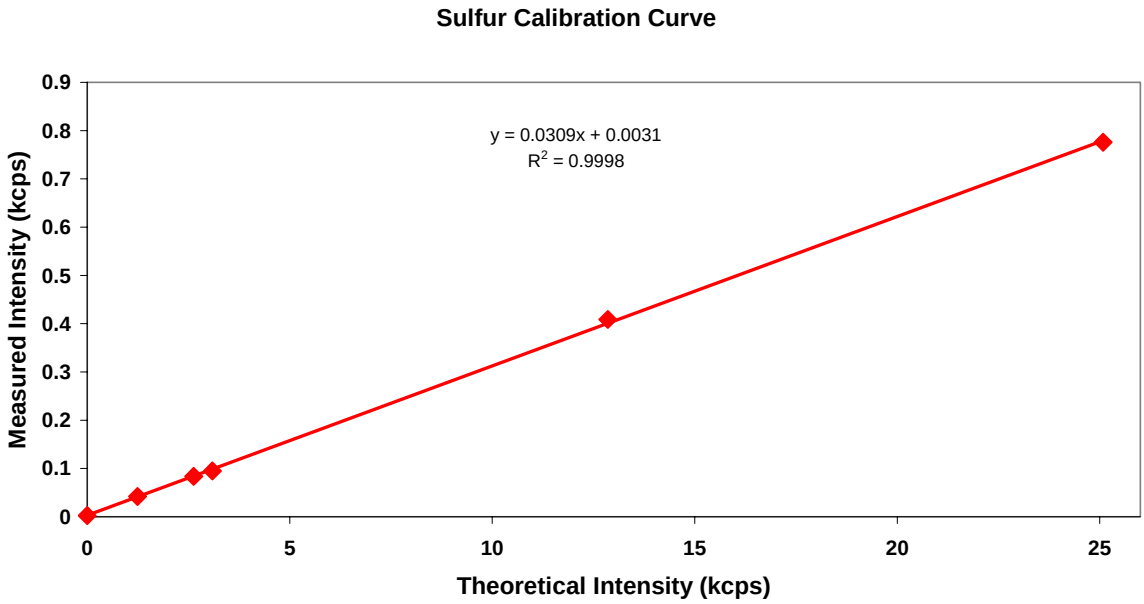

Figure D - 10. Sulfur calibration curve using all five High Purity standards.

7.5 Appendix E: WD-XRF Results

Table E - 1. WD-XRF Analysis of RPP Acidified Simulants.

Envelope A (AN-105 & AP-101 simulant), Envelope C (AN-107 simulant), and Envelope B/D (AZ-101 simulant) Analysis

The instrument was calibrated using five High Purity standards for each run.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced for calibration and analysis)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 2-mL DI water + 2-mL HNO_3

	Al µg/mL	Ca µg/mL	Cr µg/mL	Mo µg/mL	Ni µg/mL	K µg/mL	Na µg/mL	SO_4 µg/mL	PO_4 µg/mL	Cl µg/mL
AN-105	12212	<10	710	42	<2	4070	148748	557	362	5264
AN-105	11100	<10	678	38	<2	3824	124132	389	368	4934
AN-105	11448	<10	678	42	<2	3866	128410	330	374	4858
AN-105	11432	<10	684	40	<2	3908	135206	318	368	4978
AN-105	11020	<10	682	42	<2	3884	125158	389	356	4948
AN-105	11196	<10	692	38	<2	3860	126640	383	356	4978
AN-105	11222	<10	674	42	<2	3842	129978	372	319	4988
AN-105	11542	<10	690	42	<2	3868	123876	360	374	4922
AN-105	11514	<10	678	42	<2	3824	125558	395	356	5000
AN-105	11216	<10	678	44	<2	3818	125028	395	331	4852
AN-105	11378	<10	680	42	<2	3942	129452	479	392	4992
Average	11390		684	41		3876	129273	389	356	4972
STDEV	339		11	2		74	7652	65	18	115
%RSD	3		2	5		2	6	17	5	2
AP-101	7384	<10	160	12	<2	28204	131148	3835	1318	1592
AP-101	6968	<10	158	14	<2	27878	118704	3637	1288	1570
AP-101	6968	<10	154	14	<2	27734	121234	3541	1282	1526
AP-101	6834	<10	152	14	<2	27682	116576	3643	1245	1532
AP-101	6988	<10	154	12	<2	28140	122696	3667	1294	1588
AP-101	7018	<10	156	14	<2	28158	122508	3661	1263	1586
AP-101	7186	<10	148	12	<2	28108	123634	3649	1226	1568
AP-101	7208	<10	158	16	<2	28630	124344	3757	1355	1588
AP-101	6842	<10	156	16	<2	28094	117380	3709	1306	1564
AP-101	7200	<10	158	16	<2	28228	127208	3757	1331	1562
AP-101	7130	<10	148	12	<2	28382	125608	3739	1368	1596
Average	7060		155	14		28086	122543	3686	1291	1568
STDEV	178		4	2		273	4459	82	39	23
%RSD	3		2	12		1	4	2	3	1
AN-107	340	154	<5	24	294	1056	141956	4830	859	1278
AN-107	384	160	<5	20	308	1210	160290	5087	914	1354
AN-107	250	148	<5	18	306	1178	142918	4872	908	1348
AN-107	288	144	<5	22	304	1218	143358	4937	987	1356
AN-107	218	138	<5	22	304	1172	144806	4997	957	1362
AN-107	172	142	<5	22	308	1216	143372	4967	938	1362
AN-107	370	152	<5	22	308	1172	143570	4967	914	1372
AN-107	252	150	<5	18	304	1192	138156	5003	987	1354
AN-107	268	148	<5	22	306	1186	146320	4973	963	1370
AN-107	270	132	<5	20	310	1158	146298	5021	920	1342
AN-107	228	154	<5	22	306	1186	149464	5111	963	1370
Average	281	147		21	305	1176	145104	4966	935	1350
STDEV	67	8		2	4	47	5821	73	40	27
%RSD	24	6		9	1	4	4	1	4	2
AZ-101	<80	<10	140	24	<2	766	11158	875	<30	262
AZ-101	<80	<10	154	26	<2	968	9434	917	<30	296
AZ-101	<80	<10	162	24	<2	910	10088	791	<30	268
AZ-101	<80	<10	150	26	<2	918	9872	809	<30	284
AZ-101	<80	<10	142	26	<2	918	8870	749	<30	270
AZ-101	<80	<10	150	26	<2	954	10726	809	<30	300
AZ-101	<80	<10	150	24	<2	920	8754	827	<30	308
AZ-101	<80	<10	148	26	<2	908	10044	839	<30	284
AZ-101	<80	<10	152	26	<2	904	9490	827	<30	274
AZ-101	<80	<10	150	24	<2	858	9382	851	<30	264
AZ-101	<80	<10	148	26	<2	932	9368	845	<30	306
Average			150	25		902	9782	829		281
STDEV			6	1		56	761	46		16
%RSD			4	4		6	8	6		6
Blank	66	5	0	0	1	24	0	29	1	0
Blank	90	7	6	0	0	41	0	27	3	0
Blank	87	3	1	0	0	59	**2352**	2	9	0
Blank	2	0	0	2	0	69	0	0	8	0
Blank	0	0	0	1	0	73	0	0	12	0
Blank	45	6	0	0	1	53	0	0	4	0
Blank	18	6	2	0	0	61	0	3	12	0
Blank	1	4	0	0	0	74	0	0	8	0
Blank	11	2	2	1	1	55	0	7	4	0
Blank	83	7	0	0	1	74	0	11	7	0

