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Waste Tank Safety Program

Annual Status Report for FY 1993
Task 3: Organic Chemistry

R. B. Lucke
T.T.W. Clauss
R. Hoheimer
S. C. Goheen

February 1994

Prepared for Westinghouse Hanford Company
under a Related Service Agreement
with the U.S. Department of Energy
Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

Battelle

PNL-8979-3

PACIFIC NORTHWEST LABORATORY
operated by
BATTELLE MEMORIAL INSTITUTE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-AC06-76RLO 1830

Printed in the United States of America

Available to DOE and DOE contractors from the
Office of Scientific and Technical Information, P.O. Box 62, Oak Ridge, TN 37831;
prices available from (615) 576-8401. FTS 626-8401.

Available to the public from the National Technical Information Service,
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Pacific Northwest Laboratory
Richland, Washington 99352

Summary

This report describes the organic analytical support provided by Pacific Northwest Laboratory (PNL) to the Westinghouse Tank Vapor Issue Resolution Program. It describes progress in the first year of what is anticipated to be a multi-year effort at PNL. The focus of this task is to assist in the design and/or selection of sampling and analytical methods and to perform appropriate analyses of Hanford waste tank vapor samples. Major accomplishments in this task for FY 1993 include

- Procurement set up and operation of a laboratory for analysis of SUMMA™ Canister samples
- Validation of a sampling and analysis method for determining levels of normal paraffin hydrocarbon (NPH) in Tank 241-C-103
- Receipt and set up of an ion trap mass spectrometer for the potential field analysis of tank vapor samples.

Work was done to validate the NPH sampling and analysis methods and to prepare the SUMMA™ laboratory. Activities in the first year have addressed the preparation for anticipated sampling of the Hanford tanks. This report emphasizes those issues directly relating to developing a laboratory capability and analytical methods for analyzing Occupational Safety and Health Administration (OSHA) versatile sampling tubes and passivated canister samples collected from Hanford tank farms.

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Introduction

This task supports the tank-vapor project, mainly by providing organic analytical support and by analyzing Tank 241-C-103 (Tank C-103) vapor-space samples, collected via SUMMA™ canisters, by gas chromatography (GC) and GC/mass spectrometry (MS). In the absence of receiving tank-vapor samples, we have focused our efforts toward validating the normal paraffin hydrocarbon (NPH) sampling and analysis methods and preparing the SUMMA™ laboratory. All required milestones were met, including a report on the update of phase I sampling and analysis on August 15, 1993. This update described the work involved in preparing to analyze phase I samples (Appendix A). This report describes the analytical support provided by Pacific Northwest Laboratory (PNL)[a] to the Hanford Tank Safety Vapor Program.

Progress During FY 1993

Procure and Install SUMMA™ GC/MS System at PNL

- Laboratory 13A in the 329 building was made available for the SUMMA™ laboratory.
- A GC/MS system procured by PNL was dedicated to air-sample analysis. The Hewlett- Packard 5971 GC/MS was received March 9. Installation and on-site training was provided April 2-6. The purchase order is shown in Appendix E.
- A SUMMA™ canister sample concentrating system for source-air and ambient-air analysis was procured by PNL. This included an automation system, a calibration system, and a cleaning system for SUMMA™ canisters. This system was from Entech and required a sole source justification. The system was installed in laboratory 13A of the 329 building on April 22-23. The purchase order is shown in Appendix F.

SUMMA™ GC/MS Analysis Technology Transfer

- Technology transfer from Oregon Graduate Institute (OGI) took place in FY 1993. Rei Rasmussen of OGI is a world leader in SUMMA™ canister analysis. Rich Lucke represented PNL on a visit to Rei Rasmussen of OGI on December 8, 1992. They discussed SUMMA™ canister analysis and future technology transfer to PNL. Additional technology exchanges with Rei occurred when he visited Richland on February 4, 1993, for a vapor team meeting and on March 7, 1993, for a vapor conference meeting.
- Technology transfer from Battelle Columbus Ohio (BCO) took place in FY 1993. Battelle Columbus is one of the first laboratories to do analysis with passivated SUMMA™ canisters, and they have much experience with SUMMA™ canister sampling and analysis of components extending beyond the TO-14 list. We have implemented a collaborative arrangement with BCO through an inter-laboratory agreement (ILA) with PNL (Appendix C). Rich Lucke and Therese Clauss visited BCO in March 1993 to discuss air sample analysis using SUMMA™ canisters and the EPA TO-14 method. Tours of the laboratory in operation were provided. Mike Holdren and Debbie Smith of BCO visited PNL's SUMMA™ laboratory the week of August 9 to evaluate PNL's SUMMA™ capability through an ILA. Mike and Debbie were impressed with PNL's

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[a] Pacific Northwest Laboratory is operated for the U.S. Department of Energy by Battelle Memorial Institute under Contract DE-AC06-76RLO 1830.

SUMMA™ system. Our analytical detection at the 1-ppb level was better than Battelle Columbus's response at 10 ppb. The Entech system has a wide diversity of operational modes that gives the most flexibility for analyzing source samples and expanding the analyte list. This is very encouraging. During the visit by BCO, much effort was spent in investigating the source of siloxane peaks seen in TO-14 chromatograms. The siloxane source was isolated to a specific part of the system, and ideas were presented to reduce the amount present. Fortunately, the contaminants do not interfere with the TO-14 target component list. Blank audit canisters from our zero air generator have been prepared and are being sent to BCO to evaluate our cleaning system as per the ILA.

- Pacific Northwest Laboratory has been involved in an ongoing task to accumulate open literature papers and methods related to air toxic analysis. A search and acquisition of the literature for air-analysis methods, including SUMMA™ canister analysis and method TO-14, is ongoing, and acquired literature is kept in room 2 of the 329 building. A database of acquired air-analysis methods has been started and will be updated in the future. Appendix D shows the titles and method numbers acquired by PNL.
- Both Therese Clauss and Rich Lucke attended the American Chemical Society (ACS) short course entitled "Air Toxic Analysis by U.S. EPA Methods" at the Pittsburgh Conference, Atlanta , Georgia, March 6-7, 1993.
- Therese Clauss attended the Air Toxic Symposium by EPA and Air and Waste Management Association at Durham, North Carolina, May 3-7.

Implement SUMMA™ Canister Analysis Capability

- The SUMMA™ laboratory is functional. Numerous hardware problems were discovered and repaired on the recently purchased Entech system. Problems included faulty heaters on both sampler towers, an incorrect cryo trap configured by Entech to trap the #2 focus unit, broken transfer lines due to chemical reaction from the interface, and several vacuum leaks in the cleaning system. A service engineer from Entech spent June 29 repairing and validating the Entech system.
- Entech was subcontracted by PNL for a three-day visit (July 28-30) to assist in PNL's SUMMA™ laboratory. Detection limits, canister cleaning, blank levels, and reproducibility were investigated. A new 60-meter GC column was installed to replace the 30-meter column to improve separation and to reduce column bleed and column flow. The heated transfer line between the Entech concentrator and the GC/MS was replaced with an internally treated stainless steel tubing (Silco-steel). This will allow for better stability of polar compounds and a more rigid transfer line as compared to a fused-silica capillary tubing. This also eliminated some of the persistent contamination seen in the system.
- Over 40 SUMMA™ canisters are owned by PNL as well as a cleaning system to routinely clean four canisters at a time. The cleaning system is working, but a high vacuum pump is needed to obtain lower vacuum (<10 mtorr) and a more efficient cleaning.

Initial Development and Validation of SUMMA™ Canister Analysis Using EPA Method TO-14

- A draft strategy plan was prepared for developing and validating SUMMA™ canister analysis and is included in Appendix G.
- We have obtained excellent detection of 40 components on the TO-14 target list at the 10-ppb and 1.0-ppb levels. Figure 1 shows the GC/MS total ion chromatogram of a 10-ppb TO-14 standard that was prepared by diluting a 100-ppb Scott Specialty Gas Standard TO-14 mixture. Table 1 lists 40 components on the T-14 list and the corresponding peak number to Figure 1.

Table 1. List of TO-14 Standard Components

1. dichlorodifluoromethane (FREON-12)
2. methyl chloride (chloromethane)
3. 1,2-dichloro-1,1,2,2,-tetrafluoroethane (FREON-114)
4. chloroethene (vinyl chloride)
5. methyl bromide (bromomethane)
6. ethyl chloride
7. trichlorofluoromethane (FREON-11)
8. 1,1-dichloroethene (1,1-dichloroethylene)
9. dichloromethane (methylene chloride)
10. 1,1,2-trichloro-1,2,2-trifluoroethane (FREON-113)
11. 1,1-dichloroethane
12. cis-1,2-dichloroethene (cis-1,2-dichloroethylene)
13. trichloromethane (chloroform)
14. cis 1,2-dichloroethane
15. 1,1,1-trichloroethane
16. benzene
17. carbon tetrachloride
18. 1,2-dichloropropane
19. trichloroethylene
20. cis 1,3-dichloropropene
21. trans 1,3-dichloropropene
22. 1,1,2-trichloroethane
23. methyl benzene (toluene)
24. 1,2-dibromoethane
25. tetrachloroethene (tetrachloroethylene)
26. chlorobenzene
27. ethylbenzene
28. m-xylene (1,3-dimethylbenzene)
29. p-xylene (1,4-dimethylbenzene)
30. styrene
31. 1,1,2,2-tetrachloroethane
32. o-xylene (1,2-dimethylbenzene)
33. 1,3,5-trimethylbenzene
34. 1,2,4-trimethylbenzene
35. chloromethylbenzene, alpha (benzyl chloride)
36. m-dichlorobenzene (1,3-dichlorobenzene)
37. p-dichlorobenzene (1,4-dichlorobenzene)
38. o-dichlorobenzene (1,2-dichlorobenzene)
39. 1,2,4-trichlorobenzene
40. hexachloro-1,3-butadiene

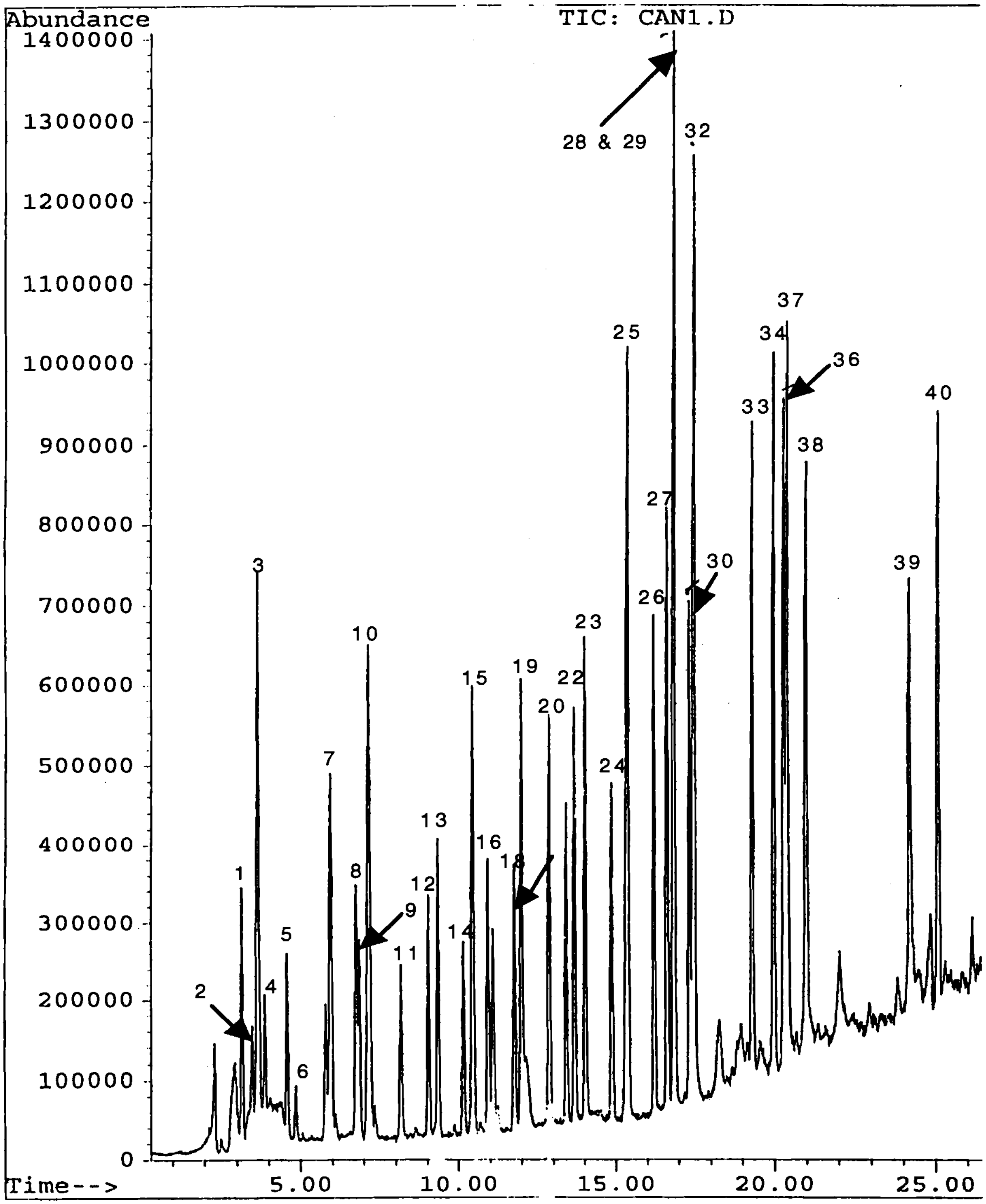

Figure 1. GC/MS Total Ion Chromatogram of a 10-ppb TO-14 Standard Mixture from a SUMMA™ Canister.

Aerosol Support

- Organic analysis was provided to the aerosol task to support the validation studies for the aerosol/ flammability issues. Results are not included in this report, but can be obtained from the Task 2 Annual Report and from the published report PNL-8875. This was a major effort by the organic task and consumed over 25% of their time in FY 1993. Hundreds of laboratory samples and standards were extracted and analyzed by GC/MS.

- A tributyl phosphate (TBP) study was added to the aerosol study. Ryan Hoheimer {Northwest Organization of Colleges and Universities for Science (NORCUS) student} worked under the supervision of T. Clauss on expanding the Occupational Safety and Health Administration (OSHA) versatile sampler (OVS) sorbent tubes to include TBP. Calibration of GC/MS and extraction efficiencies were determined. The extraction efficiency was determined to be an average of 101% and was determined from triplicate spikes at three different concentrations.

Ion Trap Analysis

- The ion trap mass spectrometer was acquired and delivered in FY 1993. Training included a visit to Oak Ridge National Laboratory (ORNL) in October by two PNL staff (R. B. Lucke and S. A. Clauss). The ion trap was received at PNL and installed by ORNL on October 15, 1993, in room 9A of the 329 building. As specified in our statement of work, additional vapor-program work was not done on the ion trap in FY 1993. Two proposals for the feasibility and validation of the ion trap mass spectrometer in regards to Tank C-103 headspace analysis were delivered to WHC and are included in Appendix B. A proposal on the validation of the ion trap mass spectrometer as applicable to Tank C-103 was delivered to WHC in December 1993. After a meeting with WHC and DOE, a reduced scope proposal was requested and delivered to WHC in April 1993.

Practice SUMMA™ Method with Phase 1 Samples and Optimize for Hanford Waste Tanks

- Because of continuous sampling delays beyond PNL control, phase I sampling did not take place in FY 1993, and samples were not received.

Assistance to WHC

- Teamwork with the WHC vapor program is ongoing, and continuous support to WHC was provided. This included attendance at the vapor-team meetings, vapor-conference meetings, sub-TAP meetings, and special sub-task meetings. The PNL organic task has been dedicated to support WHC and has been available to assist WHC in all matters related to the vapor program. In addition, PNL kept a proactive communication with WHC and Northwest Instrument Systems, Inc. (NISI) in implementing TO-14 and the direct GC/MS.

- Three gas bottles of a diagnostic test mixture were procured from Scott Specialty Gases for the vapor program. One bottle was provided to WHC for the mobile laboratory work. Because of concerns from WHC on the validity of the constituents present in the diagnostic test mixture, PNL analyzed the mixture and determined that butyl benzene was present rather than n-butanol. Scott Specialty Gases confirmed this and provided credit to the procurement.

- Staff at PNL worked with Jim Huckaby to validate a technique to determine the depth of the organic layer in Tank C-103. This included the preparation of a simulated Tank C-103 mixture with NPH, tributyl phosphate (TBP), and a sodium nitrite water solution. Studies using an ohm meter and a wire electrode that pierces through the various tank layers indicate that the depth of the organic layer can be readily determined.

- A work plan to analyze the high-efficiency particulate air (HEPA) and Carbitrol filters obtained from the riser of Tank C-103 was prepared and delivered to WHC. A copy is provided in Appendix H.

Phase Zero Completion

- Analysis of phase zero mobile analytical laboratory truck (MALT) samples from August 1992 were completed in October 1992, and a final report was delivered to WHC. A copy of the report is provided in Appendix I.

Battelle
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Telephone (509) 376-3286

August 10, 1993

Jerry Osborne
Tank Vapor Issues Resolution Manager
Westinghouse Hanford Company
Richland, Washington 99352

Dear Jerry,

The Tank Vapor Project milestones include a report on August 15, 1993, describing the completed analyses of phase 1 samples from Tank 241-C-103. As you know, we have not yet received samples from this tank and are therefore unable to report these analytical results.

Meanwhile, we have been preparing for phase 1 samples. Some of the activities we have been involved in are summarized below:

- Preparing for the sampling and analysis of tank vapors to test for their flammability.
- Identifying and preparing laboratory space for developing Summa laboratory capability.
- Procuring equipment and supplies for the Summa laboratory for tank-vapor analyses.
- Participating in frequent strategy meetings to assist in program direction.
- Evaluating literature.
- Arranging a collaborative arrangement with Battelle Columbus to help validate our Summa laboratory capabilities.

Details of some of these activities are provided in the enclosed material. These activities have allowed us to prepare for phase 1 and phase 2 samples. Our Summa laboratory is now functional. The instrument is operating, and standards are being evaluated. This week, we will host two staff from

Battelle, Columbus, who are leading experts in the operation of a Summa laboratory. Their role is to help us in the operation and validation of our laboratory.

As soon as the samples from phase 1 become available to us, we will make every effort to provide results at the quickest possible convenience. Once results have been obtained and evaluated, I will be reporting them to you.

Sincerely,

Steven C. Goheen, Ph.D.
Program Manager
Tank Vapor Project.

SCG:lja

Enclosure

cc: Ronald Hall
James Huckaby
Benjamin Johnson
Bruce Lerner
Michael Ligotke
Richard Lucke
Michael Story

SUMMARY REPORT OF PNL ACTIVITIES FOR PHASE I OF THE TANK VAPOR PROGRAM

Summary: The following document outlines PNL's activities for Phase I of the tank farm vapor program. Because of continuous sampling delays beyond PNL control, Phase I sampling has yet to take place. Activities up to this date have dealt solely with the preparation for anticipated sampling of the Hanford tanks. This report addresses only those issues directly relating to developing a laboratory capability and analytical methods for analyzing sorbent tube and passivated canister samples collected from Hanford tank farms. Once a survey sample from the tank farms has been received by PNL for analysis, method development and analysis for the completion of tasks listed for Phase I of the tank farm vapor program will follow. It is expected that from the first sampling and analysis, questions surrounding the issue of tank flammability will be answered. Survey summa canisters analyzed by PNL will aid in PNL's attempt to expand the model method target analyte list to include polar compounds of interest and components found in tank samples. This report describes the needs and corresponding actions already executed by the PNL group for Phase I of the program.

I. Laboratory Space Availability

Needs	Action
• The method development group needed enough laboratory space to house a GC/MSD interfaced with a canister concentrator, a standards dilution unit, and a canister conditioning oven (approximately 45 square feet of bench space and 5 square feet of hood space). Additional floor space was needed to allow for two auto-sampling manifolds to be hooked up to a concentrator. Additional bench and hood space was needed to allow chemists to do minimal bench work.	• Laboratory 13A in the 329 building was reserved for housing the GC/MS and cryo-concentrator instruments. Obsolete equipment previously housed in laboratory 13A was removed and excessed.
• The laboratory used for summa canister analysis had to be capable of handling up to 2 µCi sample levels.	• Laboratory 13A was cleaned and downgraded leaving two-thirds of the lab to do vapor work and keeping one-third available for the occasional need to do work on radioactive samples.

II. Ordering of Equipment

Needs	Action
• Analysis of summa canister samples required GC/MS instrument.	• GC/MS instrument was ordered in 12/92 and received in 2/93. The installation of the instrument was completed in 3/93. Additional training on the operating system of this instrument was received on 3/25/93.
• Analysis of summa canisters required a canister concentrator, a means for making in-house gas standards, and a means for conditioning used canisters. • A source of clean air was needed for blending gas standards and making gas sample blanks.	• An Entech cryo-concentrator with a standards dilution unit and a canister conditioner, was been sole-sourced ordered in 12/92 and received in 4/93. Installation of this equipment was completed in 6/93. Additional training on operation, use and maintenance of this instrument was received in 7/93. • A clean air generator and pump were ordered (5/93), received (6/93), and installed (7/93).

