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MERCURY SPECIATION STUDY

REVISED FINAL WORK PLAN
SITE 8 - NITROGLYCERIN PLANT OFFICE

NAVAL ORDNANCE STATION
INDIAN HEAD, MARYLAND

FOR THE
ENVIRONMENTAL ENGINEERING BRANCH
CHESAPEAKE DIVISION (CODE 114)
NAVAL FACILITIES ENGINEERING COMMAND
WASHINGTON, D.C. 20374

BY
HAZWRAP SUPPORT CONTRACTOR OFFICE
MARTIN MARIETTA ENERGY SYSTEMS, INC.
OAK RIDGE, TENNESSEE 37831

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1.0 INTRODUCTION

Naval Ordnance Station (NOS) Indian Head, Maryland is included in the Naval Installation Restoration Program (IRP). The IRP, originally named the Naval Assessment and Control of Installation Pollutants, was established to identify the presence of suspected contamination at Navy and Marine Corps lands resulting from past operations and, if needed, to institute remedial actions.

The Chesapeake Division (CHESDIV) of Naval Facilities Engineering Command has specifically requested the support of the Department of Energy (DOE) in conducting Site Characterization, Feasibility Study, and Remedial Design activities to develop a solution to the mercury contamination problem at Site 8--Nitroglycerin Plant Office (Bldg. 766). Energy Systems, operators of Hazardous Waste Remedial Actions Program, Support Contractor Office, Oak Ridge, Tennessee, has been assigned responsibility for managing this effort for the Navy under the interagency agreement (1791-1791-A1) with the Department of Energy (DOE). E.C. Jordan Co. (Jordan) acting as subcontractor to Energy Systems will assist in conducting Site Characterization Studies, conducting a Feasibility Study, and preparing Remedial Design documents to address the mercury contamination problem at NOS Indian Head, Maryland.

The preparation of the Mercury Speciation Study Work Plan fulfills the requirements of Subtask 11.1--Speciation Study Work Plan and Subtask 11.2--Implementation of the Speciation Study as outlined in Statement of Work (SOW) Amendment No. 2--Tasks 10, 11, and 12--Mercury Speciation and Treatability Study, Site 8, Nitroglycerin Plant Office, Naval Ordnance Station, Indian Head, Maryland.

This Work Plan is submitted in six parts with two appendices. Section 1.0 provides a description of Naval Ordnance Station (NOS) Indian Head and the proposed scope of the Mercury Speciation Study. Section 2.0 presents a summary of site conditions, a summary of the findings from previous investigations, and the technical program from the field program to the production of the final Mercury Speciation Study Report. Section 3.0 presents the project management organization with descriptions of the responsibilities of key personnel. Section 4.0 presents the detailed schedule for the completion of the project, estimated to take approximately 17 weeks. Section 5.0 provides a list of referenced documents and Section 6.0 contains a list of acronyms used in the report.

Also appended to the Work Plan are the site-specific project Health and Safety Plan (HASP) (Appendix A), and the Quality Assurance Project Plan (QAPP) (Appendix B). The overall HASP and QAP for IRP projects have already been prepared and are included herein by reference only. Copies of these plans are available from Martin Marietta Energy Systems, Inc., (Energy Systems). Copies of these plans have been sent to Naval Facilities Engineering Command Chesapeake Division (CHESDIV) and NOS.

1.1 Background

NOS Indian Head covers 3,423 acres and is bounded by the Potomac River, Mattawoman Creek, Chicamuxen Creek, the town of Indian Head, and private property. It is situated in the northwestern section of Charles County, Maryland, 25 miles southwest of Washington, D.C. Figure 1 shows the location of the Naval Ordnance Station.

The mission of NOS is to provide material and technical support for assigned weapons systems, weapons, or components and to perform additional tasks as directed by the Naval Sea Systems Command. These tasks may include research, development, engineering, production, and quality surveillance for weapons systems, propulsion, unconventional explosives, cartridge-actuated and propellant-actuated devices, and chemicals. Disciplines represented at NOS include expertise in weapons systems, propulsion, explosives development, and propellant and explosives chemistry.