Table E - 2. WD-XRF Analysis of RPP Basic Simulants.

Envelope A (AN-105 & AP-101 simulant), Envelope C (AN-107 simulant), and Envelope B/D (AZ-101 simulant) Analysis

The instrument was calibrated using five High Purity standards for each run.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced for calibration and analysis)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

	Al µg/mL	Ca µg/mL	Cr µg/mL	Mo µg/mL	Ni µg/mL	K µg/mL	Na µg/mL	SO_4 µg/mL	PO_4 µg/mL	Cl µg/mL
AN-105	12056	<10	732	42	<2	4154	139278	419	386	5530
AN-105	11912	<10	724	42	<2	4124	138900	539	350	5348
AN-105	12668	<10	758	42	<2	4344	148654	641	411	5686
AN-105	11672	<10	746	42	<2	4234	138102	413	386	5476
AN-105	13594	<10	764	46	<2	4324	146582	395	411	5498
Average	12380		745	43		4236	142303	482	389	5508
STDEV	772		17	2		98	4925	106	25	121
%RSD	6		2	4		2	3	22	6	2
AP-101	7634	<10	172	12	<2	30872	138186	4045	1410	1754
AP-101	7518	<10	162	14	<2	30636	135542	4075	1380	1716
AP-101	7518	<10	180	14	<2	30204	130056	4158	1380	1760
AP-101	7274	<10	176	16	<2	30478	125096	3859	1325	1698
AP-101	7684	<10	168	18	<2	31016	139492	4039	1472	1742
Average	7526		172	15		30641	133674	4035	1393	1734
STDEV	158		7	2		321	6008	109	54	26
%RSD	2		4	15		1	4	3	4	2
AN-107	370	166	<5	18	334	1290	156644	5513	1067	1540
AN-107	472	172	<5	22	338	1306	168762	5632	1079	1548
AN-107	406	170	<5	22	338	1370	165494	5567	1055	1548
AN-107	258	170	<5	20	332	1304	151164	5405	987	1496
AN-107	224	144	<5	24	332	1342	157294	5423	1055	1508
Average	346	164		21	335	1322	159872	5508	1049	1528
STDEV	103	12		2	3	33	7133	96	36	24
%RSD	29.9	7.1		10.8	0.9	2.5	4.5	1.7	3.4	1.6
AZ-101	<80	<10	162	26	<2	1014	8678	869	<30	310
AZ-101	<80	<10	164	26	<2	980	16346	1067	<30	318
AZ-101	<80	<10	172	26	<2	1116	11308	1103	<30	316
AZ-101	<80	<10	176	26	<2	1030	11794	887	<30	302
AZ-101	<80	<10	170	30	<2	1042	13484	935	<30	312
Average			169	27		1036	12322	972		312
STDEV			6	2		50	2834	106		6
%RSD			3	7		5	23	11		2
Blank	51	6	6	0	1	65	0	75	0	0
Blank	66	5	0	0	1	24	0	29	1	0
Blank	90	7	6	0	0	41	0	27	3	0
Blank	87	3	1	0	0	59	**2352**	2	9	0
Blank	2	0	0	2	0	69	0	0	8	0

Table E - 3. WD-XRF Analysis of RPP Acidified Simulants using Drift Correction.