III. Order Expendable Equipment.

Needs	Action
• Summa canisters.	• 20 6-Liter canisters have been purchased by PNL.
• Tedlar Bags.	• 10 bags have been purchased for this program.
• Sorbent tubes for aerosol sampling.	• 80 sorbent tubes have been purchased for this program.
• Chemical standards and solvents for aerosol analysis.	• Supplies are ordered as needed.
• Gases (He, liquid N_2, CO_2) and gas standards.	• Supplies are ordered as needed.
• Assorted GC columns, tubing, syringes, sample vials.	• Supplies are ordered as needed.

IV. Technology Transfer/Consulting/Technical Training

Needs	Action
• Open communication between the two groups analyzing canister samples, PNL and WHC chemists, needed to be established.	• PNL chemists have met with WHC chemists and M. Story to establish communications between the two groups and to report laboratory progress. Both groups identified individual group needs as well as objectives of the sampling plan (sampling train) and potential contacts for expertise in the area of canister sample analysis (10/22). • PNL chemists continue to attend bi-weekly meetings conducted by WHC project leaders to report activities and progress made toward bring summa canister technology to the Hanford site.
• Other experts in area of summa canister sampling (method TO-14) and canister analysis needed to be identified. Meetings with these experts needed to be arranged for the purpose of a technology transfer between the groups. • A back-up laboratory which could provide similar (TO-14) service had to be identified to validate future standards and analyze duplicate samples. • Site visits to other laboratories with summa capability had to be arranged for the purposes of technology transfer.	• Oregon Graduate Institute (OGI), Tigard, OR, was identified as a laboratory experienced in TO-14 analysis. • PNL joined WHC and other scientists working on this program in a visit to the OGI laboratory. • PNL has put into place, an inter-laboratory agreement (ILA) between PNL and Battelle, Columbus (BCO) scientists in the Environmental Phyusics and Chemistry group. The BCO group has expertise in the area of summa canister sampling and analyzing components not listed on the TO-14 target list. They are well recognized and published in this area. • PNL scientists working on summa canister method development have visited the BCO laboratory for the purposes of technology transfer (4/1).

• Scientists at PNL who were assigned the task of summa canister method development needed to gain information on air toxic regulations, the latest instruments designed for these analyses, and information pertaining to the latest studies in this area.	• PNL scientists attended a short course "Analysis of Air Toxics by Compendium Method TO-14" sponsored by the American Chemical Society in conjunction with the Pittsburgh Conference (3/6/93). • PNL scientists attended the Pittsburgh Conference in Atlanta, GA (3/8-3/12/93). • PNL scientists attended the Air Waste Management Association symposium on the "Measurement of Toxic and Related Air Pollutants" in Durham , NC (5/4-5/7/93).

V. Literature Searches

Needs	Action
• A review of available information from tank 103C was needed. Information pertaining to sampling which has been done, the analyses performed on the samples, sample results, and a list of worker complaints .	• Some of the available information from tank 103C samplings were reviewed by PNL.
• A literature search of work done by other groups using passivated canister methods to sample for volatile organics in ambient air was needed.	• Literature reference files have been started by PNL for papers covering passivated canister sampling techniques and thermal desorption tubes. A full "summa" literature search through PNL library was completed (11/4). • PNL acquired copies of the OSHA methods, NIOSH methods and "Compendium for the Measurement of Toxic Organic Compounds in Ambient Air" also-known-as EPA compendium methods TO-1,2,..14.
• PNL needed to research and review existing methods for measuring and analyzing VOC's in ambient air to use as references in validating PNL's method.	• Review of pertinent methods has begun and will be done as time permits.

VI. Laboratory Method Development

Needs	Action
• A method was needed for measuring NPH in aerosol samples.	• PNL developed an analysis method for the determination of normal paraffin hydrocarbons (C_{11}-C_{16}) in air. PNL chemists also worked with PNL engineers to develop a sampling method for NPH onto OSHA versatile samplers for the collection of NPH.
• PNL must develop the capability for making standards in-house and have them validated by another lab, or compare them against certified standards.	• The first step in this effort is to learn how to dilute and analyze TO-14 compounds. Work in this area has begun. Battelle Columbus experts in TO-14 will be visiting PNL during the week of 8/9-8/13 to help PNL develop this capability.
• The instrument will be optimized to meet the TO-14 method requirements. PNL will establish that they can perform the TO-14 analysis with the required QA as per method, before modifying the instrument for tank vapor analytes.	• This work is currently in progress.
• Using method TO-14 as a model, modifications will be made to the target list to accommodate the tank vapor matrix and potential components.	• Action toward this effort cannot begin before the tanks are sampled and PNL receives a trial canister sample to explore.
• The level of method development needed (validation, QA/QC, etc.), must be determined before technology transfer to the WHC mobile lab can take place.	• Again, action in this area is highly dependent upon actual sampling dates. Work toward this end is targeted for fiscal '94.

Progress to date (summary):

A laboratory reasonably free of organic vapor contaminants has been dedicated for the purpose of serving as a summa canister analysi laboratory. Purchased equipment for this project has been installed and scientists are in the process of becoming familiar with all the instruments and methods to be used for TO-14 analysis. PNL scientists have established a close working relationship with Battelle Columbus summa canister technology experts in an effort to provide the most up-to-date scientific information possible to this project. PNL gives continuous updates to WHC vapor team leaders and attends regularly scheduled project meetings. Additional tasks assignments issued by WHC are executed by PNL staff in a conscious effort to provide continuous support to the vapor team and project a positive working relationship.

Remaining Actions Needed:

The waste tanks need to be sampled to give PNL a trial sample to study and begin the extension of the TO-14 analyte list.

Appendix A

Report on Phase I Update

Appendix B

Ion Trap Proposals

Battelle
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January 4, 1993

Jerry Osborne
Westinghouse Hanford Company
P. O. Box 1970
Richland, WA 99352

Dear Jerry:

Subject: Ion Trap Proposal

I have attached a final copy of the Ion Trap proposal including cost information. I have not heard any more information from you regarding this proposal since we sent the draft several months ago. Please let me know if you would like us to expand or adjust the scope. We are anxious to work with this instrument, given the opportunity and funding.

Sincerely,

Steven C. Goheen

Steven C. Goheen, Ph.D.
Senior Research Scientist
Chemical Sciences Department

SCG:gkr

Encl.

cc: R. M. Bean, PNL, w/o enc.
T. R. W. Clauss, PNL, w/o enc
R. B. Lucke, PNL, w/o enc.
G. M. Hong, PNL, w/o enc.
D. M. Strachan, PNL, w/o enc.

margaret

PROPOSED PROGRAM PLAN FOR THE ION TRAP ON THE TANK VAPOR PROGRAM

R. B. Lucke, S. C. Goheen, T. R. W. Clauss, G. M. Mong

INTRODUCTION: This program plan describes how PNL would use the ion trap spectrometer (ITS) from Oak Ridge National Laboratory (ORNL) to support the Hanford Tank Vapor Program. The ITS was developed as a screening tool to observe and potentially identify organic material in the gas phase. To develop a method using the ITS which would be useful to the Hanford Tank Vapor Program would require a program like the one described in this plan.

This plan is composed of 5 major components. These include the following:

I Field survey

II. Method development

III. Preparation of Standard Operating Procedure (SOP)

IV. Method Validation

V. Field use.

Background:

This instrument is a vendor bought ion trap from Finnigan with attached sampling modules developed by ORNL. The ITS is a newer generation to the ion trap detector (ITD). It is not the Finnigan ion trap mass spectrometer (ITMS) which has MS/MS and high resolution capabilities. It is not interfaced with a gas chromatograph. The modules attached to the ITS include an air sampling module, a purge module for water and soil samples, and a thermal desorption module to use with samples collected on thermo desorption tubes. Two minute analysis or on-line monitoring is possible with this system. It should be noted that the ion trap by itself is best suited for ambient air analysis or specific point source analysis in which there are only a few analytes present. In the case of source analysis, or complex mixtures (tank 103C), it may be of limited use. To our knowledge, complex mixtures have not been studied with this instrument.

Its usefulness under these conditions would need careful evaluation. Such an evaluation is included as part of this program. However, the ITS was developed by DOE for use in the field for problems like monitoring tank 103C. This program provides an opportunity for DOE to evaluate the usefulness of the ITS for these or similar complex mixtures, and if possible, to develop a validated procedure for use in monitoring Hanford tanks. As an example, the high sensitivity of the ITS may provide useful ambient air sampling data, acting as a sensitive trigger which determines the timing of more sophisticated sampling.

OPERATIONAL STATUS OF THE ION TRAP:

The ion trap is now set up and running at PNL in the 329 building. The three sampling modules (air , purge, and thermal desorption) are working fine. Because of the complexity of the headspace in the Hanford Tank Farms, there is a need for chemical ionization in addition to the existing electron impact ionization on the ion trap. It is our understanding that this ion trap has this capability but it is not working due to a hardware or software problem. ORNL is aware of this and is working with Finnigan to correct this.

PROPOSAL

LABORATORY ANALYSIS WITH THE ION TRAP:

Below is a more detailed outline of our program plan. Descriptions of all these areas are not contained in this draft. More detail will be provided upon request.

I Field survey

- Evaluation of potential problems using ITS in the field
- Direct tank sampling
- Ambient air sampling near 103C
- Soil sampling near 103C
- Other applications may be identified in the field.
- Evaluation of data collected from field survey.

II. Method development

- Study field survey data.
- Analyze simulated field conditions in the laboratory

- Evaluate multiple samples for precision and accuracy determination
- Pattern recognition
- Separation methods (filters, membranes, or simulated GC)
- Interference investigations (water, ammonia, TBP, etc.)
- Report data collected, recommendations made.

III. Preparation of Standard Operating Procedure (SOP)

- Draft prepared from method development results.
- Test draft procedure in the laboratory
- Modifications, if needed
- Field test
- Final SOP delivered to WHC

IV. Method Validation

- Follow SOP with standards in the laboratory
- Identify precision and accuracy
- Compare results with established methods (e.g., SUMMA)
- Report on results to WHC

V. Field use.

- Field validation against SUMMA
- Used to determine events at tanks (e.g. when to sample with SUMMA)
- Survey dose when workers are in the tank farm
- Validate existing survey techniques (e.g. OVM)

Detailed descriptions of a sampling of the component areas which may be studied in this program are given below:

I Field survey

Continuous Monitoring of Ambient Air: The ion trap is designed for detailed ambient air analysis. For example, if the OVM monitors used by the IH personnel are showing 18 ppm organics, the ion trap may confirm this and possibly identify some of the organic components. If changes are noted in the ambient atmosphere, the ITS data may act as an immediate trigger for other sampling plans.

On-line Monitoring of Tank Headspace: The ion trap can be configured for on-line monitoring of the headspace stream during the purge and sample period. The data provided can be evaluated and used to access the usefulness of the ITS in the analysis and characterization of headspace gases from Hanford waste tanks. Sample splitting can be accomplished to allow part of the sample to be screened by ITS analysis, the pattern of components recognized, and the rest of the sample collected by SUMMA and analyzed in detail by other mass spectral analyses.

Soil Analysis in 103C Tank Farms: Using the purge module, perform screening analysis of soil samples taken in and around 103C tank farms. This will provide information pertaining to possible organic components in the soil at 103C and if they are contributing to the unknown vapors. This will directly address safety concerns voiced by tank farm workers.

Thermal Desorption Analysis: Multisorbent tubes and DAAMS tubes can be analyzed by the ion trap in 3 minute analysis times. The ion trap uses the same type of sorbent tubes used in earlier Phase 0 sampling for ORNL. Sampling with the sorbent tubes followed by thermal desorption into the ion trap provides a concentration method such that the detection limits are dramatically improved. This is for ambient air sampling around the tank farms and not for direct tank sampling.

II. Method development

Analysis of Simulated 103C Tank Headspace: The concentration and content of the headspace vapors in 103C tank may result in limited application of the ion trap for direct sampling. The data obtained by the ion trap may be too complex to be useful since no gas chromatograph or separation system is used prior to the ion trap. This study will use the ion trap in direct analysis of a simulated 103C Tank headspace. The simulant will consist of a nonradioactive sample composed of TBP, NPH, 1-butanol, acetone, acetonitrile, and selected alkylnitriles. The data from this analysis will be evaluated to determine the applicability in the characterization of the headspace vapors by ion trap.

Laboratory Headspace Analysis of Organic Layer from Tank 103C: A grab sample of the organic layer in tank 103C is scheduled to be taken by WHC. Direct headspace analysis of the organic layer will be provided.

Chemical Ionization with Ion Trap: PNL will work closely with ORNL and Finnigan to restore chemical ionization capability of the ITS. Chemical ionization will be compared with the existing electron impact source on the ion trap. Chemical ionization will provide less mass spectral fragmentation such that analysis of complex mixtures will be more useful, resulting in a significant increase in the utility of ITS analysis of multicomponent samples.

Pattern Recognition Study: Data obtained from the direct headspace analysis by the ion trap, a pattern recognition study will be done as an attempt to deconvolute the data, and correlate characteristics leading to events. This phase must be conducted with concomittant GCMS studies of SUMMA canisters or with simulants of known composition.

Development of Separation Method(s) Prior to the Ion Trap: The complexity of tank 103C headspace limits the ITS's use in direct analysis . An up front separation may be required prior to the ion trap analysis. PNL will explore separation and filtration methods prior to the ion trap. Filtration and separation methods to consider are membrane fibers and a capillary transfer line to separate like components prior to the ion trap.

III. Preparation of Standard Operating Procedure (SOP)

Once best application of ITS is determined, SOP written for specific application, which includes additional equipment required, if any. Develop an SOP detailing procedures to be followed for a number of potential sampling/analysis scenarios, to include tank sampling.

IV. Method Validation

Validation of SOP under field conditions using samples of known composition (e.g., SUMMA canisters that have been well characterized or lab standards. Parallel ambient air sampling with summa canisters and compare content/level results.

V. Field use.

Mobile Laboratory: The ion trap has to be protected from the environment when used in the field. A mobile laboratory is required such that 110V is provided for the ion trap. The WHC mobile laboratory will provide space and power for the ion trap. In addition, PNL will purchase a portable generator such that the ion trap can be ran from the back of a Suburban or a truck with a canopy in case the WHC mobile laboratory is not available.

- If it is determined that best use is ambient sampling is best application, could be used as an alarm with calibrated standards in field, for determining when organics too high for people to be working without air-line.
- If source and filtration/or separation is best application, follow SOP in field with additional, if any, peripherals hooked up to ITS. Intermittent parallel sampling with other methods could be used to determine if ITS dirty, or loss of calibration has occurred over time.

Cost

The cost of this program is contingent upon the schedule. An estimate of cost is approximately 1 FTE for the duration of the program. It is anticipated that at this funding level, it would take approximately one year to prepare a SOP.

Battelle
Pacific Northwest Laboratories
Battelle Boulevard
P.O. Box 999
Richland, Washington 99352
Telephone (509) 376-3206
FAX: (509) 376-2329

February 19, 1993

Jerry Osborne
Westinghouse Hanford Company
P. O. Box 1970
Richland, WA 99352

Dear Jerry:

Subject: REDUCED SCOPE PROGRAM PLAN FOR THE ION TRAP ON THE TANK VAPOR PROGRAM

Attached is a reduced scope work statement on the vapor tank program. If you have any questions, please contact me.

Thanks.

Sincerely,

Steven C. Goheen, Ph.D.
Senior Research Scientist
Chemical Sciences Department

SCG:gkr

Enc.

cc: R. M. Bean, PNL, w/enc.
S. A. Clauss, PNL, w/enc.
J. C. Fulton, WHC, w/enc.
R. B. Lucke, PNL, w/enc.
G. M. Mong, PNL, w/enc.
M. Story, NI, w/enc.
D. Strachan, PNL w/enc.

REDUCED SCOPE PROGRAM PLAN FOR THE ION TRAP ON THE TANK VAPOR PROGRAM

R. B. Lucke, G. M. Mong, S. A. Clauss, and S. C. Goheen*

Pacific Northwest Laboratory, P. O. Box 999, Richland, WA 99352

INTRODUCTION: Pacific Northwest Laboratory (PNL) has been requested by the U. S. Department of Energy, Headquarters (DOE-HQ), Richland Operations Office (DOE-RL), and Westinghouse Hanford Company (WHC) to provide a revised program plan to evaluate the ion trap spectrometer (ITS) from Oak Ridge National Laboratory (ORNL). The previous Ion Trap Plan was a full one year study on the evaluation, method development, and validation of the ITS as applied to complex mixtures, such as tank 103C. This program plan includes a reduced scope from that of the initial proposal. In this revised plan, the ITS will be evaluated in the laboratory for its applicability to the study of vapors such as those emitted from tank 103C. This plan will allow an evaluation of the ITS to be done prior to field application so that chemical interferences or physical problems will be identified before the expense of transfer to Tank 103C is affected.

PROPOSAL: This plan is composed of the following components using the ORNL air sampling module on the ITS: These include the following:

- optimization of the ORNL air sampling module
- analysis of Summa canisters with samples taken directly from inside tank 103C (These samples will be provided by WHC.)
- preparation and analysis of a simulated mixture of major components known to be emitted from tank 103C (part of this study will provide information on relative sensitivity of the various compounds and identify mutually interfering components. This is essential to define those species which will mask or otherwise overwhelm the detection of other compounds in the vapor sample.)
- laboratory analysis of the headspace from a dip sample of the organic layer in tank 103C (in addition to summa cannistor sampling, analysis of the dip sample will provide further information on the applicability of the ITS as applied to Tank 103C.

The ORNL air sampling module is a prototype module that is very sensitive to the operating parameters. Any variable that is not precisely optimized for the concentration level of the sample may result in major response loss and unresolved masses such that the results may be ambiguous. In addition to the ITS parameters, six operating parameters on the ORNL air sampling module influence its performance: pulse duration setting, pulse delay setting, Helium pressure, Helium flow, sample flow, and split flow. All of these parameters must be manually controlled, and only the pulse delay and pulse duration knobs have numerical settings. Hence, an evaluation of the optimization of the air sampler must be done to obtain meaningful mass spectra from the varying concentration within the vapor samples. Calibration of the ITS will consist of an autotune of PFTBA.

This study is reduced in scope from our earlier proposal and only includes use of the air sampling module. It does not include the following:

- calibration with gas standards.
- evaluation of the thermal desorption unit
- evaluation of the purge unit for liquid, solid, and sludge samples
- field use of the ion trap.

DELIVERABLES: Deliverables include a letter report to WHC that will include all pertinent mass spectra. Recommendations on future directions will be provided by PNL, but PNL will not be responsible in interpreting the mass spectra.

BUDGET: Estimated funding needs: The requested funding is expected to allow us to perform the minimal work needed to demonstrate the applicability of the ITS to tank 103C vapor. In the event that the data appears promising, additional work and additional funding will be recommended.

$ 30K	Staff
$ 10K	Standards, flow controllers, and supplies
$ 40K	Total

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*(509) 376-3286

Appendix C

Inter-Laboratory Agreement with Battelle Columbus Ohio

RECEIVED

NOV 11 1992

S.C. GOHEEN

TO: Theres Klaus
FROM: Debbie Smith
SUBJECT: Visit To Battelle Columbus

As per our conversation on 11/5/92, I am enclosing estimated costs for the analysis of ten (10) canisters (blanks and standards) using EPA Method TO-14 as well as costs for an initial and a follow-up visit to Battelle Columbus by Battelle PNL staff. Although your main focus will be the analysis of canisters using EPA Method TO-14, I believe it would be very beneficial for your staff to become familiar with our groups capabilities in atmospheric processes, emission characterization and ambient air sampling and analysis methods. In my cost estimate for your visit, I have included our staff time to review our TO-14 equipment and procedures with you. I also hope that you can stay long enough so that I can show you our other facilities for ambient air characterization. I have included costs for an initial visit of one or two days and a follow-up visit of one day.

Analysis of 10 Canister Samples (blanks and standards) @ $300 per sample (see cost sheet)	$3,000
Initial visit of one day and follow-up visit of one day	$5,000
Initial visit of two days and follow-up visit of one day	$7,500

Please call me at (614) 424-4114 if you need any further information.

PRICE LIST*

for

ANALYTICAL SERVICES FOR AIR TOXICS

Analysis of canisters for up to 41 compounds on the reverse side of this page (Compendium Method TO-14)	$300
Analysis of canisters for up to 41 compounds on reverse side of this page plus analysis for C_2 through C_6 hydrocarbons	$400
Analysis of canisters for compounds that are not on the 41-compound list	Quoted at client's request
Canister cleaning	$ 50
Canister rental (includes clean-up)	$150/month
Evaluation of canister sampler (Includes humidified ultra-zero air and calibration gas challenge)	$500
Adsorbent trap analysis for up to 41 compounds on the reverse side of this page	$500
Adsorbent trap analysis for compounds that are not on the 41-compound list	Quoted at client's request

Send samples to:

Mr. Patrick J. Callahan
Battelle Memorial Institute
505 King Avenue
Columbus, OH 43201-2693

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* This price list is in effect as of August 1, 1991. Prices are quoted on a per sample basis. Minimum charge is for 5 samples. Return shipment of canisters from Battelle to U.S. locations will be by UPS ground service or equivalent. For express air delivery add $15 per container.

Justification for an interlaboratory agreement for the exchange of technical information between Pacific Northwest Laboratories and Battelle Columbus Division pertaining to the analysis of passivated canister samples by GC/MS.