NOS hosts two major tenants: the Naval Explosive Ordnance Disposal Technology Center (NEODTC) and the Naval School, Explosive Ordnance Disposal (NSEOD). NEODTC was established to develop procedures for rendering safe conventional and special weapons, guided missiles, biological and chemical munitions, tools, and equipment. The mission of NSEOD is to train officers and enlisted personnel in methods and procedures for recovery, evaluation, rendering safe, and disposal of surface, underwater, conventional, and nuclear explosive ordnance.

Over a period of approximately 20 years, mercury was released in small amounts from sink and floor drains at the Nitroglycerin Plant Office (Bldg. 766) into a storm drain. A portion of this mercury discharged into an ephemeral stream and, eventually, to a tidal pond system located downgradient of the building. The manhole, stream and tidal pond comprise Site 8.

The presence of mercury in sediments in the stream and tidal pond at NOS resulted in a study to delineate the extent of contaminated sediments and the feasibility of in-situ treatment or removal actions to mitigate potential hazards to public health and the environment. Jordan was authorized by the U.S. Department of the navy through its contractor, Martin Marietta Energy Systems to conduct a Feasibility Study/Remedial Design at NOS. In December of 1987, E.C. Jordan completed a Supplemental Site Characterization (Task 2) (E.C. Jordan, 1987) that assessed the distribution and fate of mercury in sediments surface water at Site 8. The characterization included estimation of the mass of mercury entrained in site sediments. Based on these estimates of mercury mass Feasibility Study and Remedial Design Alternatives Evaluation Report was a completed in February of 1988 (E.C. Jordan, 1988). Prior to selection of a remedial alternative, a Mercury Speciation and Treatability Study was proposed to better define the type(s) of mercury contamination present.

Methods for determining mercury's fate in the environment have recently been developed in the course of mercury contamination studies conducted at Oak Ridge Research Institute (ORRI). These analytical procedures allow investigators to determine what chemical species of mercury are present in the environment.