Envelope A (AN-105 & AP-101 simulant), Envelope C (AN-107 simulant), and Envelope B/D (AZ-101 simulant) Analysis

The instrument was calibrated one time using four High Purity standards[1] and then daily using a High Purity standard as a drift monitor[2]

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced for calibration and analysis)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 2-ml DI water + 2-ml HNO_3

	Al µg/mL	Ca µg/mL	Cr µg/mL	Mo µg/mL	Ni µg/mL	K µg/mL	Na µg/mL	SO_4 µg/mL	PO_4 µg/mL	Cl µg/mL
AN-105	12780	<10	676	40	<2	3936	135294	425	331	5060
AN-105	11704	<10	676	38	<2	3970	137024	461	258	5376
AN-105	11774	<10	704	40	<2	3944	134608	443	337	5546
AN-105	12140	<10	704	38	<2	4016	144198	467	307	5464
AN-105	11678	<10	688	42	<2	3952	134026	461	300	5366
Average	12015		690	40		3964	137030	452	307	5362
STDEV	466		14	2		32	4162	17	32	184
%RSD	4		2	4		1	3	4	10	3
AP-101	7372	<10	152	12	<2	28612	128112	3721	1282	1594
AP-101	7704	<10	156	12	<2	28974	135590	3835	1380	1752
AP-101	7198	<10	158	14	<2	28024	120392	3781	1318	1724
AP-101	7710	<10	158	14	<2	29192	138208	3925	1417	1750
AP-101	7384	<10	162	14	<2	28460	127822	3829	1300	1724
Average	7474		157	13		28652	130025	3818	1339	1709
STDEV	225		4	1		455	7058	75	57	66
%RSD	3		2	8		2	5	2	4	4
AN-107	374	158	<5	22	308	1200	146588	4997	920	1386
AN-107	474	170	<5	16	312	1198	157870	5207	987	1490
AN-107	404	142	<5	20	306	1192	145062	3236	944	1516
AN-107	398	146	<5	18	298	1212	159768	5249	1006	1496
AN-107	418	160	<5	20	310	1184	148280	5081	920	1474
Average	414	155		19	307	1197	151514	4754	955	1472
STDEV	37	11		2	5	10	6799	855	39	51
%RSD	9	7		12	2	1	4	18	4	3
AZ-101	<80	<10	150	24	<2	922	12114	875	<30	280
AZ-101	<80	<10	154	24	<2	910	12590	893	<30	326
AZ-101	<80	<10	158	24	<2	876	12092	917	<30	332
AZ-101	<80	<10	164	24	<2	952	14848	887	<30	308
AZ-101	<80	<10	158	24	<2	896	14300	929	<30	324
Average			157	24		911	13189	900		314
STDEV			5	0		29	1295	22		21
%RSD			3	0		3	10	2		7
Standard A[3]	9316	512	261	4	392	259	39963	938	2573	39
Standard A[3]	9692	538	264	4	392	259	43063	977	2631	47
Standard A[3]	9781	520	268	3	397	239	44069	965	2692	45
Standard A[3]	9718	526	267	4	399	262	44116	986	2680	43
Standard A[3]	9749	519	268	5	395	254	43616	983	2628	47
Average	9651	523	266	4	395	255	42965	970	2641	44
STDEV	190	10	3	1	3	9	1731	19	48	3
%RSD	2	2	1	18	1	4	4	2	2	8
Blank	80	16	1	0	2	70	0	4	0	0
Blank	63	7	1	0	2	59	0	6	2	0
Blank	77	0	0	0	1	66	0	3	0	0
Blank	122	5	2	0	1	61	0	13	0	0
Blank	69	9	2	0	0	51	0	24	0	0

[1]High Purity standards B, C, D, and E were used to calibrate the instrument.
[2]High Purity standard D was used as the drift monitor.
[3]High Purity standard A was used as the check standard.

Table E - 4. Quality Control Check Standards Analysis for the Basic and Acidified Runs.

The instrument was calibrated using five High Purity standards for each run.

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced for calibration and analysis)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 8-mL of Standard

	Al µg/mL	Ca µg/mL	Cr µg/mL	Mo µg/mL	Ni µg/mL	K µg/mL	Na µg/mL	SO_4 µg/mL	PO_4 µg/mL	Cl µg/mL
Standard A	9315	523	262	5	383	258	40808	929	2487	37
C of A	8850	500	250	5	375	225	36500	900	2450	50
Standard B	3693	<10	80	11	5	16905	67020	1968	638	859
C of A	3650	5	85	10	5	17000	64500	1950	650	800
Standard C	9280	130	386	48	127	2352	60590	9386	1021	133
C of A	9500	125	400	50	130	2400	62000	9650	1050	135
Standard D	9221	378	892	61	735	3333	62353	18704	714	57
C of A	9600	400	900	60	750	3400	65800	19000	750	70
Standard E	8980	39	294	30	37	3128	89164	2361	1174	2107
C of A	9150	40	300	30	40	3250	89000	2400	1200	2150
Standard C	9229	115	396	51	130	2343	58949	9629	1039	122
C of A	9500	125	400	50	130	2400	62000	9650	1050	135
Standard A	9083	512	256	4	385	235	38580	929	2422	42
C of A	8850	500	250	5	375	225	36500	900	2450	50
Standard B	3589	<10	89	12	3	16914	63300	1932	644	855
C of A	3650	5	85	10	5	17000	64500	1950	650	800
Standard C	9732	121	402	50	130	2455	63370	9869	1085	135
C of A	9500	125	400	50	130	2400	62000	9650	1050	135
Standard D	9830	388	905	62	758	3408	68631	19225	791	52
C of A	9600	400	900	60	750	3400	65800	19000	750	70
Standard E	8892	44	291	29	36	3124	90938	2331	1107	2122
C of A	9150	40	300	30	40	3250	89000	2400	1200	2150
Standard B	3804	<10	86	10	4	17214	67835	2004	696	669
C of A	3650	5	85	10	5	17000	64500	1950	650	800