Battelle Columbus Division (BCD), has unique capabilities, facilities and know-how which are necessary for the performance of PNL's work pertaining to the Waste Tank Vapor Program (WTVP). This is a justification for the pursuit of an interlaboratory agreement between Battelle Columbus Division Department of Environmental Physics and Chemistry (BCD), and Pacific Northwest Labortories (PNL) staff working on Task 03 of the Waste Tank Vapor Program, organic analysis, for the exchange of technical information pertaining to the analysis of summa canister samples by GC/MS. This technique is of importance to the WTVP because summa canister samples are part of the sampling scheme proposed by the Hanford Vapor Team in an effort to characterize waste tank head-space vapors. Task 03 will use EPA Compendium Method TO-14 as a model and the Department of Environmental Physics and Chemistry at the Battelle Columbus Division is funded by the U.S. Environmental Protection Agency to design and perform studies which will support eventual revision of the TO-14 method. BCD is recognized as experts in this technology and more importantly, in the expansion of the TO-14 method. Transfer of this particular expertise and the use of BCD's unique facility to validate PNL in this area, is necessary for PNL staff to achieve the mile-stone goals as written in their statement of work for this project. Establishment of this ILA is written into PNL's feasibility study which is part of PNL's Project Plan to Westinghouse Hanford (WHC). Because of their facility capability, it is also essential for BCD to perform as a referee laboratory to run standards for PNL's validation. Specific actions required by both BCD and PNL are detailed in a statement of work.

Statement of Work for the exchange of technical information between Pacific Northwest Laboratories and Battelle Columbus Laboratories, pertaining to the analysis of passivated canister samples by GC/MS.

This statement of work (SOW) describes an exchange of technical information between Battelle Columbus Laboratories, Department of Environmental Physics and Chemistry (BCL), and Pacific Northwest Labortories (PNL) staff working on Task 03 , organic analysis for Phase I characterization of the Waste Tank Vapor Project (WTVP). In particular, information pertaining to the analysis of summa canister samples by GC/MS is of importance to the WTVP because summa canister samples are part of the Phase I sampling scheme proposed by the Hanford Vapor Team. Task 03 will use EPA Compendium Method TO-14 as a model. The Department of Environmental Physics and Chemistry at Battelle Columbus is well recognized as leaders in the use of the TO-14 method. This SOW describes the information necessary to train PNL staff to use the TO-14 method and the requirements for BCL to perform as a referee laboratory to run standards for PNL. Specific actions required by both BCL and PNL are detailed below.

I. Battelle Columbus Laboratories (BCL) staff performing summa canister analysis as per EPA compendium method TO-14 shall:

1. Allow PNL staff working Task 03 of the WTVP and other Hanford scientists working on the WTVP to visit the BCL laboratory dedicated to summa canister analysis and to discuss with PNL staff specific items pertaining to EPA compendium method TO-14. This visit will allow the Hanford Vapor Team to observe BCL's capability in this area and may include, but is not limited to a formal presentation to be prepared by BCL, discribing their capability in this area. This initial visit is to be arranged by the PNL staff working on Task 03 of the WTVP to take place on a mutually agreed upon date (by PNL, BCL and Westinghouse Hanford). This visit will include:

   - contact with BCL staff, Robert Coutant, Budget Manager (614-424-5247), Debra L. Smith, Researcher (614-424-4114), and Michael W. Holdren, Researcher;

   - disclosure by BCL staff of specific requirements for implementing and validating TO-14 by PNL project scientist and any recommendations for demonstrating laboratory TO-14 analysis capability including instrument system control;

   - disclosure by BCL staff, of the procedures and protocols used to limit known interferences and which describe requirements and recommendations relating to the cleanliness of the proposed laboratory to do TO-14 analysis. Procedures should be supported by data which demonstrates that detection limits of 0.2 ppbv per analyte (in the TO-14 method) are achieved;

   - disclosure by BCL staff, of specific requirements for making in-house TO-14 standards and relevant standard storage protocols to include temperature and humidity conditions, and records of shelf-life stability studies for such standards;

- disclosure by BCL staff, of method(s) for cleaning and evacuating summa canisters, including QA criteria, labeling, and chain-of-custody procedures.

2. Analyze summa canisters (TO-14 standards and blanks combined) supplied by PNL, to verify PNL's standard making and canister cleaning capability. The target analytes with required limits of quantitation for each analyte are listed in Table 1. BCL must demonstrate that the GC/MS system used is clean as specified in TO-14, by showing that the system used is capable of detecting 0.2 ppbv of each analyte.listed in Table 1.

3. Allow PNL WTVP staff up to two visits to the BCL laboratory after PNL receives their own equipment for TO-14. PNL will notify BCL of the site visit dates in writing not less than 2 weeks prior to the visit dates. The site visits will include, but is not limited to, contact with the individuals named in item I.1, and will cover, but will not be limited to discussion of topics specified below:

   - modifying TO-14 to analyze (specific) polar compounds, including any additional QA requirements for laboratory validation of non-method TO-14 compounds;
   - disclosure of method(s) for making polar standards in summa canisters and disclosure of relevant standard storage protocoles, to include temperature, humidity, and shelf-life of such standards;
   - techniques for removal of water prior to analyzing for polar compounds, and techniques for splitting samples from canisters onto thermal desorption tubes.
   - analytical considerations affecting the GC/MS analysis and detection limits of volatile polar compounds.

   One of the site visits will also include a demonstration of the analysis of a summa canister sample containing polar compounds, by modified TO-14 method, to demonstrate capability in this area should PNL require an outside laboratory to validate summa canister standards containing volatile polar compounds.

4. BCL shall provide a schedule of expenses detailing the consultation costs incurred by BCL for each site visit and which directly pertain and the information exchange related to EPA compendium method TO-14 provided to PNL staff.

II. Pacific Northwest Laboratories, (PNL) staff working on WTVP shall:

1. Visit the BCL laboratory dedicated for summa canister analysis on a mutually agreed upon date. This initial visit to the BCL laboratory will include other Hanford scientists working on the WTVP, and will be arranged by PNL. Specific tasks associated with this action will include:

   - PNL staff to notify BCL by letter of the proposed site visit and will list specific topics for discussion and deliverables required upon visit to BCL;

- PNL staff will cover travel and expense costs for 2 PNL staff members and the PNL WTVP project manager (if required) working on the WTVP during their visit the BCL site. BCL consultation and presentation costs will be covered by PNL as quoted by BCL.

2. Send summa canisters (TO-14 standards and blanks combined) to BCL for analysis of target analytes listed in Table 1 by GC/MS, at blank and calibration standard levels for each analyte listed in Table 1. BCL must meet the requirements of I.2 of this SOW before PNL will use BCL results to validate both standards making capability and canister cleaning methods used by PNL, and to determine that laboratory is ready to perform TO-14 analysis.

3. Make up to two visits to BCL after PNL receives their equipment, and PNL staff have had the opportunity to attempt TO-14 methodology at PNL. The site visits will also address problems associated with modifying TO-14 for analytes suspected in tank 103C. Action items for these visits will include but are not limited to:

    - notifying BCL in writing, of the specific dates chosen for the visits within 2 weeks of the date specified;
    - discussion of items listed in section I.3. of this SOW;
    - observation of BCL demonstration of analysis of summa canister samples containing polar compounds using modified a TO-14 method.

4. Provide the payment mechanism to cover consultation costs incurred by BCL for each site visit and the exchange of information pertaining to EPA method TO-14 as specified by this SOW. Payment will consist of fund transfers taking place within 30 days after receipt of invoices from BCL. Payment for sample validation will consist of fund transfers taking place after PNL reviews and accepts the analysis data provided that BCL has invoiced PNL for the services provided. PNL shall be allowed 60 days from receipt of the data to review and accept the analytical results before the PNL project manager will approve payment for the services provided.

INTERLABORATORY AUTHORIZATION 193771-A-H1, SUPPLEMENT NO. 1

Supplement For: Provide for Additional Services and Increase the Estimate of Cost/Maximum Obligation

Battelle

I. This document is not a contract. It is a modification to an Interlaboratory Authorization (ILA) issued by PACIFIC NORTHWEST LABORATORIES, BATTELLE MEMORIAL INSTITUTE (PNL), pursuant to Contract DE-AC06-76RLO 1830 with the U.S. Department of Energy (DOE) to BATTELLE COLUMBUS OPERATIONS, BATTELLE MEMORIAL INSTITUTE (BCO) for the purpose of acquiring further needed services in accordance with BMI policy.

II. STATEMENT OF WORK: BCO shall perform the work set forth in Attachment B, as revised June 18, 1993.

III. PERIOD OF PERFORMANCE: No change.

IV. KEY PERSONNEL: No change.

V. ESTIMATE OF COST/MAXIMUM OBLIGATION: The estimated cost and maximum obligation of the work under this ILA is $23,001 ($9,914 increase). This amount is maximum and shall not be exceeded without the prior written approval of PNL Subcontracts. Facilities Cost of Capital Money (FCCOM) is not an allowable cost under this ILA. No fixed fee is included in this ILA.

INTERIM INDIRECT COST RATES: The following indirect cost rates shall be used for invoicing:

Direct Labor Fringe	41%
Indirect Costs Overhead	87%
G&A	20%

INDIRECT COST RATE CEILINGS: No reimbursement shall be made for any amount of indirect costs in excess of the rates specified below; Provided, however, that in the event the final indirect cost rates are less, reimbursement shall be at such lower rates:

Total Overhead	158%

VI. BILLING INSTRUCTIONS: No change.

PACIFIC NORTHWEST LABORATORIES
BATTELLE MEMORIAL INSTITUTE

By Ellen McCoy
Ellen McCoy
Subcontract Specialist

Date June 21, 1993

BATTELLE COLUMBUS OPERATIONS
BATTELLE MEMORIAL INSTITUTE

By H Michael Sewell

Title CONTRACTING OFFICER

Date 7/19/93

H. MICHAEL SEWELL
CONTRACTING OFFICER

ATTACHMENT B

Revised June 18, 1993

ILA 193771-A-H1

STATEMENT OF WORK

This ILA covers an exchange of technical information between PNL and BCO pertaining to the analysis of passivated canister samples. The exchange will involve three separate visits of PNL staff to BCO as well as analysis of canister samples provided to BCO by PNL. The work is in furtherance of Task 03, organic analysis for Phase I characterization of the Waste Tank Vapor Project (WTVP). In particular, information pertaining to the analysis of summa canister samples by GC/MS is of importance to the WTVP because summa canister samples are part of the Phase I sampling scheme proposed by the Hanford Vapor Team. Task 03 will use EPA Compendium Method TO-14 as a model. This Statement of Work describes the information necessary to train PNL staff to use the TO-14 method and the requirements for BCO to perform as a referee laboratory to run standards for PNL. Specific actions required by BCO and PNL are detailed below:

I. BCO staff performing summa canister analyses as per EPA Compendium Method TO-14 shall:

   1. Allow PNL staff working Task 03 of the WTVP to the BCO laboratory dedicated to summa canister analysis and to discuss with PNL staff specific items pertaining to EPA Compendium Method TO-14, as specified below. This visit will include:

      - Contact with BCO staff, Robert Coutant, Budget Manager, Debra L. Smith, Research, and Michael W. Holdren, Researcher;

      - Disclosure by BCO staff of specific requirements for implementing and validating TO-14 by PNL project scientist and any recommendations for demonstrating laboratory TO-14 analysis capability including instrument system control;

      - Disclosure by BCO staff of the procedures and protocols used to limit known interferences and which describe requirements and recommendations relating to the cleanliness of the proposed laboratory to do TO-14 analysis. Procedures should be supported by data which demonstrates that detection limits of 0.2 ppbv per analyte (in the TO-14) method are achieved;

      - Disclosure by BCO staff of specific requirements for making in-house TO-14 standards and relevant standard storage protocols to include temperature and humidity conditions, and records of shelf-life stability studies for such standards;

      - Disclosure by BCO staff of method(s) for cleaning and evacuating summa canisters, including QA criteria, labeling, and chain-of custody procedures.

- (Revised June 18, 1993). Two (2) BCO staff members shall visit PNL for the purpose of exchanging technical information. The visit is tentatively scheduled for July 6 through July 9, 1993.

2. Analyze up to ten (10) summa canisters (TO-14 standards and blanks combined) supplied by PNL, to verify PNL's standard making and canister cleaning capability. The target analytes with required limits of quantitation for each analyte are listed in Table 1. BCO must demonstrate that the GC/MS system used is clean as specified in TO-14, by showing that the system used is capable of detecting 0.2 ppbv of each analyte listed in Table 1.

3. Allow PNL WTVP staff to visit the BCO laboratory 1-2 months after PNL receives their own equipment for TO-14. PNL will notify BCO of the site visit date in writing not less than 2 weeks prior to the visit date. The site visit will include, but not be limited to, contact with the individuals named in "1" above, and will cover, but not be limited to, discussion of topics specified below:

   - Modifying TO-14 to analyze (specific) polar compounds, including any additional QA requirements for laboratory validation of non-method TO-14 compounds;
   - Disclosure of method(s) for making polar standards in summa canisters and disclosure of relevant standard storage protocols, to include temperature, humidity and shelf-life of such standards;
   - Techniques for removal of water prior to analyzing for polar compounds, and techniques for splitting samples from canisters onto thermal desorption tubes.
   - Analytical considerations affecting the GC/MS analysis and detection limits of volatile polar compounds.

   This visit will also include a demonstration of the analysis of a summa canister sample containing polar compounds, by modified TO-14 method, to demonstrate capability in this area should PNL require an outside laboratory to validate summa canister standards containing volatile polar compounds.

4. At completion of the project, BCO will provide a letter report containing the information exchanged during the visits as well as the consultation costs.

II. PNL staff working on WTVP shall:

1. Visit the BCO laboratory dedicated for summa canister analysis on a mutually agreed upon date. Specific tasks associated with this action will include:

   - PNL staff to notify BCO by letter of the proposed site visit and listing specific topics for discussion and deliverables required upon visit to BCO;

Appendix D

Compilation of Methods for Air Toxic Analysis

The following attached list of methods for air toxic analysis is provided for reference. This list is not complete and is an ongoing task.

Agency	Method No.	Title
EPA	IP-1A	Determination of Volatile Organic Compounds (VOC) in Indoor Air, Stainless Steel Canister
EPA	IP-1B	Determination of Volatile Organic Compounds (VOCs) in Indoor Air, Solid Adsorbent Tubes
EPA	IP-2A	Determination of Nicotine in Indoor air XAD-4 Sorbent Tubes
EPA	IP-2B	Determination of Nicotine in Indoor Air Treated Filter Cassette
EPA	IP-3A	Determination of Carbon Monoxide (CO) or Carbon Dioxide (CO_2) in Indoor Air Nondispersive INfrared (NDIR)
EPA	IP-3B	Determination of Carbon Monoxide (CO) or Carbon Dioxide (CO_2) in Indoor Air Gas Filter Correlation (GC)
EPA	IP-3C	Determination of Carbon Monoxide (CO) or Carbon Dioxide (CO_2) in Indoor Air Electrochemical Oxidation
EPA	IP-4A	Determination of Air Exchange Rate in Indoor Air Perfluorocarbon Tracer (PFT)
EPA	IP-4B	Determination of Air Exchange Rate in Indoor Air Tracer Gas
EPA	IP-5A	Determination of Nitrogen Dioxide in Indoor Air Continuous Luminox Monitor
EPA	IP-5B	Determination of Nitrogen Dioxide in Indoor Air Palmes Diffusion Tube
EPA	IP-5C	Determination of Nitrogen Dioxide in Indoor Air Passive Sampling Device
EPA	IP-6A	Determination of Formaldehyde and Other Aldehydes in Indoor Air Solid Adsorbent Cartridge
EPA	IP-6B	Determination of Formaldehyde and Other Aldehydes in Indoor Air Continuous Colorimetric Analyzer
EPA	IP-6C	Determination of Formaldehyde and Other Aldehydes in Indoor Air Passive Sampling Device
EPA	IP-7	Determination of Benzo(a)Pyrene [B(a)P] and Other Polyunuclear Aromatic Hydrocarbons (PAHs) in Indoor Air

Agency	Method No.	Title
EPA	IP-8	Determination of Organochlorine Pesticides in Indoor Air
EPA	IP-9	Determination of Reactive Acidic and Basic Gases and Particulate Matter in Indoor Air
EPA	TO-1	Determination of Volatile Organic Compounds in Ambient Air using Tenax Adsorption and Gas Chromatograph (GC/MS)
EPA	TO-10	Determination of Organochlorine Pesticides in Ambient Air using Low Volume Polyurethane Foam (PUF) Sampling with Gas Chroamtography/Electron Capture-Detector (GC/ECD)
EPA	TO-11	Determination of Formaldehyde in Ambient Air using Adsorbent Cartridge Followed by High Performance Liquid Chromatography (HPLC)
EPA	TO-12	Determination of Non-methane Organic Compounds (NMOC) in Ambient Air using Cryogenic Preconcentration and Direct Flame Ionization Detectrion (PDFID)
EPA	TO-13	Determination of Polynuclear Aromatic Hydrocarbons (PAHs) in Ambient Air using High Volume Sampling with Gas Chromatography/Mass Spectrometry (GC/MS) and High Resolution Liquid Chromatography
EPA	TO-14	Determination of Volatile Organic Compounds (VOCs) in Ambient Air using SUMMA® Polished Canister Sampling and Gas Chromatographic (GC) Analysis
EPA	TO-2	Determination of Volatile Organic Compounds in Ambient Air by Carbon Molecular Sieve Adsorption and Gas Chromatography/Mass Spectrometry (GC/MS)
EPA	TO-3	Determination of Volatile Organic Compounds in Ambient Air using Cryogenic Preconcentration Techniques and Gas Chromatography with Flame Ionization and Electron Caputre Detection
EPA	TO-4	Determination of Organochlorine Pesticides and Polychlorinated Biphenyuls in Ambient Air
EPA	TO-5	Determination of Aldehydes and Ketones in Ambient Air using High Performance Liquid Chromatography (HPLC)
EPA	TO-6	Determination of Phosgene in Ambient Air using High Performance Liquid Chromatography (HPLC)
EPA	TO-7	Determination of N-Nitrosodimethylamine in Ambient Air using Gas Chromatography
EPA	TO-8	Determination of Phenol and Methylphenols (Cresols) in Ambient Air using High Performance Liquid Chromatography (HPLC)
EPA	TO-9	Determination of Polychlorinated Dibenzo-p-Dioxins (PCDDs) in Ambient Air using High-Resolution Gas Chromatography/High-Resolution Mass Spectrometry

Agency	Method No.	Title
ICMARA	101	Determination of C_1 through C_5 Atmospheric Hydrocarbons
ICMARA	102	Separation and Determination of Polynuclear Aromatic Hydrocarbons and Benzo[a]pyrene
ICMARA	102A	Extraction and Cleanup Procedures for Polynuclear Aromatic Hydrocarbons in Atmospheric Particulate Matter
ICMARA	102B	Separation and Microanalysis of Airborne Particulate Matter for Benzo[a]pyrene using Thin Layer Chromatography and Spectrofluorimetry
ICMARA	102C	Measurement of Benzo[a]pyrene and benzo[k]fluoranthene by Spectrofluorimetry
ICMARA	102D	Measurement of Polynuclear Aromatic Hydrocarbgons using Liquid Chromatography with Fluorescence Detection
ICMARA	108	Continuous Determination of Total Hydrocarbons in the Atmosphere (Flame Ionization Method)
ICMARA	109	Flame Ionization Detector
ICMARA	114	Determination of Acrolein Content of the Atmosphere (Colorimetric)
ICMARA	116	Determination of Formaldehyde Content of the Atmosphere (Colorimetric Method)
ICMARA	117	Determination of Formaldehyde Content of the Atmosphere (MBTH Colorimetric Method—Application to Other Aldehydes)
ICMARA	118	Determination of Mercaptain Content of the Atmosphere
ICMARA	121	Determination of Phenols in the Atmosphere (Gas Chraomtographic Method)
ICMARA	122	Determination of C_1-C_5 Aldehydes in Ambient Air and Source Emissions as 2, 4-Dinitrophenylhydrazones by HPLC
ICMARA	128	Determination of Continuous Carbon Monoxide Content of the Atmosphere (Nondispersive Infrared Method)
ICMARA	133	Determination of O_2, N_2, CO, CO_2, and CH4 (Gas Chromatographic Method)

Agency	Method No.	Title
ICMARA	134	Constant Pressure Volumetric Gas Analysis for Oxygen, Carbon Dioxide, Carbon Monoxide, and Nitorgen (Orsat)
ICMARA	135	Determination of Volatile Organic Compounds in Architectural and Industrial Surface Coatings
ICMARA	201	Chloride Content of the Atmosphere
ICMARA	202	Determination of Free Chlorine Content of the Atmosphere (Methyl Organe Method)
ICMARA	203	Determination of Fluoride Content of the Atmosphere and Plant Tissues (Manual Methods)
ICMARA	204	Determination of Fluoride Content of the Atmosphere and Plant Tissues (Semiautomated Method)
ICMARA	205	Determination of Fluoride Content of Plant Tissues (Potentiometric Method)
ICMARA	206	Determination of Gaseous and Particulate Fluorides in the Atmosphere (Separation and Collection with sodium Bicarbonate Coated Glass Tube and Particulate Filter)
ICMARA	207	Determination of Gaseous and Particulate Fluorides in the Atmosphere (Separation and Collection with a Double Paper Tape Sampler)
ICMARA	301	Determination of Particulate Antimony Content of the Atmosphere
ICMARA	302	Determination of Arsenic Content of Atmospheric Particulate Matter
ICMARA	303A	General Method for the Preparation of Non-Tissue Environmental Samples for the Trace Metal Analysis
ICMARA	311-322	Determination of a Number of Metals in Atmsopheric Particulate Matter
ICMARA	317	Determination of LElemental Mercury in Ambeitn and Workroom Air by Collection on Silver Wool and Atomic Absorption Spectroscopy
ICMARA	319	Determination of Molybdenum Content of Atmospheric Particulate Matter by Atomic Absorption Spectrophotometry
ICMARA	401	Determination of Ammonia in the Atmosphere (Indophenol Method)