C of A - Certificate of Analysis

7.6 Appendix F: IC and ICP Simulant and Standard Data

Table F - 1. IC Data for Envelope A, AN-105 Simulant.

	Cl mg/L	SO_4 mg/L
AN-105	4500	148
AN-105	4610	119
AN-105	4700	137
AN-105	4540	152
AN-105	4660	146
Average	4600	140
STDEV	83	13
%RSD	2	9

Table F - 2. ICP Data for Envelope A, AN-105 Simulant.

	Ag mg/L	Al mg/L	B mg/L	Ca mg/L	Cd mg/L	Cr mg/L	Fe mg/L	K mg/L
AN-105	3.25	10900	23.9	<3.81	2.02	645	1.19	3850
AN-105	2.94	10800	28.4	<3.81	2.61	663	0.714	3710
AN-105	2.83	10900	25.9	<1.29	2.42	664	1.00	3720
AN-105	3.90	10900	26.4	<1.29	2.57	656	1.04	3780
AN-105	3.53	10900	26.6	<1.29	2.48	668	0.935	3980
AN-105	3.52	11000	24.6	<1.29	2.21	666	1.63	3970
AN-105	3.24	9780	21.7	<1.29	1.83	573	1.05	3480
AN-105	2.58	9560	22.2	<1.29	1.73	580	0.503	3450
AN-105	3.43	10700	24.6	<1.29	2.37	644	0.755	3860
AN-105	3.07	10900	15.3	<1.29	2.24	636	0.731	3870
Average	3.23	10600	24.0		2.25	640	0.95	3770
STDEV	0.4	517	4		0.3	35	0.3	183
%RSD	12	5	15		14	5	33	5

	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	Pb mg/L	SO_4 mg/L	Si mg/L	Zn mg/L
AN-105	39.2	128000	1.16	280	15.2	422	84.3	5.59
AN-105	41.8	120000	<0.558	313	17.1	416	88.0	5.71
AN-105	41.3	124000	<0.349	298	16.8	407	84.5	5.48
AN-105	40.8	123000	<0.349	296	17.6	398	82.1	5.42
AN-105	41.6	127000	<0.349	297	18.2	407	81.9	5.77
AN-105	39.7	129000	0.592	291	15.1	401	84.2	5.31
AN-105	35.4	113000	0.392	261	13.6	357	71.1	4.70
AN-105	35.9	110000	<0.349	270	16.4	357	81.2	5.15
AN-105	40.0	124000	<0.349	298	17.1	407	83.1	5.01
AN-105	41.9	126000	<0.349	284	16.8	410	82.2	4.76
Average	39.8	122000		289	16.4	398	82.3	5.29
STDEV	2	6346		15	1	23	4	0.4
%RSD	6	5		5	8	6	5	7

Table F - 3. IC Data for Envelope A, AP-101 Simulant.

	Cl mg/L	SO_4 mg/L
AP-101	1460	3460
AP-101	1490	3390
AP-101	1500	3440
AP-101	1510	3460
AP-101	1510	3480
Average	1490	3450
STDEV	21	34
%RSD	1	1

Table F - 4. ICP Data for Envelope A, AP-101 Simulant.

	Al mg/L	B mg/L	Ba mg/L	Ca mg/L	Cd mg/L	Cr mg/L	Fe mg/L	K mg/L
AP-101	6750	11.7	0.532	<3.81	0.975	148	2.36	27600
AP-101	6730	13.8	0.675	<3.81	1.44	146	1.84	27200
AP-101	6820	12.5	0.562	<1.29	1.31	154	2.35	27300
AP-101	6860	12.5	0.575	<1.29	1.22	149	2.50	27900
AP-101	6910	13.3	0.584	<1.29	1.39	159	2.44	28900
AP-101	6910	12.7	0.575	<1.29	1.19	156	2.63	29000
AP-101	7050	12.8	0.610	<1.29	1.18	154	2.43	29000
AP-101	6880	12.5	0.721	<1.29	1.51	154	1.60	28400
AP-101	6730	11.9	0.638	<1.29	1.26	147	1.88	28100
AP-101	6830	2.46	0.581	<1.29	1.30	148	1.83	28300
Average	6850	11.6	0.605		1.28	152	2.19	28200
STDEV	99	3	0.1		0.2	4	0.4	673
%RSD	1	28	9		12	3	16	2