Agency	Method No.	Title
ICMARA	404	Determination of Nitrate in Atmospheric Particulate Matter (Brucine Method)
ICMARA	405	Determination of Nitric Oxide Content of the Atmosphere
ICMARA	406	Determination of Nitrogen Dioxide Content of the Atmosphere (Gries-Saltzman Reaction)
ICMARA	407	Determination of LTotal Nitrogen Oxides as Nitrate (Phenoldisulphonic Acid Method)
ICMARA	408	Analysis for Atmospheric Nitrogen Dioxide (24-H Average)
ICMARA	411	Determination of Oxidzing Substances in the Atmosphere
ICMARA	413	Determinalin of Ozone in the Atmosphere by Gas-Phase chemiluminescence Instruments
ICMARA	415	Determination of Atmospheric Nitric Acid
ICMARA	416	Continuous Monitoring of Atmospheric Nitric Oxide and Nitrogen Dioxide by Chemiluminescence
ICMARA	417	Continuous Monitoring of Ozone In the Atmosphere by Ultraviolet Photometric Instruments
ICMARA	501	High Volume Measurement of Size Classified Particulate Matter
ICMARA	502	Particle Fallout Container Measurement of Dustfall from the Atmosphere
ICMARA	503	Continuos Tape Sampling of Coefficient of Haze
ICMARA	507	Integrating Nephelometer Measurement of Scattering Coefficient and Fine Particle Concentrations
ICMARA	601	Determination of Gross Alpha Radioactivity Content of the Atmosphere
ICMARA	602	Determination of Gross Beta Radioactivity Content of the Atmosphere

ICMARA	603	Determination of the Iodine-131 Content of the Atmosphere (Particulate Filter Charcoal Gamma)
ICMARA	606	Determination of Radon-222 Content of the Atmosphere
ICMARA	609A	Determination of Tritium Content of Water Vapor in the Atmosphere
ICMARA	609B	Determination of Elemental Tritium Content of the Atmosphere
ICMARA	700	Determination of Hydrogen Sulfide Content of the Atmosphere
ICMARA	704A	Determination of Sulfur Dioxide Content of the Atmosphere (Tetrachloromercurate Absorber/Pararosaniline Method)
ICMARA	704B	Determination of Sulfur Dioxide Content of the LAtmosphere (Formaldehyde Absorber/Pararisabukube Netgid(
ICMARA	704C	Determination of Sulfur Dioxide Content of the Atmosphere (Hydrogen Peroxide Method)
ICMARA	707	Continuous Monitoring of Atmospheric Sulfur Dioxide with Amperometric Instruments
ICMARA	708	Determination of Mercaptan Content of the Atmosphere
ICMARA	709	Determination of Sulfur-Containing Gases in the Atmosphere (Continuous Method with Flame Photometric Detector)
ICMARA	709A	Determination of Sulfur-Containing Gases in the Atmosphere (Following Chromatographic-Separation, with the FPD)
ICMARA	709B	Determination of Sulfur-Containing Gases in the Atmosphere (Total Gaseous Sulfur with the FPD)
ICMARA	711	Determination of Gaseous Sulfuric Acid and Sulfur Dioxide in Stack Gases
ICMARA	713	Semi-Continuous Determination of Atmospheric Particulate Sulfur, Slulfuric Acid and ammonium Sulfates
ICMARA	714	Determination of Sulfur Dioxide kEmissions in Stack Gases by Pulsed Fluorescence

Agency	Method No.	Title
ICMARA	720A	Suppressed Anion Chromatography
ICMARA	720B	Nonsuppressed Anion Chromatography
ICMARA	720C	Flow Injection Determation of Aqueous Sulfate (Methylthymol Blue Method)
ICMARA	720D	Barium Sulfate Turbidimetry
ICMARA	720E	Barium Perchlorate Microtitration
ICMARA	720F	Barium Chloranilate Spectophotometry
ICMARA	730	Determination of Sulfur in Particulate Material by X-Ray Fluorescence
ICMARA	801	Determination of Ammonia in Air
ICMARA	804	As, Se, and Sb in Urine and Air by Hybride Generation and Atomic Absorption Spectrometry
ICMARA	805	Determination of Chnloride in Air
ICMARA	806	Determination of Free Chlorine in Air
ICMARA	807	Determination of Chromic Acid Mist in Air
ICMARA	808	Determination of Cyanide in Air
ICMARA	809	Determination of Fluiorides and Hydrogen Fluoride in Air
ICMARA	810	Determination of Gaseous and Particulate Fluiorides in Air
ICMARA	811	Determination of Fluoride in Urine

Agency	Method No.	Title
ICMARA	812	Determination of Hydrogen Suilfide in Air
ICMARA	815	Determination of Mercury in Urine
ICMARA	816	Determination of Nitric Oxide in Air
ICMARA	817	Determination of Nitrogen Dioxide in Air
ICMARA	818	Determination of Nitrogen Dioxide in Air
ICMARA	819	Determination of Ozone in Air
ICMARA	821	Determination of Phosgene in Air
ICMARA	822	General Atomic kAbsorption Procedure for Trace Metals in Airborne Material Collected on Filters
ICMARA	822A	General Method for Preparation of Tissue Samples for Analysis for Trace Metals
ICMARA	822B	X-Ray Fluorescence Spectrometry for Multielement Analysis of Airborne Particulate and Biological Material
ICMARA	824	Determination of Airborne Sujlfates
ICMARA	825	Determination of Acrolein in Air
ICMARA	826	Determination of Acrolein in Air
ICMARA	827	Determination of Aromatic Amines in Air
ICMARA	828	Determination of Bis-(Chloromethyl) Ether (Bis-CME) In Air
ICMARA	829	Determination of Chloromethyl Methyl Ether (CMME) anmd Bis-Chloromethyl Ether (Bis-CME) i Air

Agency		
ICMARA	830	Determination of 3,3'-"Dichloro-4,4'-Diaminodiphenylmethane (MOCA) in Air
ICMARA	831	Determination of p,p-Diphenylmethane Diisocryanate (MDI) in Air
ICMARA	832	Determination of Nitroglycerin and Ethylene Glycol Dinitrate (Nitroglycol) in Air
ICMARA	833	Determination of N-Nitrosodimethylamine in Ambient Air
ICMARA	834	Determination of Organic Solvent Vapors in Air
ICMARA	835	Determination of EPN, Malathion and Parathion in Air
ICMARA	836	Determinatino of LTotal Particulate kAromatic Hydrocarbons (TpAH) in Air; Ultrasonic Extraction Method
ICMARA	837	Determination of 2,4-Toluenediisocryanate (TDI) in Air
NIOSH	0500	Nuisance Dust, total
NIOSH	0600	Nuisance dust, respirable
NIOSH	1000	Allyl chloride
NIOSH	1001	Methyl chloride
NIOSH	1002	Chloroprene
NIOSH	1003	Hydrocarbons, halogenated
NIOSH	1004	sym-Dichloroethyl Ether
NIOSH	1005	Methylene Chloride

Agency		
NIOSH	1006	Trichlorofluoromethane
NIOSH	1008	Ethylene Dibromide
NIOSH	1009	Vinyl Bromide
NIOSH	1010	Epichlorohydrin
NIOSH	1011	Ethyl Bromide
NIOSH	1012	Dibromodifluoromethane
NIOSH	1013	1,2-Dichloropropane
NIOSH	1014	Methyl Iodine
NIOSH	1015	Vinylidene Chloride
NIOSH	1016	1,1,1,2-Tetrachloro-2,2,-Difluoroethane and 1,1,2,2-Tetrachloro-1,2-Difluoroethane
NIOSH	1017	Bromotrifluoromethane
NIOSH	1018	Dichlorodifluoromethane and 1,2-Dichlorotetrafluoroethane
NIOSH	1019	1,1,2,2-tetrachloroethane
NIOSH	1020	1,1,2-Trichloro-1,2,2-Trifluoroethane
NIOSH	1020	1,1,2-Trichloro-1,2,2-Trifluoroethane
NIOSH	1022	Trichloroethylene

Agency	Method No.	Title
NIOSH	1024	1,3-Butadiene
NIOSH	1300	Ketones I
NIOSH	1301	Ketones II
NIOSH	1400	Alcohol I
NIOSH	1401	Alcohol II
NIOSH	1402	Alcohols III
NIOSH	1403	Alcohols IV
NIOSH	1450	Esthers I
NIOSH	1500	Hydrocarbons, BP 36-126°C
NIOSH	1550	Naphthas
NIOSH	1551	Turpentine
NIOSH	1600	Carbon Disulfide
NIOSH	1601	1,1-Dichloro-1-Nitroethane
NIOSH	1602	Dioxane
NIOSH	1603	Acetic Acid
NIOSH	1604	Acrylonitrile

Agency	Method No.	Title
NIOSH	1606	Glycidol
NIOSH	1609	Tetrahydrofuran
NIOSH	1610	Ethyl Ether
NIOSH	1611	Methylal
NIOSH	1612	Propylene Oxide
NIOSH	1613	Pyridine
NIOSH	1614	Ethylene Oxide
NIOSH	1615	Methyl tetr-Btyl Ether (Mtbe)
NIOSH	2000	Methanol
NIOSH	2001	Cresols
NIOSH	2002	Amines, Aromatic
NIOSH	2003	1,1,2,2-Tetrabromoethane
NIOSH	2004	Dimethylacetamide and Dimethylformamamide
NIOSH	2005	Nitrobenzenes
NIOSH	2007	Aminoethanol Compounds
NIOSH	2008	Chloroacetic Acid

.Agency	Method No.	Title
NIOSH	2010	Amines, Aliphatic
NIOSH	2500	2-Butanone
NIOSH	2501	Acrolein
NIOSH	2503	Mevinphos (Phosdrin)
NIOSH	2504	Tetraethyl Pyrophosphate (Tepp)
NIOSH	2505	Furfuryl Alcohol
NIOSH	2506	Acetone Cyanohydrin
NIOSH	2507	Nitroglycerin/egon
NIOSH	2508	Isophorone
NIOSH	2510	1-Octanethiol
NIOSH	2513	Ethylene Chlorohydrin
NIOSH	2514	Anisidine
NIOSH	2515	Diazomethane
NIOSH	2516	Dichlorofluoromethane
NIOSH	2517	Pentachloroethane
NIOSH	2518	Hexachloro-1,3-cyclopentadine

Agency	Method No.	Title
NIOSH	2519	Ethyl chloride
NIOSH	2520	Methyl Bromide
NIOSH	2521	Methyl Cyclohexanone
NIOSH	2522	Nitrosamines
NIOSH	2523	1,3-cyclopentadiene
NIOSH	2524	Dimethyl Sulfate
NIOSH	2525	1-Butanethiol
NIOSH	2526	Nitroethane
NIOSH	2527	Nitromethane
NIOSH	2528	2-Nitropropane
NIOSH	2529	Furfural
NIOSH	2530	Biphenyl
NIOSH	2531	Glutaraldehyde
NIOSH	2533	Tetraethyl Lead
NIOSH	2534	Tetramethyl Lead
NIOSH	2535	Toluene-2,4-Diisocyanate

Agency	Method No.	Title
NIOSH	2536	Valeraldehyde
NIOSH	2537	Methyl Methacrylate
NIOSH	2538	Acetaldehyde
NIOSH	25392540	Aldehydes, screeningDiethylenetriamine, Ethylenediamine, & Triethylenetetramine
NIOSH	2541	Formaldehyde
NIOSH	3500	Formaldehyde
NIOSH	3501	Formaldehyde
NIOSH	3502	Phenol
NIOSH	3503	Hydrazine
NIOSH	3505	Tetramethyl Thiourea
NIOSH	3506	Acetic Anhydride
NIOSH	3507	Acetaldehyde (HPLC)
NIOSH	3508	Methyl Ethyl Ketone Peroxide
NIOSH	3509	Aminoethanol Compounds II
NIOSH	3700	Benzene
NIOSH	3701	Trichloroethylene

NIOSH	3702	Ethylene Oxide
NIOSH	4000	Toluene
NIOSH	5000	Carbon Black
NIOSH	5001	2,4-D and 2,4,5-T
NIOSH	5002	Warfarin
NIOSH	5003	Paraquat
NIOSH	5004	Hydroquinone
NIOSH	5005	Thiram
NIOSH	5006	Carbaryl
NIOSH	5007	Rotenone
NIOSH	5008	Pyrethrum
NIOSH	5009	Benzoyl Peroxide
NIOSH	5010	Bromoxynil and Bromoxynil Octanoate
NIOSH	5011	Ethylene Thiourea
NIOSH	5012	EPN, Malathion and Parathin
NIOSH	5013	Dyes, Benzidine, Q-Anisidine, and kq-Tolidine

Agency	Method No.	Title
NIOSH	5014	Chlorinated Terphenyl
NIOSH	5016	Strychnine
NIOSH	5017	Dibutyl Phosphate
NIOSH	5018	2,4,7-Trinitrofluoren-9-One
NIOSH	5019	Azelaic Acid
NIOSH	5020	Dibutyl Phthalate and Di(2-Ethylhexyl) Phthalate
NIOSH	5021	o-Terphenyl
NIOSH	5022	Arsenic, Organo-
NIOSH	5023	Coal Tar Pitch Volatiles
NIOSH	5025	Chlorinated Diphenyl Ether
NIOSH	5026	Mineral Oil Mist
NIOSH	5027	Ribavirin
NIOSH	5029	4,4'-Methylenedianiline
NIOSH	5030	Cyanuric Acid
NIOSH	5031	Asparatame
NIOSH	5500	Ethylene Glycol

Agency	Method No.	Title
NIOSH	5502	Aldrin and Lindane
NIOSH	5503	Polychorobiphenyls
NIOSH	5504	Organotin Compunds
NIOSH	5506	Polynuclear Aromatic Hydrocarbons (HPLC)
NIOSH	5508	Kepone
NIOSH	5509	Benzidine and 3,3-Dichlorobenzidine
NIOSH	5510	Chlordane
NIOSH	5512	Pentachlorophenol
NIOSH	5514	Demeton
NIOSH	5515	Polynuclear Aromatic Hydrocarbons
NIOSH	5516	2,4-&,2,6-Toluenediamine
NIOSH	5517	Polychlorobenzenes
NIOSH	5518	Naphthylamines
NIOSH	5519	Endrin
NIOSH	5521	Isocyanates
NIOSH	6004	Sulfur Dioxide

Agency	Method No.	Title
NIOSH	6005	Iodine
NIOSH	6006	Diborane
NIOSH	6007	Nickel Carbonyl
NIOSH	6008	Stibine
NIOSH	6009	Mercury
NIOSH	6010	Hydrogen Cyanide
NIOSH	6011	Bromine and Chlorine
NIOSH	6402	Phosphorus Trichloride
NIOSH	6600	Nitrous Oxide
NIOSH	6601	Arsine
NIOSH	6601	Oxygen
NIOSH	6700	Nitrogen Dioxide
NIOSH	6701	Ammonia
NIOSH	7013	Aluminum
NIOSH	7020	Clacium
NIOSH	7024	Chromium

Agency	Method No.	Title
NIOSH	7027	Cobalt
NIOSH	7029	Copper
NIOSH	7030	Zinc
NIOSH	7048	Cadmium
NIOSH	7056	Barium, soluble compounds
NIOSH	7074	Tungsten (soluble and insoluble)
NIOSH	7082	Lead
NIOSH	7102	Beryllium
NIOSH	7105	Lead
NIOSH	7200	Welding and Brazing Fume
NIOSH	7300	Elements (ICP)
NIOSH	7400	Fibers
NIOSH	7401	Alkaline dusts
NIOSH	7402	Asbestos Fibers
NIOSH	7500	Silica
NIOSH	7501	Silica, Amorphous

Agency	[illegible]	[illegible]
NIOSH	7502	Zinc Oxide
NIOSH	7504	Vanadium Oxides
NIOSH	7505	Lead Sulfide
NIOSH	7506	Boron Carbide
NIOSH	7600	Chromium Hexavalent
NIOSH	7601	Silica, Crystalline
NIOSH	7602	Silica, Crystalline
NIOSH	7603	Quartz
NIOSH	7604	Chromium, Hexavalent
NIOSH	7900	Aresenic
NIOSH	7901	Arsenic Trioxide
NIOSH	7902	Fluorides
NIOSH	7903	Acids, Inorganic
NIOSH	7904	Cyanides
NIOSH	7905	Phosphorus
NIOSH	8000	Alad

Agency	Method No.	Title
NIOSH	8001	Pentachlorophenol
NIOSH	8002	2-Butanone, Ethanol, and Toluene in blood
NIOSH	8003	Lead in blood and urine
NIOSH	8004	Polychlorinated Biphenyls in serum
NIOSH	8005	Elements in blood or tissue
NIOSH	8300	Hippuric Acid in urine
NIOSH	8301	Hippuric and Methyl Hippuric Acids in urine
NIOSH	8302	MBOCA in urine
NIOSH	8303	Pentachlorophenol in urine
NIOSH	8304	Benzidine in urine
NIOSH	8305	Phenol and q-Cresol in urine
NIOSH	8306	Fluoride in urine
NIOSH	8308	Benzidine in urine
NIOSH	8310	Metals in urine
NIOSH	9000	Chrysotile Asbestos
NIOSH	9002	Asbestos

Agency	Method No.	Title
NIOSH	Vinyl Chloride	
OSHA	1	Cyclohexanone
OSHA	10	Bis(chloromethyl) ether (BCME) Chloromethyl methyl ether (CMME0
OSHA	11	1,1,2-Trichloroethane
OSHA	12	Benzene
OSHA	13	Diethylnitrosamine
OSHA	14	1,1,1-Trichloroethane (Methyl chloroform)
OSHA	15	2-Nitropropane
OSHA	16	2-Butanone (MEK)
OSHA	17	N-Nitrosomorpholine
OSHA	18	Toluene 2,4-diisocyanate (TDI) Methylensebis(4-phenyl isocyanate) (MDI)
OSHA	19	Vinylidene chloride
OSHA	2	Ethylene dibromide
OSHA	20	Hydrazine
OSHA	21	Acrylamide
OSHA	22	Diphenylamine

Agency	Method No.	Title
OSHA	23	Diphenylnitrosamine
OSHA	24	4,4'-Methylenebis(o-chloroaniline) (MOCA)
OSHA	25	Maleic anhydride
OSHA	26	Methyl mercaptain
OSHA	27	N-Nitrosodimethylamine, N-Nitrosodiethylamine, N-Nitrosodi-n-propylamine, N-Nitrosodi-n-buylmaine, N-Nitrosopieridine, N-Nitrosopyrrolidine, N-Nitrosomorpholine
OSHA	28	Acrylic acid
OSHA	29	Enfluarane, Halothane
OSHA	3	Ethylene dichloride
OSHA	30	Ethylene oxide
OSHA	31	N-Nitrosodiethanolamine
OSHA	32	Phenol, Cresol
OSHA	33	The Confirmation of the Urea Derivatives of MDI and TDI by TEA
OSHA	34	Dimethylamine
OSHA	35	Naphthalene
OSHA	36	Ethylamine
OSHA	37	Acrylonitrile

Agency	Method No.	Title
OSHA	38	N-Nitrosomethylethylamine, N-Nitrosodiisorpropylamine, N-Nitrosomethyl-n-butylamine, N-Nitrosoethyl-n-butylamine, N-Nitrosodiamylamine
OSHA	39	Pentachlorophenol
OSHA	4	Vinyl chloride
OSHA	40	Pentachlorophenol
OSHA	41	Diethylamine
OSHA	42	Toluene 2,6-dissocyanate (TDI) 1,6-Hexamethylene diisocyanate (HDI) Toluene 2,4-diisocyanate
OSHA	43	Ethylene glycol dinirate (EGDN) Nitroglycerin
OSHA	44	2,4-Dinitrotoluene (DNT) 2,4,6-Trinitrotoluene (TNT)
OSHA	45	2,3,4,6-Tetrachlorophenol
OSHA	46	1-Nitropropane 2-Nitroproane
OSHA	47	Methylenebis(4-phenyl isocyanate) (MDI)
OSHA	48	Petroleum distillate fractions
OSHA	49	Ethylene oxide (passive monitor)
OSHA	5	Chloroform
OSHA	50	Ethylene oxide (adsorbent tube)
OSHA	51	Vinyl acetate

Agency	Method No.	Title
OSHA	52	Formaldehyde
OSHA	53	2-Methoxyethanol 2-Methoxyethyl acetate 2-Ethoxyethanol 2-Ethoxyethyl acetate
OSHA	54	Methyl isocyanate
OSHA	55	Methyl 2-cyanoacrylate Ethyl 2-cyanoacrylate
OSHA	56	1,3-Butadiene
OSHA	57	4,4'-Methylenedianiline
OSHA	58	Coal tar pitch volatiles and coke oven emissions Phenanthrene Anthracene Pyrene Chrysene Benzo(α)pyrene
OSHA	59	Methylene chloride
OSHA	6	Dimethylnitrosamine
OSHA	60	Ethylenediamine Diethylenetriamine Triethylenetetramine
OSHA	61	Phosgene
OSHA	62	Chloropyrifos (Dursban) DDVP (Dichlorvos) Diazion Malathion Parathion
OSHA	63	Carbaryl
OSHA	64	Glutaraldehyde
OSHA	65	Benzidine 3,3'-Dichlorobenzidine 2,4-Toluenediamine 2,6-Toluenediamine
OSHA	66	Dimethylformamide