	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	Pb mg/L	SO_4 mg/L	Si mg/L	Zn mg/L
AP-101	13.3	124000	1.10	1254	7.12	3835	54.7	5.08
AP-101	13.0	113000	<0.558	1141	7.52	3745	54.6	4.91
AP-101	13.8	117000	<0.349	1220	10.1	3685	55.2	5.04
AP-101	13.3	120000	0.396	1153	7.39	3805	52.2	4.87
AP-101	14.5	121000	<0.349	1251	10.6	3835	54.3	5.40
AP-101	13.3	123000	<0.349	1233	8.52	3775	55.7	4.89
AP-101	13.6	123000	<0.349	1214	8.46	3955	54.5	4.97
AP-101	13.7	119000	<0.349	1239	9.85	3805	56.7	5.47
AP-101	12.7	119000	<0.349	1196	9.05	3715	53.3	4.39
AP-101	14.6	118000	<0.349	1177	9.64	3955	54.6	4.37
Average	13.6	120000		1210	8.83	3810	54.6	4.94
STDEV	0.6	3302		40	1	90	1	0.4
%RSD	4	3		3	14	2	2	7

Table F - 5. IC Data for Envelope C, AN-107 Simulant.

	Cl mg/L	SO_4 mg/L
AN-107	1390	4720
AN-107	1390	4630
AN-107	1380	4770
AN-107	1690	4760
AN-107	1650	4680
Average	1500	4710
STDEV	156	58
%RSD	10	1

Table F - 6. ICP Data for Envelope C, AN-107 Simulant.

	Al mg/L	B mg/L	Ca mg/L	Cr mg/L	Cu mg/L	Fe mg/L	K mg/L	La mg/L	Mn mg/L
AN-107	235	18.9	147	0.326	9.89	13.0	1190	1.04	0.298
AN-107	232	21.9	145	0.518	11.5	12.4	1130	0.86	0.205
AN-107	233	19.7	140	0.711	11.2	12.6	1200	0.697	<0.515
AN-107	238	19.8	142	0.605	11.3	12.7	1180	1.23	<0.515
AN-107	237	20.2	147	0.633	12.1	12.6	1250	1.47	<0.515
AN-107	237	19.2	147	0.676	11.7	13.1	1220	1.03	<0.515
AN-107	241	20.0	150	1.19	12.3	15.4	1300	1.12	0.589
AN-107	241	19.6	147	0.659	11.5	12.1	1270	1.31	<0.515
AN-107	233	19.0	142	0.618	11.0	12.1	1180	1.04	<0.515
AN-107	236	9.81	145	0.498	10.2	12.5	1070	1.19	<0.515
Average	236	18.8	145	0.643	11.3	12.9	1200	1.10	
STDEV	3	3	3	0.2	1	1	67	0	
%RSD	1	17	2	35	7	7	6	20	

	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L	Si mg/L	Sr mg/L	Zn mg/L	Zr mg/L
AN-107	21.4	147000	290	905	5273	7.95	80.1	18.0	0.625
AN-107	22.1	130000	288	862	5033	10.5	73.9	17.5	0.829
AN-107	21.5	137000	301	874	5003	7.45	72.3	17.5	<35.0
AN-107	22.1	142000	302	883	5123	13.3	74.2	17.8	<35.0
AN-107	21.9	144000	309	895	5273	6.88	77.1	17.9	<35.0
AN-107	21.2	144000	307	892	5243	8.87	77.8	18.3	<35.0
AN-107	22.2	145000	314	905	5393	10.1	77.1	18.4	<35.0
AN-107	21.4	140000	303	908	5123	9.62	69.3	18.3	<35.0
AN-107	21.0	141000	294	905	5213	9.08	69.9	17.1	<35.0
AN-107	23.4	141000	306	917	5363	10.7	78.3	17.5	<35.0
Average	21.8	141000	301	894	5200	9.45	75.0	17.8	
STDEV	0.7	4818	8	17	131	2	4	0.4	
%RSD	3	3	3	2	3	20	5	2	

Table F - 7. IC Data for Envelope B/D, AZ-101 Simulant.

	Cl mg/L	SO_4 mg/L
AZ-101	248	743
AZ-101	282	685
AZ-101	263	826
AZ-101	254	740
AZ-101	247	744
Average	259	748
STDEV	14	50
%RSD	6	7

Table F - 8. ICP Data for Envelope B/D, AZ-101 Simulant.

	Al mg/L	Ca mg/L	Cr mg/L	K mg/L	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L
AZ-101	<2.56	<3.81	140	759	25.9	11500	<0.558	<80	767
AZ-101	<2.56	<3.81	139	762	25.0	11200	<0.558	<80	797
AZ-101	<4.70	<1.29	142	761	25.1	11300	0.36	<60	782
AZ-101	<4.70	<1.29	141	760	25.7	11300	<0.349	<60	800
AZ-101	<4.70	<1.29	144	805	26.0	11600	<0.349	<60	809
AZ-101	<4.70	<1.29	145	820	25.0	11800	0.642	<60	797
AZ-101	<4.70	<1.29	148	823	26.3	12000	<0.349	<60	836
AZ-101	<4.70	<1.29	141	779	24.6	11400	<0.349	<60	785
AZ-101	<4.70	<1.29	140	780	24.7	11400	<0.349	<60	812
AZ-101	<4.70	<1.29	142	765	26.7	11600	<0.349	<60	830
Average			142	781	25.5	11500			800
STDEV			3	25	0.7	247			21
%RSD			2	3	3	2			3

Table F - 9. IC Data for High Purity Standard A.