Agency	Method No.	Title
OSHA	67	Chlordane
OSHA	68	Acetaldehyde
OSHA	69	Acetone
OSHA	7	Organic solvents
OSHA	70	Pyrethrum
OSHA	71	o-Dianisidine 4,4'-Methylenebis(o-chloroaniline) (MOCA) o-Tolidine
OSHA	72	Furfural
OSHA	73	o-m-,p-Toluidine
OSHA	74	Aldicarb
OSHA	75	Vinyl Chloride
OSHA	76	Chloroacetaldehyde
OSHA	77	MEK peroxide
OSHA	78	Diphenylamine N-Isopropylaniline
OSHA	79	2-Methoxyethanol 2-Methoxyethyl acetate 2-Ethoxyethanol 2-Ethoxyethyl acetate
OSHA	8	Vinyl bromide
OSHA	80	Methylene chloride

Agency	Method No.	Title
OSHA	9	Styrene
OSHA	ID-101	Chlorine in Workplace Atmospheres and Backup Report
OSHA	ID-103	Hexavalent Chromium and Backup Report
OSHA	ID-104	Sulfur Dioxide in Workplace Atmospheres - Bubbler and Backup Report
OSHA	ID-105	Arsenic in Workplace Atmospheres and Backup Report
OSHA	ID-108	Bromine in Workplace Atmospheres and Backup Report
OSHA	ID-110	Fluoride (F- and HF) in Workplace Atmospheres
OSHA	ID-121	Metal and Metalloid Particulates in Workplace Atmospheres (Atomic Absorption)
OSHA	ID-125G	Metal and Metalloid Particulates in Workplace Atmospheres (ICP Analysis) and Backup Report
OSHA	ID-140	Mercury Vapor in Workplace Atmospheres and Backup Report
OSHA	ID-141	Hydrogen Sulfide in Workplace Atmospheres and Backup Report
OSHA	ID-142	Quartz and Cristobalite in Workplace Atmospheres
OSHA	ID-143	Zinc Oxide in Workplace Atmospheres
OSHA	ID-145	Particulate Mercury in Workplace Atmospheres
OSHA	ID-160	Asbestos in Air
OSHA	ID-172	Carbon Dioxide in Workplace Atmospheres and Backup Report

Agency	Method No.	Title
OSHA	ID-177	Iodine in Workplace Atmospheres and Backup Report
OSHA	ID-180	Phosphine in Workplace Atmospheres and Backup Report
OSHA	ID-182	Nitrogen Dioxide in Workplace Atmospheres and Backup Report
OSHA	ID-185	Confirmation of Vanadium Pentoxide in Workplace Atmospheres and Backup Report
OSHA	ID-188	Ammonia in workplace Atmospheres - Solid Sorbent and Backup Report
OSHA	ID-190	Nitric Oxide in Workplace Atmospheres and Backup Report
OSHA	ID-202	Chlorine Dioxide in Workplace Atmospheres and Backup Report
OSHA	ID-204	Qualitative X-Ray Fluorescence Analysis of Workplace Substances
OSHA	ID-205	Formaldehyde in Workplace Atmospheres (3M Model 3721 Monitor) and Product Evaluation
OSHA	ID-206	ICP Analysis of Metal/Metalloid Particulates from Solder Operations and Backup Report
OSHA	ID-207	Portland Cement (Total Dust) in Workplace Atmospheres
OSHA	ID-210	Carbon Monoxide in Workplace Atmospheres

Appendix E

Capital Procurement of GC/MS System

Battelle

Pacific Northwest Laboratories
5th & W Streets
P.O. Box 999
Richland, Washington 99352

2410A3141

IMPORTANT

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BUYER: RICK DENNIS	TELEPHONE: (509) 375-2348	DELIVERY DATE: 03/22/93
TERMS: NET 30 DAYS	SHIP VIA:	FOB: RICHLAND, WA

SELLER	SHIP TO	BILL TO
HEWLETT PACKARD ANALYTICAL DIVISION 1421 S MANHATTAN AVENUE FULLERTON CA 92631	Battelle For U.S. DOE c/o Westinghouse, 1163 Bldg Richland, WA 99352	BATTELLE ACCOUNTS PAYABLE PO BOX 999 RICHLAND, WA 99352 REF: ORDER NO. ABOVE (ATTACH BILL OF LADING OR PAID FREIGHT BILL.)

ITEM	QTY	U/M	DESCRIPTION	UNIT PRICE	TOTAL PRICE
			THIS CONFIRMS OUR ORDER PLACED WITH: MARK SASSI ON: 01/28/93		
1	1	EA	SPECTROMETER GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM TO BE CONFIGURED IN ACCORDANCE WITH HEWLETT PACKARD QUOTATION NUMBER 1111-78373.	67,894.90	67,894.90
2	1	LOT	INSTALLATION TWO DAYS ONSITE	2,200.00	2,200.00
3	1	LOT	CONSULT/TRAINING ONE DAY ONSITE	1,900.00	1,900.00
				TOTAL PRICE:	71,994.90

GSA CONTRACT AWARD

This contract is for supplies/services required in support of operating the U.S. Department of Energy's Pacific Northwest Laboratory under Contract DE-AC06-76RLO-1830. This contract is awarded pursuant to January 7, 1986, written authorization from the Department of Energy, Richland Operations Office. (A copy of this authorization is available upon request). Terms and conditions of GSA Contract No. GS-00F-5944A, Special Items 615-4, 615-6, and 615-15 shall apply. Contractor agrees not to use Battelle's name or identifying characteristics for advertising, sales promotion or other publicity purposes.

The following options and items shall be subject to open market pricing and Battelle PNL General Provisions A-287(R3) to include Clause 42, except as modified and agreed to in Attachment A by Hewlett Packard's Cliff McPhaden, Contracts Administrator, and Battelle PNL's Rick Dennis, Senior Buyer:

Option 110, Option 041, Option 043, Item 05971-60102, Item G1032C, Item 5890A W01 (2 days installation), and Item 19448A (8 hours of consulting service).

Representations and Certifications Form 369(R2) are incorporated into this purchase order.

GENERAL PROVISIONS NUMBER See above. ARE A PART OF THIS PURCHASE ORDER.	PACIFIC NORTHWEST LABORATORIES BATTELLE MEMORIAL INSTITUTE Kathleen A. Moore 1/29/93 KATHLEEN A. MOORE, MANAGER DATE OPERATIONS AND SPECIALTY PRODUCTS PROCUREMENT

TO BE CONSIDERED FOR FUTURE ORDERS . . . MEET YOUR DELIVERY COMMITMENTS!

T Clauss E.1 P8-08

Inquiry No. 193704

Options for GC/MS

Quantity	Item No.	Description
1	5890A	HP 5890 series II chromatograph
1	option 110	Capillary inlet system with electronic pressure control for split/splitless operation
1	option 116	on-column capillary inlet with electronic pressure control and temperature control programming
1	option 211	Single Flame Ionization Detector with make-up gas with adapter for 1/8" columns
1	option 201	selective detector that includes GC-MSD interface cable and capillary check-out column
1	option 039	Electronic Pressure Control board for up to two inlet options
1	option 041	split vent flow control for split/splitless inlet (mechanical or EPC versions). Provides gas savings during off hours and setting of split ratio
1	option 043	one channel of electronic pressure control General EPC
1	option L43	set of three pressure regulators with gauges
1	option 058	option to provide simultaneous cryogenic cooling of cool on column inlet and oven using liquid nitrogen
1	option 580	HPIB/RS-232C Interface
1	19243E	Packed inlet system with EPC and septum purge. Configured for 1/4" packed and series 530-u cols. Includes liners for 530u cols. To adapt for 1/8" cols, order HP19243G
1	G1035A	Wiley database of 138,000 MS spectra. For G1034A/B (DOS) software. PBM format. Uses 33-66 MB disk space. Requires 84MB or larger disk.
1	option AAD	software supplied on 3.5" 1.44 MB floppy
1	59822B	Ion gauge controller (only) for HP MSD. IG tube, fittings, installation not included
1	05971-60102	Ion source tested (EI)

Inquiry No. 193704

Quantity	Item No.	Description
1	G1201A	HP3365 ChemStation single instrument SW. Provides control, data acquisition and reduction for HP 5890A and/or HP 35900C. For use with HP vectra or other IBM PC compatible. Includes MS-DOS and Windows
1	option AAD	3 1/2 inch media option for MS-DOS and Windows/286
1	5971L	CI structured solution
1 1	G1072A M09	Chemical ionization for the 5971A MSD. Incl. CI source, interface modif., reagent flow control, & CI tune DOS (only) macros. Installation not included.
1 1	G1021A M90	High Performance ChemStation Bundle HP5971A MSD/ChemStation system w/MSD MS software, DOS, Windows, Excel, HP Vectra 486, 8 MB RAM, 430MB, 66 MHz hard disk, 3.5" floppy drive, LaserJet IV
1	G1032C	EnviroQuant target compound software
1	19448A	Installation of 5890A gas chromatograph, Electron Ionization Detector, Chemical Ionization Detector, and 1-day of on-site software training to cover MS-DOS system macro writing and use of Enviroquant software, 3-days

HEWLETT PACKARD COMPANY
SCIENTIFIC INSTRUMENTS DIVISION
5971A O.A.K.

CUSTOMER BATTELLE S/O# 248400321-001
SHIP DATE FEBRUARY 18, 1993 S/N 3304A01382

✓ o 5971A INSTRUMENT
✓ o ROUGH PUMP S/N 58318
✓ o POWER CORD 903 (INSTALLED)
✓ o LOGBOOK 05971-90004

✓ o VIDEO TAPE (05971-90001)
✓ o 5971A INTERFACE
✓ o 05971-60560 SHIP KIT

✓ o 59822B ION GAUGE CONTROLLER
S/N 6671

(ACCESSORIES)

✓ o G1072A C.I. SOURCE
S/N 3051A00630
✓ o 05970-60045

(BINDER-LOGBOOK)

✓ o EDWARDS INTERNATIONAL

✓ o DECLARATION-RETURN OF EDWARDS EQUIPMENT

HEWLETT PACKARD COMPANY

SCIENTIFIC INSTRUMENTS DIVISON
1601 CALIFORNIA AVENUE, PALO ALTO CALIFORNIA 94304

CUSTOMER: BATTELLE
SALES ORDER #248400321 001 B101
SHIP DATE:FEBRUARY 16, 1993
W. C. CODE:000

G1030A CHEMSTATION - VECTRA - 486 / 66U

G1030A - 90 DAY WARRANTY TAG S/N:3306A51245

OPTION # 140	486 / 66U VECTRA SYSTEM	
D2287A	486 / 66U VECTRA S/N:3246A02256	
D2287A # ABA	ENGLISH LOCALIZATION	
D2036A	5.25" FLEXIBLE DISK DRIVE	*(INSTALLED)*
D2381A	2 MB EXPANSION KIT	*(INSTALLED)*
D2381A	2 MB EXPANSION KIT	*(INSTALLED)*
82335B	HP-IB CARD	*(INSTALLED)*
82335B # 002	HP-IB CARD / NO SOFTWARE	
D1193A	17" ULTRA VGA DISPLAY S/N:JP24920011	
D1193A # ABA	U.S. POWER CORD	
C1413A	HP MOUSE MINIDIN	
9300-1407	HP - MOUSE PAD	
D2266-60007	DISK / DSHBD DOC ENGLISH	
D2266-60002	DOS / WIN ENGLISH MANUALS	

OPTION # 335 ***LASERJET PRINTER IV 100-120 VOLTS***

PRINTER SERIAL NUMBER:JPBG017704

C2001A	LASERJET IV PRINTER
C2001A # ABA	U.S. ENGLISH LOCALIZATION
92298A	EP-S TONER INK CARTRIDGE
92284A	CENTRONICS CABLE

G1201A - ALICE SINGLE INSTRUMENT SOFTWARE (DOS SERIES)

SOFTWARE SERIAL NUMBER: 3236A05290

OPTION # AAD	*3.5" DISK MEDIA*
G1201-60601	SINGLE INSTRUMENT S/W KIT
D1152A	MS-DOS 5.0 DUAL MEDIA
D1152A # ABA	U.S. ENGLISH VERSION
9010-0025	MICROSOFT WINDOWS 3.1

G1032C ENVIROQUANT TARGET SOFTWARE

SOFTWARE SERIAL #:3244A20517

OPTION # AAD	*3.5" DISK MEDIA*
G1032-60006	*ENVIROMENTAL MANUALS*
// //	// // //
G1032-90013	ENVIROQUANT MENU
G1032-90020	ENVIROQUANT DATA EDITOR
G1032-90021	ENVIROMENT FORM
G1034-90014	COMPUSERVE OFFER
G1032-60014	*SOFTWARE KIT*
// //	// //
G1032-10012	SOFTWARE MEDIA DISK 3.5"
G1032-90009	INSTALLATION GUIDE
G1032-90011	3.5" SUPPLEMENTAL DISK

G1034-C MUSTANG III (MS-DOS CHEMSTATION SOFTWARE)

SOFTWARE SERIAL NUMBER:3240A04049

OPTION # AAD	*DISK MEDIA*
G1034-60048	DISK MEDIA KIT 3.5"
// //	// //
G1034-10041	MS-CHEMSTATION S/W
G1030-90075	MS-CHEMSTATION CBT
G1034-90041	SUPPLEMENTAL DISK FOR G1034C # AAD
G1034-90042	*G1034-C MANUAL KIT*
// //	// //
G1034-90014	COMPUSERVE OFFER
G1034-90022	USERS GUIDE
G1034-90023	COMMANDS REFERENCE CARD
G1034-90024	LEARNING MAP
G1034-90025	MENU CARD (5989)
G1034-90032	MENU CARD (5971)
G1034-90033	MENU CARD (5970)
G1034-90036	MENU CARD DATA A
G1034-90034	MARCOS GUIDE (GI)
G1030-90070 *	VIDEO - OVERVIEW NTSC
5959-7117	ARCS CARD
9010-0027	EXCEL 4.0

G1035A WILEY 138K MASS SPECTRAL DATABASE REVB.00.00

SOFTWARE SERIAL NUMBER: 3208A30227

OPTION # AAD	3.5" DISK MEDIA
G1033-90007	Installation Guide
G1035-10002	Library Media (23 Disk)

Appendix F

Capital Procurement of Cryo SUMMA™ Canister System for the GC/MS

Pacific Northwest Laboratories
5th & W Streets
P.O. Box 999
Richland, Washington 99352

SHOW PURCHASE ORDER NUMBER
ON ALL PACKAGES, INVOICES,
AND CORRESPONDENCE.
COMPLETE PACKING LIST MUST
ACCOMPANY EACH SHIPMENT.

DATE: 02/09/93	DPAS RATING:	ORDER NO: 193711-ARD
BUYER: RICK DENNIS	TELEPHONE: (509) 375-2348	DELIVERY DATE: 03/09/93
TERMS: 3%/N15	SHIP VIA: UPS SURFACE	FOB: SIMI VALLEY, CA

SELLER	ENTECH 950 ENCHANTED WAY #101 SIMI VALLEY CA 93065	SHIP TO	Battelle For U.S. DOE c/o Westinghouse, 1163 Bldg Richland, WA 99352	BILL TO	BATTELLE ACCOUNTS PAYABLE PO BOX 999 RICHLAND, WA 99352 REF: ORDER NO. ABOVE (ATTACH BILL OF LADING OR PAID FREIGHT BILL.)

ITEM	QTY.	U/M	DESCRIPTION	UNIT PRICE	TOTAL PRICE
			THIS CONFIRMS OUR ORDER PLACED WITH: JOHN DESCHENES ON: 02/09/93		
1	1	EA	PRECONCENTRATION AUTOMATED PRECONCENTRATION UNIT	41,150.00	41,150.0
2	1	EA	CANISTER UNIT CANISTER CLEANING UNIT	7,240.00	7,240.0
3	1	EA	DILUTION UNIT STANDARDS DILUTION UNIT	11,500.00	11,500.0
			THE THREE LINE ITEMS ARE TO BE CONFIGURED WITH THE OPTIONS LISTED IN RFP 193711 AND ENTECH QUOTATION DATED 1/12/93. SEE ATTACHMENT A.		
				TOTAL PRICE:	59,890.0

GENERAL PROVISIONS NUMBER
A-287(R3)To Include Clauses 37, 41 &
42
gned Form 369(R3) Representations
and Certifications
ARE A PART OF THIS PURCHASE ORDER.

PACIFIC NORTHWEST LABORATORIES
BATTELLE MEMORIAL INSTITUTE

Kathleen A Moore 2/9/93
KATHLEEN A. MOORE, MANAGER
OPERATIONS AND SPECIALTY PRODUCTS PROCUREMENT
DATE

TO BE CONSIDERED FOR FUTURE ORDERS . . . MEET YOUR DELIVERY COMMITMENTS!

[illegible] F.1 08-08

Attachment 2

PR No. 193711

Sole Source Justification for the Procurement of an Automated Passivated Canister (Summa) Sample / Thermal Desorption Pre-concentration System w/ Canister Conditioning and Canister Standards Dilution Units.

I recommend that negotiations be conducted only with ENTECH Laboratory Automation for the supplies and services described herein.

1. Introduction

PNL has been contracted through WHC by DOE to establish headspace analysis capabilities for the Hanford Tank Safety Programs. This sole source justification is for the purchase of equipment which will be used by PNL for separating and identifying the components found in the tank farms. The following are justifications for sole source of the proposed Entech system:

- PNL must be compatible with their sponsor's (WHC) existing equipment for development, validation, and technology transfer to WHC mobile analytical laboratory group. WHC is presently implementing direct GC/MS capability in a mobile laboratory in which an Entech concentrator system already has been procured. If equipment other than the proposed Entech system is used by PNL, then results will not be compatible with the sponsor or transferable.

- The health and safety of Hanford employees working in the area is directly at risk until components of the tank vapors can be identified and proper precautions can be recommended. There are additional safety concerns regarding the potential flammability of selected single-shelled-tanks until the headspace vapors are characterized. This requires immediate action to implement analytical capabilities for characterizing the tank headspace in the field and in the laboratory. PNL is tasked with the laboratory work and must be compatible with the field work done by WHC.

- To accomplish their task, PNL must carefully coordinate their efforts with other contractors working on the same problem, i.e.., purchase the same type of equipment and meet milestone time requirements. Westinghouse Hanford (WHC) has already placed an order for a concentrating system from ENTECH as an high priority health and safety issue purchase. Since PNL is assigned the task of method development in this program PNL must use equipment compatible with that which WHC purchases. Furthermore, the laboratory method PNL develops must be made available to WHC (technology transfer) in sufficient time to coordinate with the tank site sampling dates determined by the program. This dictates that PNL must receive equipment at, or at least reasonably near the same time that WHC receives their equipment.' The entire success of this program relies on the ability of each contracting group involved to meet the time-line milestones indicated in the program.

The development of a method to perform organic analysis on tank vapor samples collected in passivated canisters (Summa) and to identify its components, requires the purchase of a system which includes: a pre-concentrating unit (to be interfaced with a GC/MS instrument) to pull aliquots from the canister sample, concentrate the vapor and focus it onto the GC/MS instrument for analysis; a unit for measuring aliquots of concentrated standards from multiple sources, blending them and delivering them to canisters for creating calibration standards; and a unit to clean and condition the canisters. The system must also include and support: a PC, cables and all software to interface Preconcentrator to GC instrument; a dot matrix printer for printing out system QA/QC reports; glass-lined-tubing for all connections coming into contact with the sample; compatible valving for each unit to support the same make (manufacturer) of passivated canister used in the sampling of tank vapors. This document covers the justification for a sole source procurement of a summa preconcentrator system with accessories; specifically ENTECH system.

2. History

There is a need to characterize Hanford tank vapor constituents immediately to address safety issues for the client WHC and DOE. The WHC tank vapor program has implemented a phase approach to characterize the tank vapors. Upon a recommendation by the Hanford Vapor Team and the Vapor Conference Panel of expert scientist from the national laboratories, the canister method of sampling followed by GC/MS analysis needs to be implemented at Hanford. Phase I requires PNL to implement this capability in the laboratory and assist WHC in the analysis of an identical system in WHC mobile analytical laboratory. The canister sampling/analysis method requires a concentrator system prior to GC/MS analysis. The proposed Entech system meets the requirement for summa analysis (EPA Method TO-14) and is identical to the WHC system.

Because this technique is new to the Hanford site, difficulties in the method development and subsequent transfer of technology between coordinating contractor groups must be minimized by purchasing compatible equipment. This will not only shorten overall method development time, but will expedite the method and equipment validation portion of the program, which is an important mile-stone before any field sampling can begin. Again, it is desirable to speed up the process of method development and validation, so that the vapor team can get to the job of solving the vapor tank characterization.

Since characterization of the vapor tank head-space is the main objective of this portion of the program, validation of the technique and method must be done by replicate analysis on compatible equipment. This dictates that PNL purchase the same equipment as the program sponsor, WHC, and the equipment of choice is the ENTECH system.

Finally, due to the "pioneering" nature of this program for the Hanford site, it is expected that with gained experience and improvements in technology, other vendors will not be precluded by the purchase of this equipment.