	Cl mg/L	SO_4 mg/L
Standard A	<100	3850
Standard A	<100	3540
Standard A	<100	3460
Standard A	<100	3420
Standard A	<100	3540
C of A	50	900
Average		3560
STDEV		169
%RSD		5

C of A - Certificate of Analysis

Table F - 10. ICP Data for High Purity Standard A.

	Al mg/L	Ca mg/L	Cr mg/L	K mg/L	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L
Standard A	8860	493	252	229	5.39	37400	372	2401	965
Standard A	8580	489	245	215	5.31	35300	359	2220	926
Standard A	8770	489	255	225	5.85	37100	381	2315	938
Standard A	8710	486	251	229	5.41	36700	372	2263	956
Standard A	8760	502	257	231	5.92	37300	379	2343	971
Standard A	8760	505	258	237	5.52	37600	382	2376	938
Standard A	8670	502	254	237	5.60	36600	380	2315	962
Standard A	8680	500	253	238	5.09	36800	374	2318	941
Standard A	8560	485	249	226	4.94	36700	363	2340	953
Standard A	8690	497	254	229	5.96	36300	381	2297	971
C of A	8850	500	250	225	5	36500	375	2450	900
Average	8700	495	253	230	5.50	36800	374	2320	952
Stdev	90	7	4	7	0.3	658	8	52	16
%RSD	1	1	2	3	6	2	2	2	2

C of A - Certificate of Analysis

Table F - 11. IC Data for High Purity Standard B.

	Cl mg/L	SO_4 mg/L
Standard B	854	7800
Standard B	883	7380
Standard B	856	6810
Standard B	824	6970
Standard B	824	6860
C of A	800	1950
Average	848	7160
STDEV	25	420
%RSD	3	6

C of A - Certificate of Analysis

Table F - 12. ICP Data for High Purity Standard B.

	Al mg/L	Ca mg/L	Cr mg/L	K mg/L	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L
Standard B	3650	4.72	85.6	17500	10.5	65700	5.91	619	2085
Standard B	3580	5.01	82.5	16700	10.0	62800	4.67	586	1980
Standard B	3430	3.46	83.6	15800	10.3	63200	4.83	626	1983
Standard B	3600	4.79	82.7	17000	9.99	66300	4.94	592	1983
Standard B	3550	5.00	84.6	17200	10.4	64900	4.70	626	2031
Standard B	3620	5.25	86.0	17700	10.2	66800	4.93	635	2040
Standard B	3540	5.86	83.9	16800	9.82	64800	5.16	607	2025
Standard B	3600	4.59	85.2	17300	9.86	66000	4.49	626	2037
Standard B	3470	4.43	82.3	16800	9.26	63700	4.61	610	2007
Standard B	3530	5.14	85.1	16700	10.9	64100	4.31	622	2124
C of A	3650	5	85	17000	10	64500	5	650	1950
Average	3560	4.83	84.2	17000	10.1	64800	4.86	615	2030
STDEV	68	0.6	1	532	0.4	1365	0.4	16	46
%RSD	2	13	2	3	4	2	9	3	2

C of A - Certificate of Analysis

Table F - 13. IC Data for High Purity Standard C.

	Cl mg/L	SO_4 mg/L
Standard C	181	26400
Standard C	195	26100
Standard C	183	24200
Standard C	173	22000
Standard C	177	25100
C of A	135	9650
Average	182	24800
STDEV	8	1770
%RSD	5	7

C of A - Certificate of Analysis

Table F - 14. ICP Data for High Purity Standard C.

	Al mg/L	Ca mg/L	Cr mg/L	K mg/L	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L
Standard C	9570	128	404	2500	50.5	63200	127	1033	10306
Standard C	9290	126	390	2360	50.0	59500	124	951	9947
Standard C	9300	119	388	2400	49.0	61200	126	966	9767
Standard C	9160	121	388	2460	48.9	61800	127	944	9887
Standard C	9400	128	404	2550	51.4	64000	132	1024	10097
Standard C	9390	126	398	2370	49.7	64000	130	1024	10097
Standard C	9240	125	390	2410	49.7	62600	129	984	9977
Standard C	9160	121	379	2490	47.2	62600	123	954	9587
Standard C	9090	120	380	2450	48.3	61200	119	975	9947
Standard C	9100	123	387	2430	51.9	61500	126	972	10156
C of A	9500	125	400	2400	50	62000	130	1050	9650
Average	9270	124	391	2440	49.7	62200	126	983	9980
STDEV	152	3	9	60	1	1403	4	33	204
%RSD	2	3	2	2	3	2	3	3	2

C of A - Certificate of Analysis

Table F - 15. IC Data for High Purity Standard D.

	Cl mg/L	SO_4 mg/L
Standard D	<100	45000
Standard D	199	40600
Standard D	116	41400
Standard D	101	39500
Standard D	121	41400
C of A	70	19000
Average	134	41600
STDEV	44	2064
%RSD	33	5

C of A - Certificate of Analysis

Table F - 16. ICP Data for High Purity Standard D.

	Al mg/L	Ca mg/L	Cr mg/L	K mg/L	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L
Standard D	9670	400	893	3520	60.2	67400	727	751	19953
Standard D	9360	397	879	3280	60.7	64300	721	705	19474
Standard D	9460	392	892	3300	59.6	66300	741	711	19444
Standard D	9500	391	888	3430	60.6	66300	732	705	19684
Standard D	9600	407	909	3420	61.5	68600	755	727	20043
Standard D	9570	409	914	3450	61.2	69700	755	742	20043
Standard D	9480	403	897	3520	60.6	68700	742	718	19983
Standard D	9520	406	892	3570	59.9	68600	739	718	19774
Standard D	9310	390	876	3440	59.3	67700	711	711	20013
Standard D	9390	402	893	3360	64.2	65700	748	708	20523
C of A	9600	400	900	3400	60	65800	750	750	19000
Average	9490	400	893	3430	60.8	67300	737	720	19900
STDEV	112	7	12	95	1	1661	14	16	317
%RSD	1	2	1	3	2	2	2	2	2

C of A - Certificate of Analysis

Table F - 17. IC Data for High Purity Standard E.

	Cl mg/L	SO_4 mg/L
Standar E	2260	10900
Standar E	2080	10900
Standar E	2110	10900
Standar E	2080	10800
Standar E	1990	10900
C of A	2150	2400
Average	2100	10900
STDEV	98	45
%RSD	95	100

C of A - Certificate of Analysis

Table F - 18. ICP Data for High Purity Standard E.

	Al mg/L	Ca mg/L	Cr mg/L	K mg/L	Mo mg/L	Na mg/L	Ni mg/L	PO_4 mg/L	SO_4 mg/L
Standard E	9080	38.9	290	3280	29.1	88100	39.0	1153	2541
Standard E	8930	40.1	294	3120	30.6	86100	38.7	1098	2487
Standard E	9040	37.3	292	3160	29.7	88900	38.7	1122	2421
Standard E	9090	37.6	287	3270	29.4	89000	38.0	1079	2424
Standard E	9120	39.5	298	3360	30.2	91300	38.6	1131	2487
Standard E	9030	38.9	290	3430	28.8	92300	38.6	1122	2421
Standard E	9000	39.5	293	3380	29.6	92000	38.5	1113	2454
Standard E	8990	37.8	285	3340	28.2	91400	36.8	1082	2367
Standard E	8790	37.7	286	3300	29.2	90300	36.7	1131	2460
Standard E	8980	37.9	288	3390	30.9	90700	37.4	1110	2514
C of A	9150	40	300	3250	30	89000	40	1200	2400
Average	9000	38.5	290	3300	29.6	90000	38.1	1110	2460
STDEV	95	1	4	100	0.8	1959	0.8	23	51
%RSD	1	3	1	3	3	2	2	2	2

C of A - Certificate of Analysis

7.7 Appendix G: XRF Sample and Standard Counting Statistics

Table G - 1. WD-XRF Counting Uncertainty of RPP Acidified Simulants.

Envelope A (AN-105 & AP-101 simulant), Envelope C (AN-107 simulant), and Envelope B/D (AZ-101 simulant) Analysis

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced for calibration and analysis)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 2-mL DI water + 2-mL HNO_3

	Al Counting Uncertainty (%)	Ca Counting Uncertainty (%)	Cr Counting Uncertainty (%)	Mo Counting Uncertainty (%)	Ni Counting Uncertainty (%)	K Counting Uncertainty (%)	Na Counting Uncertainty (%)	SO_4 Counting Uncertainty (%)	PO_4 Counting Uncertainty (%)	Cl Counting Uncertainty (%)
AN-105	0.58		0.69	0.21		0.37	0.86	1.83	3.31	0.31
AN-105	0.59		0.70	0.21		0.38	0.90	1.88	3.39	0.31
AN-105	0.59		0.70	0.21		0.38	0.89	1.94	3.35	0.31
AN-105	0.58		0.70	0.21		0.37	0.87	1.94	3.46	0.31
AN-105	0.59		0.69	0.21		0.37	0.91	1.83	3.43	0.31
AN-105	0.59		0.69	0.21		0.37	0.91	1.93	3.39	0.31
AN-105	0.59		0.70	0.21		0.38	0.90	1.93	3.41	0.31
AN-105	0.58		0.69	0.21		0.37	0.89	1.93	3.43	0.31
AN-105	0.58		0.70	0.21		0.38	0.90	1.88	3.41	0.31
AN-105	0.59		0.70	0.21		0.38	0.92	1.94	3.45	0.31
AN-105	0.61		0.72	0.22		0.39	0.95	1.97	3.48	0.32
Average	0.59		0.70	0.21		0.38	0.90	1.91	3.41	0.31
AP-101	0.74		1.27	0.22		0.16	0.91	0.77	1.94	0.54
AP-101	0.74		1.26	0.22		0.16	0.92	0.78	1.97	0.53
AP-101	0.75		1.26	0.22		0.16	0.91	0.79	1.98	0.53
AP-101	0.75		1.25	0.22		0.16	0.94	0.77	1.99	0.53
AP-101	0.74		1.24	0.22		0.16	0.91	0.77	1.95	0.53
AP-101	0.75		1.25	0.22		0.16	0.92	0.78	1.97	0.53
AP-101	0.74		1.27	0.22		0.16	0.92	0.78	2.01	0.53
AP-101	0.73		1.26	0.22		0.16	0.89	0.77	1.95	0.53
AP-101	0.75		1.25	0.22		0.16	0.93	0.77	1.97	0.53
AP-101	0.74		1.26	0.22		0.16	0.91	0.77	1.95	0.53
AP-101	0.76		1.32	0.23		0.16	0.97	0.80	1.99	0.54
Average	0.74		1.26	0.22		0.16	0.92	0.78	1.97	0.53
AN-107	3.03	2.40		0.21	0.45	0.54	0.85	0.68	2.33	0.58
AN-107	3.02	2.38		0.21	0.45	0.55	0.83	0.68	2.31	0.58
AN-107	3.02	2.36		0.21	0.45	0.54	0.85	0.68	2.28	0.57
AN-107	2.88	2.42		0.21	0.45	0.54	0.85	0.68	2.26	0.56
AN-107	3.02	2.38		0.21	0.45	0.54	0.85	0.67	2.27	0.57
AN-107	3.16	2.36		0.21	0.45	0.54	0.85	0.67	2.27	0.57
AN-107	2.96	2.39		0.21	0.45	0.54	0.85	0.67	2.29	0.57
AN-107	3.07	2.36		0.21	0.45	0.54	0.85	0.67	2.27	0.57
AN-107	2.90	2.39		0.21	0.45	0.54	0.84	0.67	2.27	0.57
AN-107	2.98	2.45		0.21	0.45	0.54	0.85	0.67	2.31	0.57
AN-107	2.87	2.39		0.22	0.47	0.56	0.89	0.69	2.36	0.58
Average	2.99	2.39		0.21	0.45	0.54	0.85	0.68	2.29	0.57
AZ-101			1.17	0.20		0.56	2.29	1.40		1.01
AZ-101			1.16	0.20		0.55	2.15	1.41		0.98
AZ-101			1.15	0.20		0.56	2.21	1.41		0.99
AZ-101			1.11	0.21		0.54	2.07	1.34		0.96
AZ-101			1.17	0.20		0.56	2.24	1.44		1.00
AZ-101			1.15	0.20		0.55	2.17	1.41		0.96
AZ-101			1.15	0.20		0.55	2.23	1.42		0.98
AZ-101			1.16	0.20		0.56	2.19	1.41		0.99
AZ-101			1.15	0.20		0.56	2.19	1.42		1.00
AZ-101			1.16	0.20		0.56	2.24	1.43		1.01
AZ-101			1.21	0.21		0.57	2.38	1.50		0.99
Average			1.16	0.20		0.56	2.21	1.42		0.99