3. Technical Requirements

Uniqueness of the Entech System:

- The Entech system is the only vendor system that is capable of handling air samples with high water content without using a nafion dryer. A nafion dryer is unacceptable because it removes polar organics in addition to the water. The Hanford vapor program requires the analysis of polar organics in high humidity air samples.
- The Entech system has a unique concentration system for analyzing polar and expanded analytes to the EPA TO-14 method. Any other system will not mirror the Entech system that WHC is implementing in their mobile laboratory.

The Entech system proposed consist of a Preconcentrator unit, a standards dilution unit, and a canister conditioning unit. Addition descriptions are as follows:

Preconcentrator Unit:

Unit must be capable of automatically extracting and cryogenically concentrating samples collected in passivated canisters (SUMMA) or on thermal desorption tubes, and focusing sample for GC injection. Sample concentration and focusing must give operator the option of automatically isolating polar and/or non-polar components in the sample and must be able to pull canister samples from sub-ambient (< 1 atmosphere absolute), ambient, or pressurized canisters.

- Unit must be equipped with multi-stage H_2O and CO_2 removal techniques that are software controlled.

- Unit must have a means for minimizing H_2O interferences when using the polar compound analysis option. All tubing used in concentrator should be silica stainless steel (glass-lined stainless steel) to minimize polar compound

- Unit must be capable of automating a self-leak check, monitor actual flow rates, pressures and heated zones, and print out QA/QC reports which lists method settings and actual readings under PC software control.

- Unit must be capable of automated unattended method switching under computer control, i.e.., a means for multiple analysis (ten sample minimum) of ambient air canister, source canister, or thermal desorption tube samples without the need for manual control. This automation must be an integral part of the unit.

Standards Dilution Unit:

- System unit for creating gas standards in passivated canisters must be capable of allowing the operator to make dilution from 1 or 2 (combined) concentrated standards, with separate mass flow control of each addition valve including the make-up gas.
- Unit must be fitted with glass-lined tubing (glt) for making polar standards. Valving for each gas addition channel must be compatible for canisters used in vapor program.

Canister Conditioning Unit:

- System unit for canister cleaning and conditioning must include a means for evacuating, heating, humidifying and pressuring canisters. Gauges used in unit must allow the operator to check for system leaks, determine the vacuum capacity of the pump and to check pressure drops through the high vacuum pump.
- Conditioning unit must be compatible with concentrator unit and gas standard dilution unit described above, i.e., all three units must accept the same make of passivated canister used.

5. Estimated Cost

Cost for items detailed on the purchase requisition is $ 57K, not to exceed $ 60K. Installation and training (2-days) are included on this PR at an estimated cost of $ 2.5K, not to exceed $ 3K.

6.0 Signatures

To best of my knowledge and belief, and based upon the above information, the ENTECH system is the only source that can adequately and effectively perform the services described herein.

Richard B. Lucke
Senior Technical Specialist II

Therese Clauss
Technical Specialist

APPROVED

David Koppenaal
Section Manager
Atomic and Molecular Chemistry Section

ENTECH
LABORATORY AUTOMATION

PRODUCT QUOTATION

950 Enchanted Way #101 Simi Valley CA 93065

Page 1

Prepared for:

TERESSE CLAUSS
BATTELE PACIFIC NORTHWEST
P.O BOX 999 MSIN:P8-08
RICHLAND, WA 99352
PH 509-376-8051
FAX 509-376-2329

Date	F.O.B.	Quotation #
10/14/92	Simi-ppd add	10316

Payment Terms

NET 30 DAYS

Expiration Date	Scheduled Shipment
11/14/92	6 weeks ARO

ITEM	PRODUCT	QTY	DESCRIPTION	UNIT COST
			AUTOMATED PRECONCENTRATION SYSTEM	
1	2000	1	Automated concentrator with control electronics, GC & CPU interface cables; heated GC and manifold transfer lines, mass flow controller	$11,700.00
2	04-01021	1	High volume cryogenic trapping module (-180 C to 300 C) with removable trap	$1,800.00
3	04-02020	1	Open tubular column focusing trap	$1,200.00
4	04-03100	1	Oven module for nafion dryer, bypass loop or packed column injection	$1,400.00
5	2016CM	1	16 position canister manifold for automated ambient level analysis	$7,200.00
6	2016LM	1	16 position loop injection manifold	$7,600.00
7	04-11120	1	Spare 1/8" Ni trap with multibed cryogenic packing	$520.00
8	CPU386SX	1	PC AT computer includes: 8/12 MHz with 60 Mb hard drive, 1.2 Mb floppy drive & vga color monitor	$1,600.00
9	LX 810	1	Epson 810 Dot matrix printer	$250.00
10	PUMP-D	1	Oilless diaphram vacuum pump (27.5" Hg)	$400.00
11	2000-RK	1	Tool kit for 2000 concentrator	$120.00
12	2000-PKA	1	Polar compound kit	$1,160.00
13	06-00611	10	6 Liter SUMMA Passivated canisters	$410.00ea
14	INSTALL	1	Two days installation and training	$2,200.00

Prepared by

Authorized agent

FEDERAL ID NUMBER 77-0249862

ENTECH
LABORATORY AUTOMATION

PRODUCT QUOTATION

950 Enchanted Way #101 Simi Valley CA 93065

Prepared for:

TERESSE CLAUSS
BATTELE PACIFIC NORTHWEST
P.O BOX 999 MSIN:P8-08
RICHLAND, WA 99352
PH 509-376-8051
FAX 509-376-2329

Date	F.O.B.	Quotation #
10/14/92	Simi-ppd add	10316

Payment Terms

NET 30 DAYS

Expiration Date	Scheduled Shipment
11/14/92	6 weeks ARO

ITEM	PRODUCT	QTY	DESCRIPTION	UNIT COST
			CANISTER CLEANING	
1	C3000	1	Automated canister conditioning system control module	$1,950.00
2	07-10010	1	Humidification chamber	$200.00
3	PUMP-HV	1	High vacuum pump	$400.00
4	PUMP-D	1	Oilless diaphragm pump	$490.00
5	OILTRP-HV	1	Stainless 3/4" ID cryogenic oil vapor trap	$400.00
6	HEATER	1	Canister heater for 4 canisters	$200.00 ea
			STANDARDS DILUTION	
7	4560	1	6 Channel Dynamic Dilution System equipped with 2 MFC's (please specify the desired range for Mass Flow controllers)	$8,200.00
8	MFC-xxxx	2	Additional MFCs with Kalrez for extended capabilities (Substitute xxxx with range in sccm) available ranges: 0005, 0010, 0050, 0100 0200, 0500, 1000, 5000	$1,650.00 ea

Prepared by

Authorized agent

FEDERAL ID NUMBER 77-0249862

PURCHASE REQUISITION

DATE 6/22/93
PAGE 1 OF 2
PRIORITY RATING DO- E
☐ SCREENED ☐ PRE INSERT
VENDOR CODE
246066
SPONSOR/OUTER

VENDOR

DO NOT WRITE IN AREA WITH BOLD LINES

PURCHASE REQUISITION FAX NO. 375-3687

DATE(S) DELIVERY REQUIRED 7/30 93
☐ STANDARD OR ☐ VITAL OR ☒ EMERGENCY

FOR EMERGENCY/VITAL INDICATE IMPACT (BUDGET OR MILESTONES) ON PROJECT/PROGRAM.
Program Delay.
Milestones will not be met.

SEE INSTRUCTION PAGE FOR DEFINITIONS.

	YES	NO
CONFIRMING	☐	☐
PRINT PO	☐	☐
MAIL PO	☐	☐

F.O.B. DELIVERY PROMISE
EXPECTED DELIVERY

DO NOT WRITE IN AREA WITH BOLD LINES.

ITEM NO.	QUANTITY	UNIT	DESCRIPTION	UNIT PRICE	TOTAL PRICE	RECEIVED
1	3	days	Training on new Entech System (PO#193711-AND) for the analysis of organic compounds in air using Summa canisters. Training will consist of the following:			
		1.	Training and operation of automated pre concentration unit configured with the polar compound mode. Best achievable analysis of an TO-14 gas mixture will be demonstrated using the purge and trap mode and the 2-dimensional concentration mode. An existing certified TO-14 gas mixture at 10ppm will be provided by PNL. Also, a test mixture consisting of acetone (50ppm), n-butanol (10 ppm), benzene (0.10 ppm) and Butyl-benzene (10 ppm) will be provided by PNL for testing.			
		2.	Training and operation of the canister cleaning system			

SPONSOR	PROGRAM/PROJECT IDENTIFICATION	ITEM	ORGANIZATION	ACCT CLASS-W/O PROJ.
SPONSOR	D5020			
X 1830 DOE-A	20061	a1	D7E13	M23270
1831 DOV-B	FUNDING CLIENT NAME			
1831 IND-C				
ENW PNL-D				

CHECK AS APPLICABLE AND OBTAIN REQUIRED SIGNATURES OR REQUIRED DOCUMENTATION
☐ SENSITIVE PROPERTY
☐ HAZARDOUS MATERIAL
☐ RADIOACTIVE MATERIAL
☐ SAFETY RELATED ITEM OR SERVICE
☐ SURVEILLANCE EQUIPMENT
☐ CONFLICT OF INTEREST

TOTAL ESTIMATED COST $ 4500
FOR BID ONLY ☐
BID DUE DATE

TECHNICAL ADMINISTRATOR RB Lucke
PAYROLL 39202 BLDG RM 329/2 PHONE 6-8705 MSIN P8-08
TA INITIALS DATE 6/22/93

DELIVER TO RB Lucke RB Lucke
AREA 300 BLDG RM 329/2 PHONE 6-8705 MSIN P8-08

FINANCIAL SPECIALIST DL Stapleton DATE 6/23/93 PHONE 4388 TM

AUTHORIZED REQUISITION APPROVALS
Roger M. Bean
LEVEL 4 OR ABOVE DATE 6/22/93

OTHER DATE
Faxed to procurement
DATE 6/23/93

ADP STUDY NO. SIGNATURE DATE

IMPACT LEVEL
☐ I
☐ II
☒ III
☐ IV
ITEM NO.

QA REQUIREMENTS ☐ YES ☒ NO
QA CLAUSES
QA REPRESENTATIVE/PHONE/DATE

INSPECTION BY: SOURCE REC.

		Yes	No
☐	AUTHOR	☐	☐
☐	QC	☐	☐
☐	OTHER	☐	☐

PO TYPE
POB CODE (1 OR 2)
POA CODE
TERMS
PP/COLLECT (01 OR 02)
CARRIER
FOB PT
COO DATE
COO PERSON
COMMODITY CODE
SHIP TO
BUYER

PURCHASING

54-1080-001 (08/92)

CONTINUATION SHEET

PURCHASE REQUISITION

Requisition No.	Vendor	Vendor No.	Purchase Order No.	Page
246066	Entech			2 of 2

Item No.	Qty	Unit	Product Code	Description	Unit Price	Total	Del. Date
				(continuation of item 1 from page 1)			
				3. Training and operation of the standard dilution system. This will include preparation of standards by the static dilution method.			
				4. Additional training on software operation of the Entech System.			
				Suggested Vendor: Entech 950 Enchanted Way #101 Simi Valley Ca 93065 805 527-5939 fax: 805 527-5687			

IBUTION: White - Purchasing Blue - Accounts Payable Green - Archer
Yellow - Cost Control Pink - Quality Assurance Goldenrod - Requisitioner

54-3000-282 (06/92)

PR No. 246066

Sole Source Justification for Training on a Recently Acquired Entech System for the Analysis of Organic Compounds in Air Using Summa Canisters

Additional 3 days of training is required on a recently acquired Entech System (PO No. 193711). The existing system was designed, manufactured, and installed by Entech. The existing Entech system was configured and installed for analysis of polar organic compounds in air specific to PNL needs by Entech. Sole source is required from Entech. Training shall include the following:

1. Training and operation of automated pre concentration unit configured with the polar compound mode. Best achievable analysis of an TO-14 gas mixture will be demonstrated using the purge and trap mode and the 2-dimensional concentration mode. An existing certified TO-14 gas mixture at 10 ppm will be provided by PNL. Also, a test mixture consisting of acetone (50 ppm), n-butanol (10 ppm), benzene (0.10 ppm), and butyl-benzene (10 ppm) will be provided by PNL for tested.

2. Training and operation of the canister cleaning system.

3. Training and operation of the standard dilution system. This will include preparation of standards by the static dilution method.

4. Additional training on software operation of the Entech System.

<u>Signatures:</u>
To the best of my knowledge and belief, and based upon the above information, training from Entech is the only source that can adequately and effectively perform the services described herein.

Richard B. Lucke
Senior technical Specialist II

DW Koppenaal 6/23/93
Approved David Koppenaal
Section Manager
Atomic and Molecular Chemistry Section

Appendix G

Draft Strategy Plan for the Development and Validation of the SUMMA™ Canister Analysis Method

Guideline for PNL Laboratory Method Development for the Analysis of Passivated Canister Samples by GC/MS

Summary: The following document outlines PNL's proposed Waste Tank Vapor Program (WTVP) action plan for analyzing passivated canister samples. This document outlines only those steps directly relating to developing a laboratory capability for anlayzing passivated canister samples collected from Hanford tank farms, by GC/MS interfaced with a canister concentrator (GC/MS/CC). U.S Environmental Protection Agency Compendium Methods TO-12 and TO-14 will be the model methods for which PNL will optimize the purchased equipment. After Phase I sampling and analysis is complete, Phase II method development will involve the expansion of the model method target list to include components of interest based on results from Phase I Tank 103C sampling.

Method TO-14 Outlined

1. Clean and verify sample canisters before sampling. (OGI)
2. Sampling procedure (OGI/WHC).
3. TO-14, Section 10.0 "Analytical System," GC/MS-SCAN
    - Set up GC/MS concentrating system. Method recommends a Nafion dryer for water removal and implementing a means for cleaning dryer with zero air between sample analyses.
    - GC/MS system must pass a BFB tune. Tune criteria and ions monitored are listed in Table 4 of TO-14. The system must meet tuning criteria before any analysis can begin.
    - GC/MS system with concentrator in place must be "challanged" with zero-humidified air. The system is considered clean if it contains less than 0.2 ppbv of each targeted volatile organic compound.
    - Initial calibration with 3 standard levels plus zero-humidified air. Must use NBS traceable or NBS/EPA CRMs in pressurized cylinders (w/mixture of target VOCs at 10 ppmv in N2) as working standards diluted with humid zero air. Method Section 10.2.3.2
    - Daily tune w/ BFB. Must be w/in 10% of absolute abundance criteria from previous day, or recalibrate. If w/in 10%, daily calibration requires only 1 point standard.
    - Follow daily point calibration with blank canister challange.
4. Canister receipt procedure: Record canister sample in dedicated book. Check canister pressure with gauge and record pressure.
5. Analyze samples. Backflush nafion dryer. Report results.
6. Clean canisters and verify clean as per method Section 11.

<u>PNL's TO-14 Action Outline:</u>

1. Locate enough laboratory space to house canister analysis equipment. The laboratory used must be reasonably free of organic contaminents. **Complete.**

2. Purchase equipment needed to perform TO-14 successfully. PNL has purchased the following equipment for this program:

   - Hewlett Packard 5971 GC/MS and data system. GC is also equipped with a FID.

   - ENTECH Model 2000 automated VOC preconcentrator equipped with three traps:

     1) Glass Beads
     2) Tenax
     3) Open tube fused silica

     This preconcentrator is also configured with RESTEK's Siljco-steel® lines for efficient transfer of polar VOC's throughout the concentrator.

   - ENTECH Model 2016CM autosampler for ambient level canister analysis and the Model 2016LM autosampler with loop injector for source level canister analysis.

   - ENTECH Model 3000 canister cleaning system equipped with canister heaters to simultaneously heat (to 100 °C) clean up to 4 canisters with humidified air.

   - ENTECH's Model 4560 Dynamic Diluter® and gas blending system fitted with mfcs covering 4 flow ranges to allow for the dilution of gas standards and samples to a known concentration with purified nitrogen, or air.

     In addition to the items listed above, PNL has purchased a Balston clean air generator, a limited number of summa canisters to use until WHC purchases the bulk of the canisters for the program, and Scott test mixtures as requested.

3. Set up and validate that the Entech system is working properly. Tests will include,

   - QA/QC run by the manufacturer upon completed set up of the system,

   - test runs of the different available modes of the concentration system,

   - validate the cleanliness of the system and ability to check for system leaks,

   - identification of consistent system interference peaks.

   Since polar analytes are of interest to this program, a glass-bead trap followed by a tenax trap is used for water, and CO_2 removal as opposed to the nafion dryer recommended in compendium method TO-14.

4. Clean and run a blank canister. It must be demonstrated that the canisters and system can be cleaned to a known level. (See item 6.)

5. A set of TO-14 standards (uncalibrated) will be run initially for practice. From these stored practice runs, the following development may be completed:

   - determine temperature program for the separation of all componenets in the mixture and retention times for analytes,

   - test recovery of all components from the canister and concentration system,

   - identification of any potential chromatographic interferences,

   - the analysis of a known low (< 5 ppb) concentration of the TO-14 mixture,

   - gain familiarity with entire system and software functions.

6. A low level standard (<1ppb) of selected analytes will be created to use for demonstrating a clean system. Because this project will deal primarily with source level samples, cleanliness to < 0.2 ppb will not be necessary. However, an appropriate 'zero' level must be recommended by the vapor technical team. Until a recommendation is made, PNL will use a 1-ppb concentration of TO-14 gas standard to determine the zero detection limit of the method.

7. Run standards to generate precision and accuracy data.

8. An attempt will be made to dilute a set of calibration standards in canisters and run them on the GC/MS to create a 3-point calibration curve. It may be later determined that two sets of calibration curve ranges, one for ambient air, and one for source sampling are needed. A BFB tune will not be a priority as discussion of this particular subject with experts in the field deem it an unneccesary step. A search for peak match identification will verify target library search capability.

9. Run standards to generate accuracy data.

The following will be performed simultaneously with TO-14 method validation:

10. PNL will receive a canister sample from the waste tank (BY-104) to make test runs with. The objective of these test runs will be

    - to observe analytes recovered from the summa canisters which are not already a part of the compendium method TO-14 target list,
    - to check system background peaks (known) against any analytes observed in the summa canister sample,
    - to gain some familiarity with the character ie., wet and concentrated nature, of waste tank samples,
    - to try and obtain some kind of precision data from waste tank samples,
    - to try to tentatively identify analytes of interest for the extension of the TO-14 target analyte list.

The following will be done with BCO's guidance:

11. Create TO-14 standards in canisters and verify capability by sending out for analysis.

12. Clean sample canisters and verify capability by sending out for analysis.

13. Demonstrate a clean GC/MS/CC system by analyzing clean canisters from BCO. Similarly, demonstrate TO-14 capability by analyzing BCO audit canisters.

14. Do a final report of TO-14 analysis ready status.

Validation of capability to analyze waste tank samples

1. Continue analysis of canister samples for qualitative information. Data generated at PNL will be compared with athat from OGI for consistancy.

Appendix H

Pacific Northwest Laboratory Work Plan for the Analysis of HEPA Filters

Battelle
Pacific Northwest Laboratories
Battelle Boulevard
P.O. Box 999
Richland, Washington 99352
FAX (509) 376-2329

January 15, 1993

Jerry Osborne
Westinghouse Hanford Company
P. O. Box 1970
Richland, WA 99352

Dear Jerry:

Subject: PNL WORK PLAN FOR ANLAYSES OF HEPA FILTERS

Enclosed is the PNL Work Plan for Analyses of HEPA Filters. If you have any questions, please contact me.

Thank you.

Sincerely,

Steven C. Goheen

Steven C. Goheen, Ph.D.
Senior Research Scientist
Chemical Sciences Department

SCG:gkr

Enc.

cc: R. B. Conrad., WHC, w/enc.
R. B. Lucke, PNL, w/enc.
D. M. Strachan, PNL, w/enc.

PNL WORK PLAN FOR ANALYSES OF HEPA FILTERS

INTRODUCTION

On December 7, 1992, the HEPA and Carbtrol filters at C-103 Tank were changed. The previous filter change was done on July 30, 1991. The two charcoal filters consist of a 55-gallon drum filled with GA-1 (charcoal saturated with phosphoric acid) and G-1 (activated charcoal). Analysis of the filters may provide valuable information regarding the tank headspace vapors, their interaction with the filters, and filter emissions. Earlier analysis of C-103 vapors down-stream of the HEPA filter reported alkane nitriles were present. A series of nitriles from propane to heptane (C_3- to C_6-) nitrile have been reported (1-7). It has also been suggested that alkane nitriles may be degradation products of polyurethane from the HEPA filters (8). It is not clear whether degraded polyurethane or direct tank emissions are the source of the nitriles. Analysis of the HEPA filter and subsequent comparison to the results of the analysis of the organic layer in the tank, vapors down-stream of the HEPA filter, and vapors upstream of the HEPA filter may help identify the source of the alkane nitriles. For example, if alkane nitriles are present and downwind of the HEPA filters, but not in the tank, these results would support the idea that the alkane nitriles are generated at the HEPA filter.

High levels of NPH have also been reported as an emission product of Tank 103C (1-7). It is not clear whether the HEPA filter acts as a reservoir for NPH from the tank. Therefore, NPH will be analyzed from the HEPA filter to estimate its concentration.