Table G - 2. WD-XRF Counting Uncertainty of RPP Basic Simulants.

Envelope A (AN-105 & AP-101 simulant), Envelope C (AN-107 simulant), and Envelope B/D (AZ-101 simulant) Analysis

Voltage: 40-kV **Current:** 1.2-mA
Atmosphere: He (99.99%)
Instrument Film: Ultralene - 4-µm (film was replaced for calibration and analysis)
Cell: 31-mm double open end
Sample film Ultralene - 4-µm
Cover film: Micro-porous Teflon (0.2-µm pores)
Analysis Time: 200-sec on peak; 100-sec on background
Volume: 4-mL **filtered** supernate + 4-mL DI water

	Al Counting Uncertainty (%)	Ca Counting Uncertainty (%)	Cr Counting Uncertainty (%)	Mo Counting Uncertainty (%)	Ni Counting Uncertainty (%)	K Counting Uncertainty (%)	Na Counting Uncertainty (%)	SO_4 Counting Uncertainty (%)	PO_4 Counting Uncertainty (%)	Cl Counting Uncertainty (%)
AN-105	0.57		0.67	0.21		0.36	0.86	1.87	3.33	0.30
AN-105	0.57		0.67	0.21		0.36	0.86	1.76	3.22	0.30
AN-105	0.57		0.67	0.21		0.36	0.86	1.71	3.26	0.30
AN-105	0.58		0.67	0.21		0.36	0.86	1.83	3.43	0.30
AN-105	0.54		0.67	0.21		0.36	0.84	1.87	3.31	0.30
Average	0.57		0.67	0.21		0.36	0.86	1.81	3.31	0.30
AP-101	0.71		1.23	0.23		0.15	0.85	0.74	1.90	0.51
AP-101	0.72		1.22	0.22		0.15	0.87	0.74	1.89	0.51
AP-101	0.73		1.24	0.23		0.15	0.91	0.74	1.92	0.51
AP-101	0.73		1.23	0.22		0.15	0.90	0.76	1.95	0.51
AP-101	0.71		1.23	0.22		0.15	0.86	0.74	1.87	0.51
Average	0.72		1.23	0.23		0.15	0.88	0.74	1.91	0.51
AN-107	2.89	2.33		0.21	0.43	0.52	0.80	0.64	2.19	0.54
AN-107	2.67	2.34		0.21	0.43	0.52	0.78	0.64	2.12	0.54
AN-107	2.96	2.35		0.21	0.44	0.53	0.82	0.65	2.16	0.55
AN-107	3.02	2.32		0.21	0.44	0.52	0.82	0.65	2.24	0.55
AN-107	3.06	2.33		0.21	0.44	0.53	0.81	0.65	2.18	0.54
Average	2.92	2.33		0.21	0.44	0.52	0.81	0.65	2.18	0.54
AZ-101			1.13	0.20		0.54	2.15	1.38		0.96
AZ-101			1.10	0.20		0.53	2.05	1.30		0.95
AZ-101			1.12	0.20		0.53	2.10	1.31		0.96
AZ-101			1.11	0.20		0.54	2.09	1.37		0.97
AZ-101			1.11	0.21		0.54	2.07	1.34		0.96
Average			1.11	0.20		0.53	2.09	1.34		0.96

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