Tributylphosphate (TBP) is a relatively non-volatile component of Hanford tank waste. It is not expected to be volatile enough to be present in the vapor phase of the tank, but is has been observed in the vapor phase outside of the HEPA filter (2,7). Consequently, it would be useful to evaluate levels of TBP in the HEPA filter to determine whether the filter sorbs TBP and acts as a reservoir, or is passive to the presence of TBP and does not collect this substance.

APPROACH

The analysis of extracts of samples will provide a fingerprint of the alkane nitriles present. The concentration of nitriles will be determined by gas chromatography/mass spectrometry (GC/MS).

In addition, a comparison of the NPH and TBP concentrations from the same sample will help us identify typical extractable levels of these contaminants in HEPA filter material.

PNL's efforts will be focused on analyzing the HEPA rather than Carbtrol filters at this time. It may be valuable for WHC to archive a sub-sample from the Carbtrol filters for future analysis if warranted. The sub-sampling of the HEPA filter will be done by WHC, and the analysis will be done by PNL. This work plan describes a sampling and analysis of HEPA filter to determine NPH and TBP levels as well as that of alkane nitriles.

SAMPLING

HEPA Filter: Ideally, a cross section of the inlet end of the HEPA and a cross section of the outlet end of the HEPA would be most valuable. Alternatively, a core sample taken through the HEPA can be used. Two core samples from the HEPA are required. The core samples should be placed in separate glass, teflon-lined capped jars. In addition, identical samples are required from a non-used equivalent (preferably of the same lot number and kept sealed at 0 to 5°C) HEPA filter. The level of radioactivity must be determined to be less than 0.2 µCi prior to PNL receipt of the samples. All radioactive samples will be returned to WHC after analysis by PNL.

Carbtrol Filter: If WHC decides to archive sub-samples from the Carbtrol filters, the following is recommended: From schematics of the Carbtrol 55-gallon drum there is a dip tube at the inlet end from the top of the drum to the bottom. Ideally, a charcoal sub-sample closest to the dip tube access is desirable. If it is possible to obtain a core cross section from the drain connection at the bottom side of the drum, then this may also be desirable. Two core samples from each charcoal filter are recommended. The core samples should be stored in separate glass, teflon-lined capped jars. In addition, identical samples are recommended to be taken from a non-used equivalent (we prefer the same lot number) Carbtrol filter. The level of radioactivity should also be determined prior to storage.

ANALYSIS

HEPA filters are not designed for trapping volatile or semivolatile organic materials for analysis. It is not known how well these filters will adsorb or desorb the various analytes of interest. Therefore, results from analyses will be for screening purposes only. Screening analysis of the HEPA filter will be accomplished on the sub-samples from the HEPA filter by solvent extraction* followed by GC/MS analysis. Specifically, semivolatiles including (alkane nitriles, such as from propane nitrile to heptane nitrile), normal paraffin hydrocarbons (NPH), and tributylphosphate

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*using methanol, methylene chloride, or other solvents

(TBP) will be targeted. If the NPH levels are below 100 ppm in the solvent extract, purge and trap GC/MS will be done on a non-extracted filter sample. If the NPH levels are above 100 ppm, then an alternative analysis by direct GC/MS of the solvent extract will be done. Samples with greater than 100 ppm NPH cannot be analyzed by purge and trap GC/MS due to interference by NPH. Components other than NPH, TBP, and alkane nitriles, but detected by GC/MS will be tentatively identified by the comparison with that of the mass spectral library, and classical mass spectral interpretation. It should be noted that HEPA filter and Carbtrol filters were not designed for chemical analysis. Extractable organics are probably present in new, non-used filters. Therefore, new, non-used filters preferably of the same size and lot number of the exposed filters are required for analysis comparison.

References:

1 W. Einfeld, R.A. Rasmussen, and L. D. Bustard (1992) Hanford Tank 103C Headspace Vapor Analysis Canister Sampling Results, Interim Report, September 22 for Jerry Osborne.

2 R. A. Jenkins, C. E. Higgins, C. Y. Ma, A. B. Hoffman, and J. H. Moneyhum (1992). Hanford Tank Vapor Characterization Support, Interim Report No. 1., September 1 to C. Odell.

3 R. B. Lucke, S. A. Clauss, and S. C. Goheen (1992). Phase Zero Malt Analysis of Volatile Headspace Gases: Samples from WHC Storage Tank 241-C-103. Letter Report to Jerry Osborne, October.

4 Internal memo 12240-SAS93-009, "C-103 Phase 0 Results", November 16, 1992, to J. W. Osborne.

5 Internal memo 28200-91-077, "Results of Cryogenic Sampling from Waste Tank C-103", November 22, 1992, to R. K. Tranbarger.

6 R. B. Lucke and J. A. Campbell (1991). "Analysis of Volatile Headspace Gases Sampled by Cryogenic Traps from WHC 241-C-103 Tank on April 10, 1991, Letter report to Rhett Trenbarger, September 11.

7 R. B. Lucke and J. A. Campbell (1990). "Analysis of Cryogenic Traps Used to Sample Volatile Headspace from 103-C and 102C Tanks". March 28 Supplement to Jan. 18, 1990 report. Prepared for Westinghouse Hanford Company.

8 Internal memo number 65453-87-106, July 22, 1987, "Interim Report on the Tank C-103 HEPA Filter Failure Problem", to Al Alstad.

Appendix I

Pacific Northwest Laboratory Letter Report on Phase Zero MALT Analysis of Volatile Headspace Gases Sampled From WHC Storage Tank 241-C-103 in August 1992

Battelle
Pacific Northwest Laboratories
Battelle Boulevard
P.O. Box 999
Richland, Washington 99352
Telephone (509) 376-2329

October 30, 1992

Jerry Osborne
Westinghouse Hanford Company
P. O. Box 1970
Richland, WA 99352

Dear Jerry:

Enclosed is a copy of the PNL results from Phase 0 MALT samples that WHC provided in methanol. We analyzed them using GC/MS. All the pertinent data and tabulated results are attached.

We need to point out, however, that our results are incomplete. We were not provided with the relationship between the amount of air sampled and the levels of analytes in the sample. In addition, the descriptions of the samples in the table which we provided in the introduction was created from a telephone conversation with WHC staff rather than a written document. It is due to these uncertainties that we have been unable to provide a concise table indicating the levels of the various constituents in the tank vapor. Therefore the descriptions need careful review by your staff. Once they provide the data and assignments, you will need to recalculate the values for the concentrations of vapor constituents. Reliable results can be obtained by correcting any possible errors in the table included in the introduction and applying the values in Table 2 and the correction factors from your staff. Therefore, as we have agreed, the final analytical chemistry report will be issued by your staff using this PNL data coupled with the WHC data addressed above.

For your consideration, I suggest that the final report be co-authored by your staff and ours. Co-authored reports have been published by staff members from our respective Tank Safety Projects as part of the overall team effort. This is just a suggestion on our part and.

Phase Zero Halt Analysis of Volatile Headspace Gases Samples from WHC Storage Tank 241-C-103

R.B. Lucke
S.A. Clauss
S.C. Goheen

October 1992

Prepared for
Westinghouse Hanford Company

Pacific Northwest Laboratory
Richland, Washington 99353

INTRODUCTION

This is a report of results from gas chromatography/mass spectrometry (GC/MS) analysis of Mobile Analytical Laboratory Trap (MALT) samples obtained from Tank 103C during Phase Zero of the Hanford Vapor Program. Phase Zero is a trial sampling and trial analysis of the vapor from Tank 103C to get sampling/analysis experience and preliminary component identification. Because the manifold set-up at the tank during Phase Zero does not allow a representative samples to be obtained from the tank headspace, the sample is not meant for accurate characterization. The purpose of Phase Zero is to identify potential sampling and analytical concerns before candidate techniques are evaluated. The sample inventory is tabulated below. Analyses included direct GC/MS for volatile and semi-volatile organic compounds, and direct GC/MS for ammonia. The MALT system was designed by WHC and is not discussed in this report[1]. All concentrations are reported for the methanol extract solutions and not the vapor. In order to calculate concentrations in the headspace, the MALT air-sampling-volume and the methanol-rinse-volume must be obtained from the MALT personnel at WHC.

Samples

WHC ID	Description
C103-810-1A1	Rep 1, Trap 2
C103-810-2A1	Rep 1,Trap 1
C103-810-SA1	Rep 1, Blank
C103-811-1A2	Rep 2, Trap 2
C103-811-2A2	Rep 2,Trap 1
C103-811-SA2	Rep 2, Blank
C103-728-F1	Carbatrol, Trap 1
C103-728-B1	Carbatrol, Trap 2
C103-728-BL1	Carbatrol, Blank

The above descriptions of the WHC samples were obtained by telephone conversation with Courtney Jones of WHC. The letters and numbers after the last hyphen in the above WHC Identifications are referred in subsequent tables.

SAMPLE PREPARATION

The cryogenic samples were received in 40 mL glass vials with Teflon®-lined caps from WHC on August 13, 1992. Samples were stored at 4°C +/- 2°C prior to analysis. Sample identifications were recorded in Battelle Pacific

Northwest Laboratory notebook # 54820. All subsequent information was recorded in this notebook. Samples were analyzed as received with no sample preparation.

ANALYSIS METHOD

GC/MS analyses of the cryogenic traps was achieved by following procedure PNL-ALO-334. Analysis was achieved using the direct injection GC/MS method. Different GC conditions were used for volatile/semivolatile analysis than for the ammonia analysis. Tentative identification of unknowns was accomplished using mass spectral interpretation and comparison of mass spectra from the EPA/NIST library of mass spectra. The HP5890/5970 GC/MS used for the analyses is in Lab 8B of the 329 building. The ammonia was quantitated using the external standard method using a three point calibration curve. The volatile/semi-volatile organics were quantitated using certified internal standards using the extracted base ion chromatograph of each targeted analyte. Non-targeted analytes were tentatively quantitated using the internal standard method and the relative response factor (RRF) of either acetone or tridecane. The choice between acetone or tridecane was based on the structure of the compound in question. For example, the alkanenitriles were quantitated using tridecane because of their long alkane backbone, and butanal was quantitated using acetone because of its carbonyl group.

QA/QC

The QA/QC procedures described in PNL-ALO-334 were followed. Instrument blanks, tune/mass calibration, internal standards, calibration, and calibration check samples were all met. This report does not address the efficiency of the HALT sampling system technique.

DATA

The GC/MS raw data were archived on a magnetic storage tape as permanent storage and is located in Lab 8B of the 329 building. Hard copy output data which include several pages of ion chromatograms, mass spectra, calibration/quantitation data, and library-searched spectra are stored in the

GC/MS file cabinet located in room 2 of the 329 Building. In this report, the final quantitation tables and only pertinent information are included. Data are also maintained in Laboratory Record Book (LRB) # BNW-5482.

RESULTS AND CONCLUSIONS

Tables 1 and 2 show the GC/MS results for volatiles and semivolatile organics while Table 3 shows the results for ammonia. Undecane, ethanol, methylene chloride, and dimethyldimethoxysilane were detected in all the MALT samples. Since their concentrations are similar for Trap #1, Trap #2, and the field Blank, they are probably artifacts from the sampling procedure. Trap #1 samples from 810 and 811 (#C103-810-2A1 and #C103-811-2A2) contained ammonia and significant amounts of organic compounds. Normal Paraffin Hydrocarbon (NPH) was the main constituent. NPH consist of n-C11, n-C12, n-C13, n-C14, and n-C15. Butanol was the other major constituent in these samples. Some unidentifiable alkanes and alkenes were also found in addition to a series of normal alkanenitriles. Normal alkanenitriles were previously reported at trace levels in Tank 103C by Lucke and Campbell (1990)[2]. Quantitative results were provided for target analytes and compounds determined by mass spectrometry. Non-targeted analytes quantitative results are to be used as an estimate only. Estimated levels of non-targeted analytes are potentially less accurate than targeted analytes.

REFERENCES

1. Ulbricht, W.U. 1991. "Report on 241-C Tank Farm Air Sampling Results of 1989". Westinghouse Hanford Company, WHC-SD-WM-RPT-019. Richland, Washington.

2. Lucke, R.B. and J.A. Campbell. 1990. "Analysis of Cryogenic Traps used to Sample Volatile Headspace From 103-C and 102-C Tanks (Direct Injection GC/MS analysis of cryotrap samples and purge and trap GC/MS analysis of sample WHU #27). Letter RB Lucke to William Ulbricht, Westinghouse Hanford Company.

Appendix

Pages	Item
A1 - A13	Quantitation Spreadsheet Results
A14 - A16	Selected Chromatograms
A17 - A18	Selected Mass Spectra

Table 1
Components Identified using GC/MS

				Sample Number								
				728			810			811		
Pk #	Ret Time	Component	Quant Note	B1	BL1	F1	1A1	2A1	SA1	1A2	2A2	SA2
1	2.52	Acetone	Ion 43					X			X	
2	6.20	1-Butanol	Ion 41					X			X	
3	6.43	Benzene	Ion 78									
4	16.6	Undecane	Ion 57	X	X	X	X	X	X	X	X	X
5	18.1	Dodecane	Ion 57					X			X	
6	19.5	Tridecane	Ion 57					X			X	
7	20.3	Tetradecane	Ion 57					X			X	
8	22.1	Pentadecane	Ion 57								X	
10*	2.31	Ethanol	TIC vs RRF DMK	X	X	X	X	X	X	X	X	X
11	3.10	Methylene chloride	TIC vs RRF DMK	X	X	X	X	X	X	X	X	X
12	3.60	Butadiene	TIC vs RRF C13					X				
13	4.11	Butanal	TIC vs RRF C13					X			X	
14	5.95	Dimethyldimethoxysilane	Not Quant	X	X	X	X	X	X	X	X	X
15	9.12	Pentanenitrile	TIC vs RRF C13					X			X	
16	9.86	Methylbutylketone	TIC vs RRF C13					X				
17	10-11	column bleed	Not Quant	X	X	X	X	X	X	X	X	X
18	11.8	Hexanenitrile	TIC vs RRF C13					X			X	
19	12.3	C7 or C8 ketone	TIC vs RRF C13					X			X	
20	13.8	C7 or C8 ketone	TIC vs RRF C13					X			X	
21	13.9	Heptanenitrile	TIC vs RRF C13					X			X	
22	14.3	C7 or C8 ketone	TIC vs RRF C13					X			X	
23	20.7	Siloxane	TIC vs RRF C13				X					
24	22.7	Siloxane	TIC vs RRF C13				X	X		X	X	
25	25.1	Siloxane	TIC vs RRF C13				X	X		X	X	

Minor alkanes and alkenes were not assigned numbers. See chromatograms.

Peaks #1-8 were identified and quantified using authentic standards. See text.

*Peak #9, which was acetonitrile, is not listed in this table because it was only found in the standards.

Peaks #10-25 were tentatively identified by MS. Quantitation was estimated using a response factor from a different standard.

Table 2

Quantitation of Components in C103
Phase 0 Malt Extracts

Results in mg/L*

		728			810			811		
Pk #	Component	B1	BL1	F1	1A1	2A1	SA1	1A2	2A2	SA2
1	Acetone					34			20	
2	1-Butanol					81			96	
3	Benzene									
4	Undecane	1.6	2.4	2.2	2.2	39	2.2	2.0	29	2.3
5	Dodecane					286			262	
6	Tridecane					379			310	
7	Tetradecane					59			40	
8	Pentadecane					1.2			0.9	
-	NPH Total**	1.6	2.4	2.2	2.2	763	2.2	2.0	641	2.3
10	Ethanol	73	57	58	51	91	53	54	63	55
11	Methylene chloride	176	147	144	133	136	126	125	130	122
12	Butadiene					0.8				
13	Butanal					2.6			3.2	
15	Pentanenitrile					1.1			0.2	
16	Methylbutylketone					1.5				
18	Hexanenitrile					1.2			1.5	
19	C7 or C8 ketone					1.6			1.6	
20	C7 or C8 ketone					2.1			1.8	
21	Heptanenitrile					1.7			1.8	
22	C7 or C8 ketone					0.9			1.1	
23	Siloxane				4.6					
24	Siloxane				7.7	8.7		2.6	1.4	
25	Siloxane				2.6	4.7		1.5	1.5	

Peaks #1-8 were quantified using authentic standards and internal standard.
Peaks #9, 14, 17, and minor alkanes and alkenes were not quantified.
Peaks #10-25 were quantified using a response factor from a different standard.
*Concentration of the methanol extracts. Not the concentration of the tank headspace.
**NPH is normal paraffin hydrocarbons. Sum of peaks 4 through 8.

Table 3
Ammonia Concentration in Tank C103 Phase 0 MALT Extracts

WHC ID	MALT ID	mg/L*
C103-728-F1	Carbatrol Trap1	-
C103-728-B1	Carabtrol Trap 2	-
C103-728-BL1	Rinse Blk	-
C103-810-2A1	Rep 1/Trap 1	190
C103-810-1A1	Rep 1/Trap 2	-
C103-810-SA1	Rinse Blk	-
C103-811-2A2	Rep 2/Trap 1	130
C103-811-1A2	Rep 2/Trap 2	-
C103-811-SA2	Rinse Blk	-

- Below detection limit of the analysis method (20 mg/L)

* Concentration of the methanol extracts. Not the concentration of the tank headspace.

NH3 Results for the Analysis of the C103 Samples

Sample	Area	Conc (M)	mg/mL
810 2A1	9759222	0.011	0.19
811 2A2	4114307	0.008	0.13
			Percent
Check 1	17761036	0.016	78%
Check 2	17519507	0.016	78%

C103 728 F1

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	1.58
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	1.58

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.55E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	72.86	0.48
Methylene Chloride	11	3.1	175.65	0.48

Areas used for above			Area
Ethanol	10	2.3	18064978
Methylene Chloride	11	3.1	43551035

C103 728 B1

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	2.36
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	2.36

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.86E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	57.41	0.48
Methylene Chloride	11	3.1	146.80	0.48

Areas used for above			Area
Ethanol	10	2.3	17058960
Methylene Chloride	11	3.1	43624799

C103 728 BL1

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	2.16
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	2.16

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.88E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	57.63	0.48
Methylene Chloride	11	3.1	143.95	0.48

Areas used for above			Area
Ethanol	10	2.3	17341758
Methylene Chloride	11	3.1	43319681

C103 810 1A1

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	2.23
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	2.23

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	2.00E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	51.11	0.48
Methylene Chloride	11	3.1	133.29	0.48
Siloxane	23	20.7	4.57	15.6
Siloxane	24	22.7	7.74	15.6
Siloxane	25	25.1	2.57	15.6

Areas used for above			Area
Ethanol	10	2.3	16333916
Methylene Chloride	11	3.1	42596984
Siloxane	23	20.7	47442476
Siloxane	24	22.7	80415185
Siloxane	25	25.1	26686620

C103 810 2A1

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result	
Acetone	1	2.5	33.98	
1-Butanol	2	6.2	81.30	
Benzene	3	-	nd	
Undecane	4	16.6	38.51	
Dodecane	5	18.1	285.50	
Tridecane	6	18.2	379.00	
Tetradecane	7	19.5	59.20	manual calculation
Pentadecane	8	20.1	1.19	
NPH Total		-	763.40	

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	2.06E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	91.31	0.48
Methylene Chloride	11	3.1	135.85	0.48
Butadiene	12	3.6	0.81	15.6
Butanal	13	4.15	2.61	15.6
Pentanenitrile	15	9.1	1.06	15.6
methylbutyl ketone	16	9.7	1.45	15.6
Hexanenitrile	18	11.8	1.22	15.6
C7 or C8 ketone	19	12.3	1.61	15.6
C7 or C8 ketone	20	13.7	2.09	15.6
Heptanenitrile	21	13.9	1.69	15.6
C7 or C8 ketone	22	14.3	0.94	15.6
Siloxane	24	22.7	8.65	15.6
Siloxane	25	25.1	4.69	15.6

Areas used for above			Area	
Ethanol	10	2.3	30082263	
Methylene Chloride	11	3.1	44754958	
Butadiene	12	3.6	8620473	
Butanal	13	4.15	27934069	Sum of 4.1 and 4.2 peak
Pentanenitrile	15	9.1	1.14E+07	
methylbutyl ketone	16	9.7	15563565	
Hexanenitrile	18	11.8	13062343	
C7 or C8 ketone	19	12.3	17252491	
C7 or C8 ketone	20	13.7	22353341	
Heptanenitrile	21	13.9	18079235	
C7 or C8 ketone	22	14.3	10069930	
Siloxane	24	22.7	92610739	
Siloxane	25	25.1	50234452	

C103 810 SA1

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	2.16
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	2.16

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.88E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	53.32	0.48
Methylene Chloride	11	3.1	125.76	0.48

Areas used for above			Area
Ethanol	10	2.3	16021521
Methylene Chloride	11	3.1	37786069

C103 811 1A2

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	2.02
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	2.02

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.88E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	54.00	0.48
Methylene Chloride	11	3.1	124.58	0.48
Siloxane	24	22.7	2.55	15.6
Siloxane	25	25.1	1.45	15.6

Areas used for above			Area
Ethanol	10	2.3	16223700
Methylene Chloride	11	3.1	37431553
Siloxane	24	22.7	24887132
Siloxane	25	25.1	14187227

C103 811 2A2

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result	
Acetone	1	2.5	19.74	
1-Butanol	2	6.2	96.46	
Benzene	3	-	nd	
Undecane	4	16.6	28.91	
Dodecane	5	18.1	261.90	
Tridecane	6	18.2	309.70	
Tetradecane	7	19.5	39.60	manual calculation
Pentadecane	8	20.1	0.90	
NPH Total		-	641.01	

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.54E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	62.65	0.48
Methylene Chloride	11	3.1	130.38	0.48
Butanal	13	4.15	3.15	15.6
Pentanenitrile	15	9.1	0.17	15.6
Hexanenitrile	18	11.8	1.47	15.6
C7 or C8 ketone	19	12.3	1.55	15.6
C7 or C8 ketone	20	13.7	1.81	15.6
Heptanenitrile	21	13.9	1.79	15.6
C7 or C8 ketone	22	14.3	1.10	15.6
Siloxane	24	22.7	1.42	15.6
Siloxane	25	25.1	1.51	15.6

Areas used for above			Area
Ethanol	10	2.3	15403165
Methylene Chloride	11	3.1	32055260
Butanal	13	4.15	25147089
Pentanenitrile	15	9.1	1359906
Hexanenitrile	18	11.8	11728569
C7 or C8 ketone	19	12.3	12356997
C7 or C8 ketone	20	13.7	14427479
Heptanenitrile	21	13.9	14302519
C7 or C8 ketone	22	14.3	8803643
Siloxane	24	22.7	11380824
Siloxane	25	25.1	12089353

C103 811 SA2

All concetrations in mg/L

Target Analyte Results

Compound	Peak #	Time	Result
Acetone	1	-	nd
1-Butanol	2	-	nd
Benzene	3	-	nd
Undecane	4	16.6	2.34
Dodecane	5	-	nd
Tridecane	6	-	nd
Tetradecane	7	-	nd
Pentadecane	8	-	nd
NPH Total		-	2.34

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	1.76E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
Ethanol	10	2.3	55.23	0.48
Methylene Chloride	11	3.1	122.30	0.48

Areas used for above			Area
Ethanol	10	2.3	15531922
Methylene Chloride	11	3.1	34397603

Calibration Check 1:10 Standard

Treating Standard data as if it were a sample

All concetrations in mg/L

Target Analyte Results

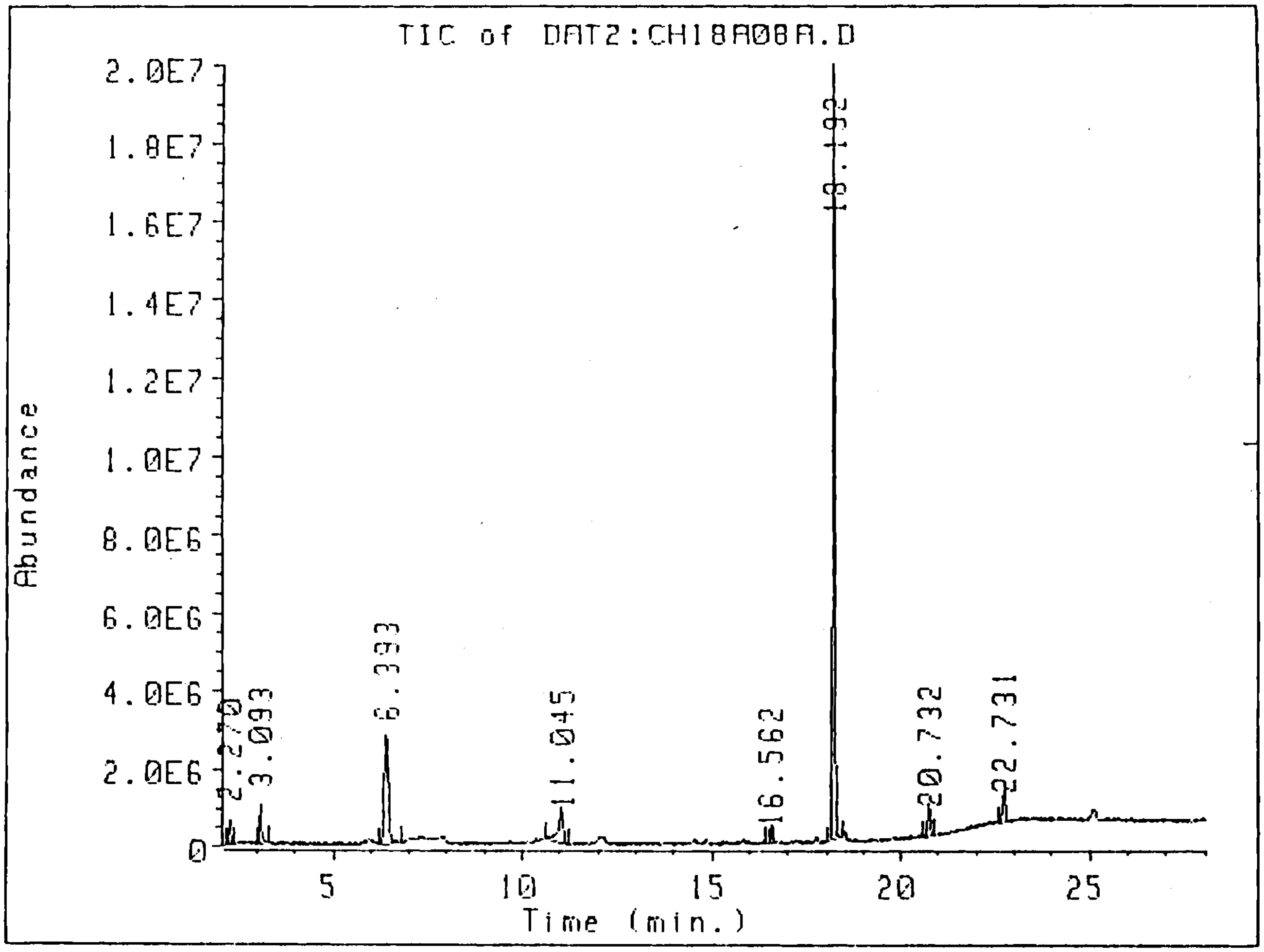
Compound	Peak #	Time	Result
Acetone	1	2.5	248.50
1-Butanol	2	6.2	281.80
Benzene	3	6.5	271.40
Undecane	4	16.6	2.02
Dodecane	5	18.1	124.30
Tridecane	6	19.5	308.00
Tetradecane	7	20.8	280.80
Pentadecane	8	22.1	14.98
NPH Total		-	730.10

Internal Standard of Benzene d6 TIC

		Conc.	Areas
Benzene d6 TIC		300.00	3.59E+08
RRF Acetone	0.48		
RRF Tridecane	15.6		

Results of Analytes Tentatively Identified using GC/MS

Tentative Id.	Peak #	Time	Result	RRF
None Noted				



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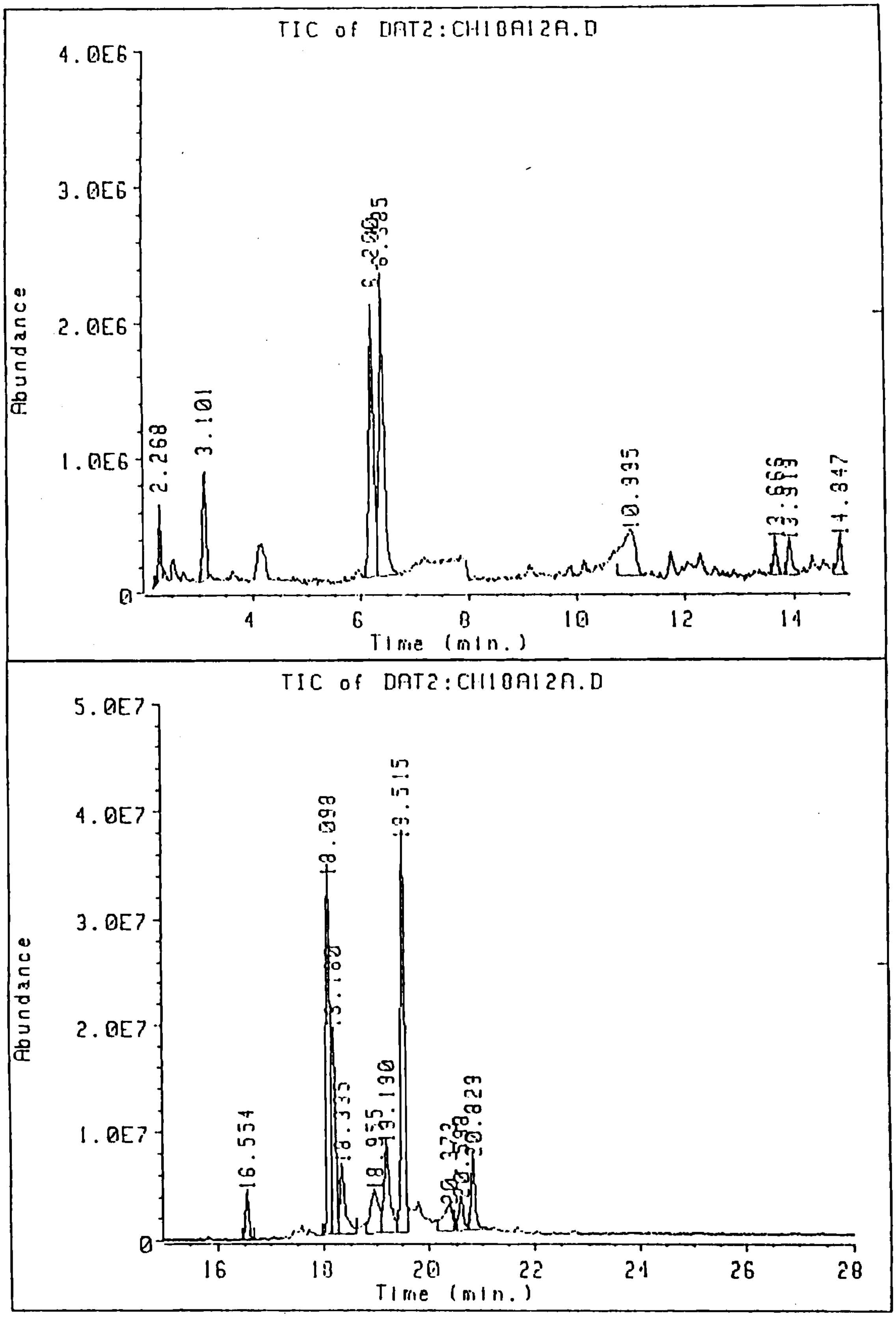
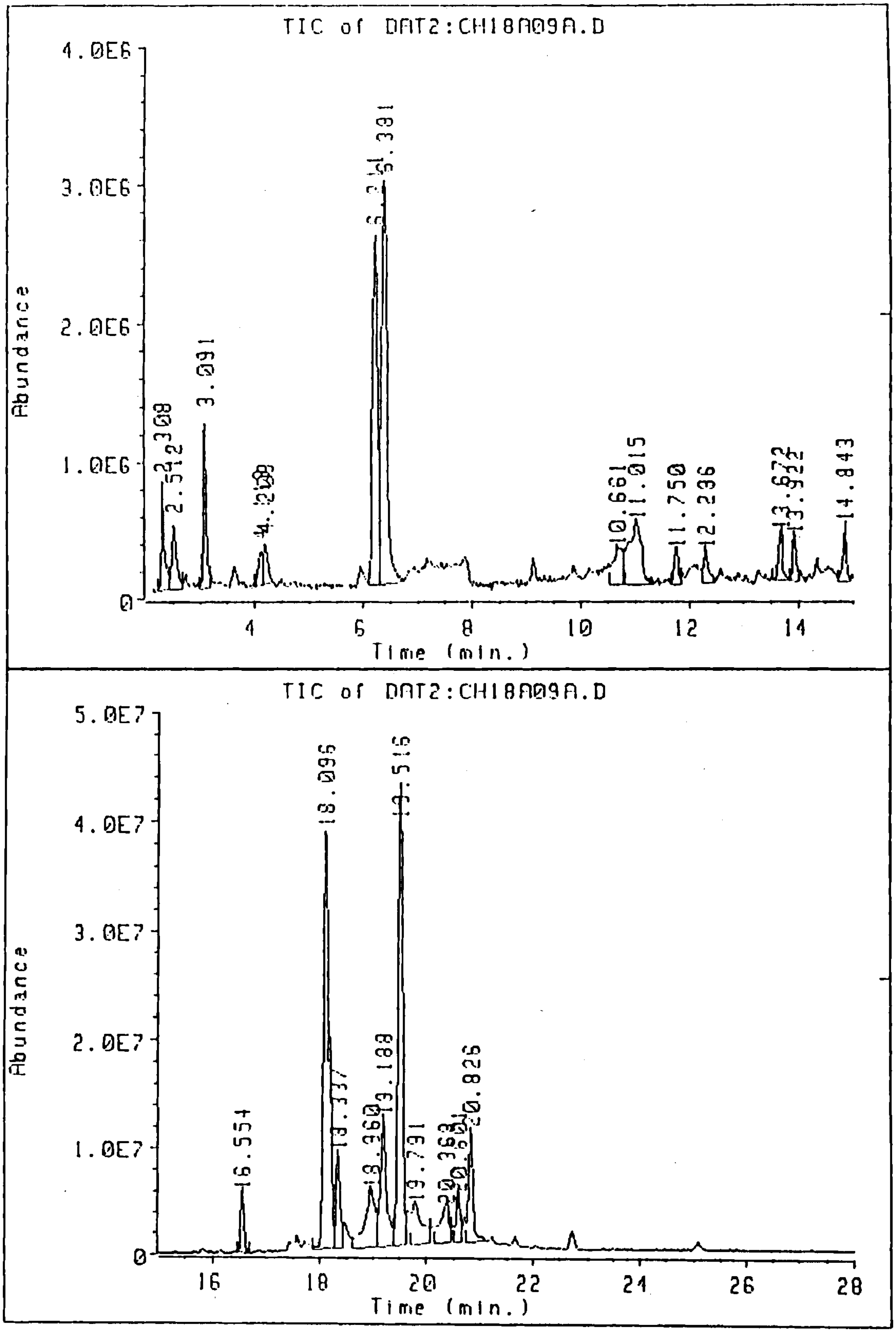


Trap 1. C103-811 2A2



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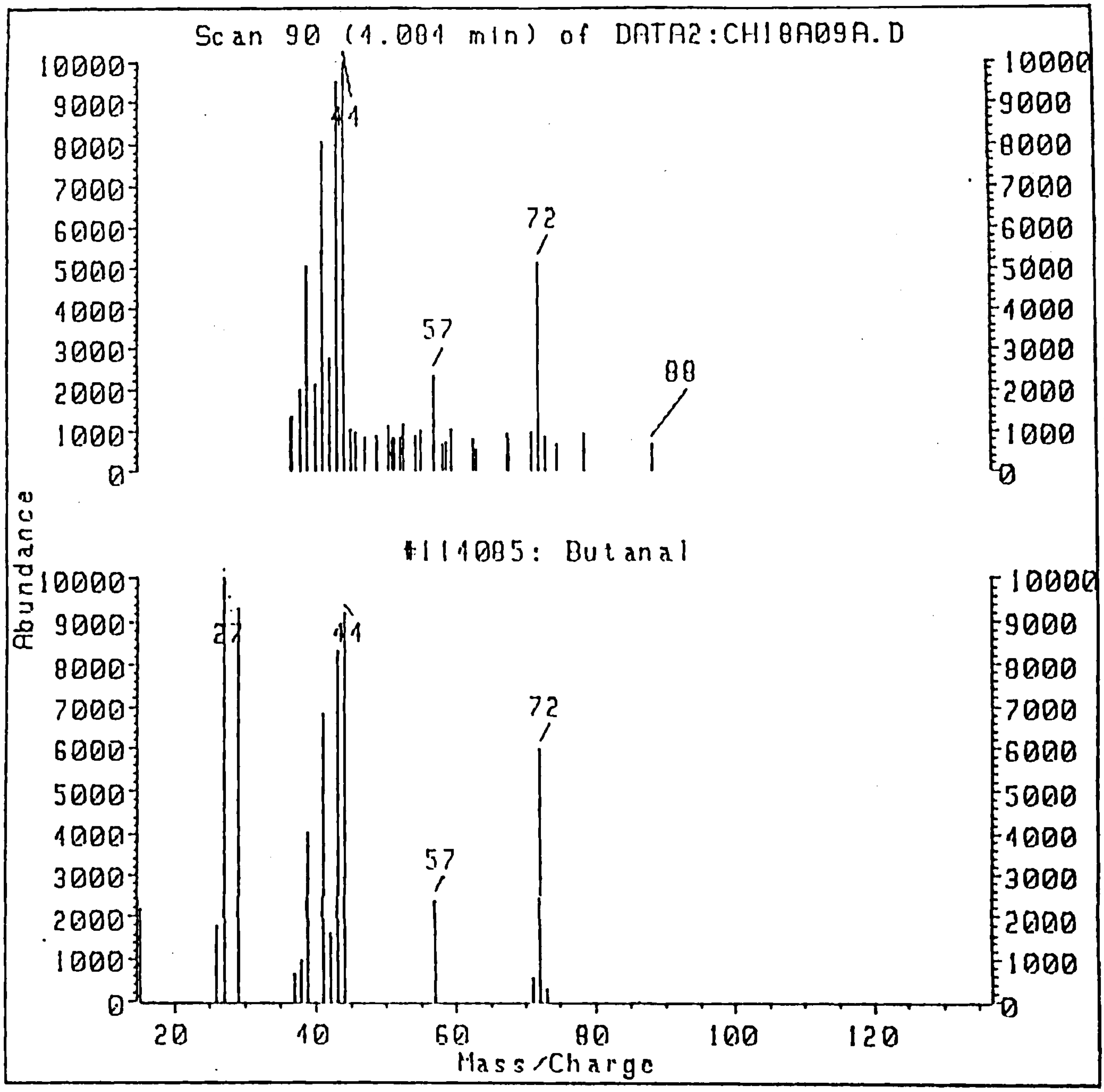

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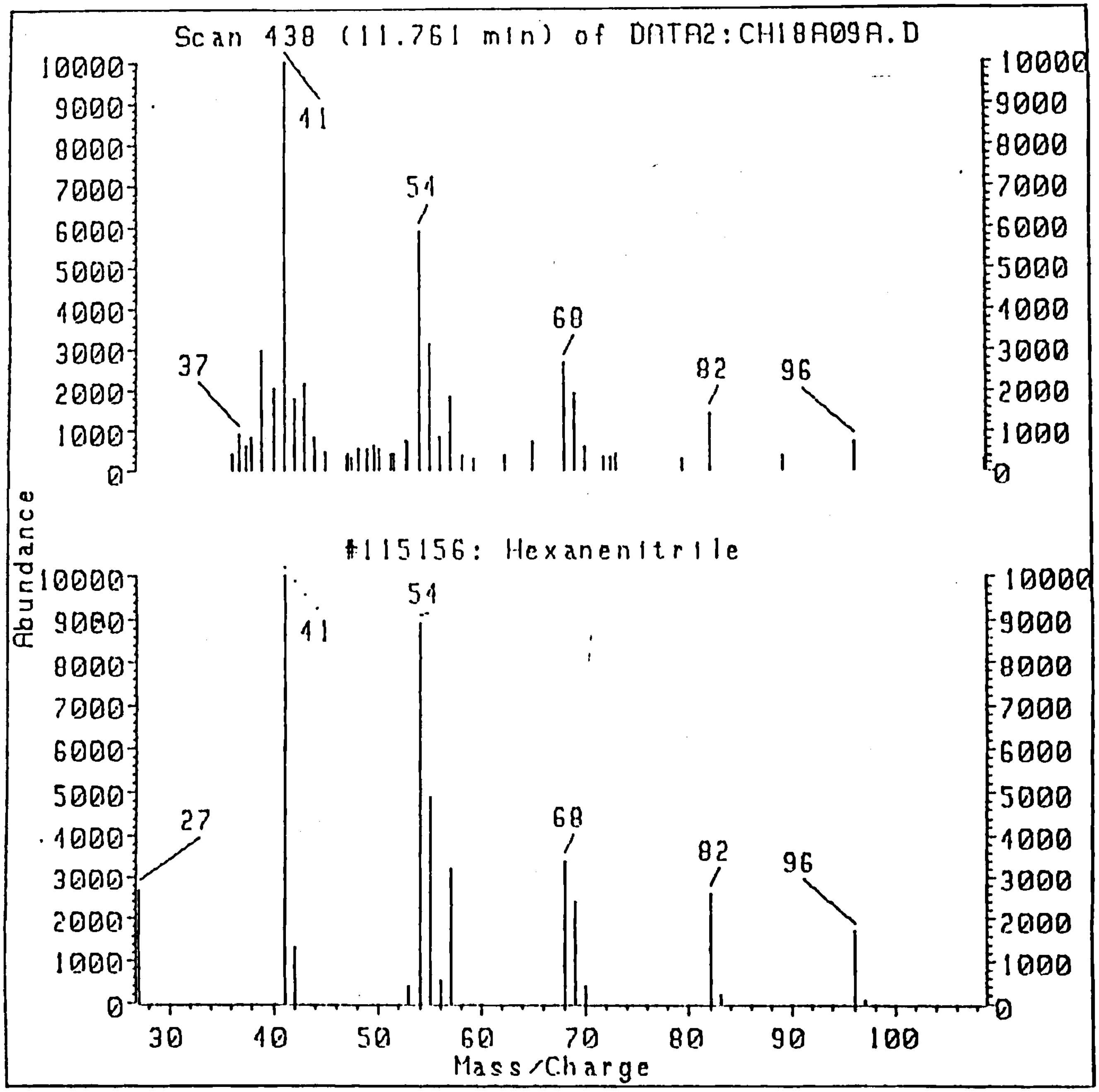

Upper - Hexanenitrile in C-103 810 2A1

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