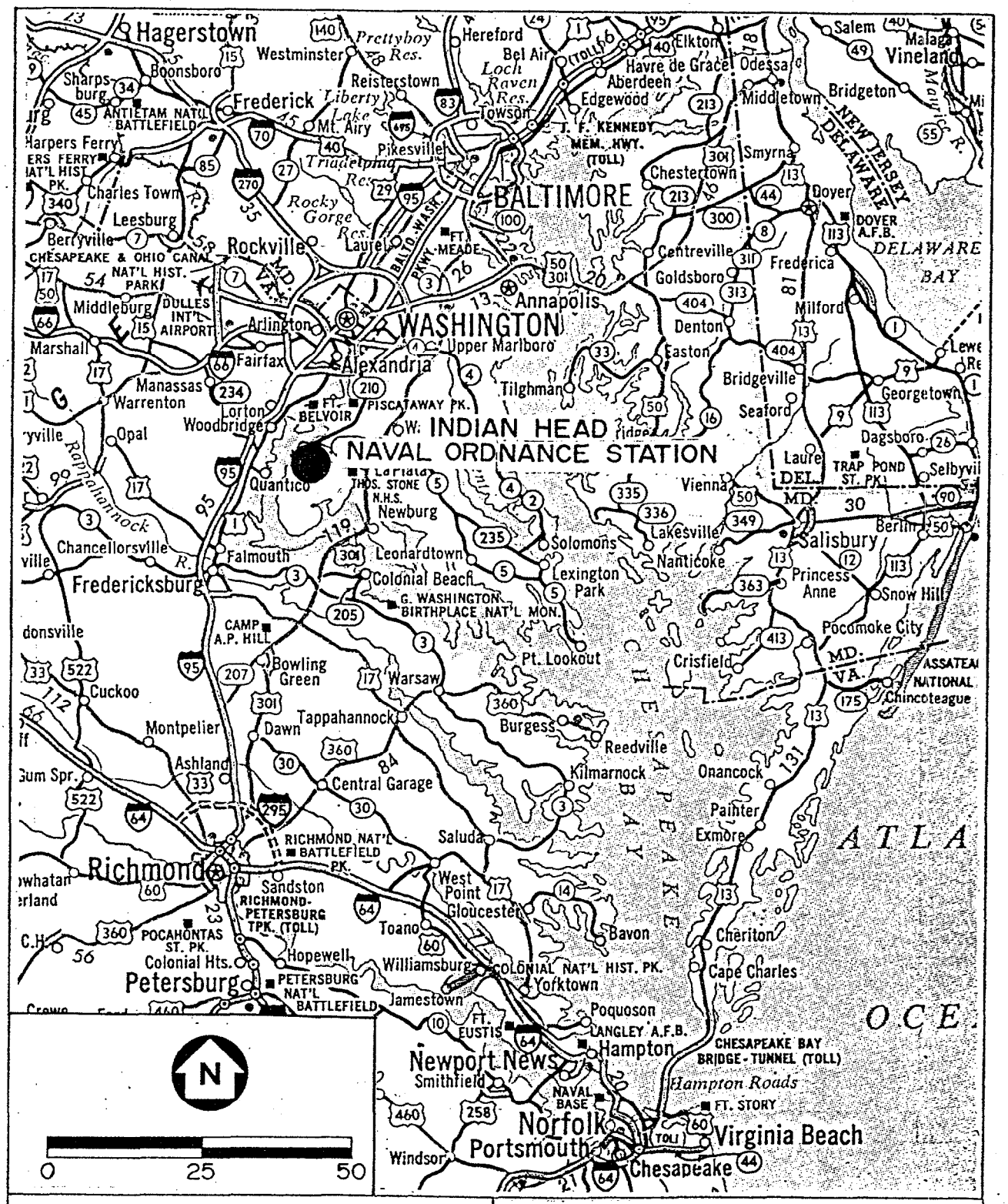

FIGURE 1

LOCATION OF NOS INDIAN HEAD

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Elemental mercury, inorganic mercury compounds, and methyl mercury can be differentiated via these analytical procedures.

Investigators at ORRI are also developing in situ remedial processes to transform the more mobile forms of mercury to a more stable inorganic form as mercuric sulfide. It may prove feasible to apply this technology to mercury contaminants at Site 8. Such a transformation could allow for these insoluble compounds to be left in place without adverse effects on human health or the environment.

Application of the analytical procedures in discerning mercury's speciation in the environment allows the contamination problem to be better defined and will consequently aid in developing alternatives for cleanup.

1.2 Purpose and Scope

1.2.1 Purpose

The purpose of the Mercury Speciation Investigation is to determine what forms of mercury are present in the stream and tidal pond at Site 8. the results of the investigation are necessary to effectively evaluate remedial technologies and to implement treatability studies.

The importance of determining the relative amounts and/or forms of mercury present in site sediments is due to the fact that the toxicity and bioavailability of mercury varies according to its chemical form. Characterization of the types of mercury will allow a more precise characterization of risks associated with the contamination and, in turn, the level of clean up required.

Mercury associated with sediments is cycled in the aquatic environment between sediments, surface water, and biota. Mercury in sediments exists as several forms: organomercury complexes (such as methyl mercury), inorganic mercury complexes, adsorbed (bound) elemental/ionic mercury, and elemental mercury (Schindler and Alberts, 1977). The hazards associated with environmental mercury exposures are related to the amount and types of mercury released from sediments to the overlying water. The toxic forms of mercury which can be released from sediments to surface water include the mercuric ion (Hg^{+2}), mercurous ion (Hg_2^{2+}), elemental mercury (Hg^o), and methyl mercury (CH_3-Hg^+) (Schinder and Alberts, 1977). The stability of mercury compounds (inorganic and organic complexes) in sediments along with the relative amounts of elemental and methyl mercury and other physical factors will determine the release of toxic mercury species.

Methyl mercury is considered to be the most hazardous mercury species to humans and aquatic life (U.S. Environmental Protection Agency, 1985; Eisler, 1987). This form of mercury is highly stable and lipid soluble (Beiger and Jesnolov, 1979) making it highly toxic and readily accumulated by aquatic organisms. Studies have shown that methyl mercury in sediments is accumulated by benthos with subsequent transfer to fish (Hildebrand et al., 1980). Methyl mercury ingested by humans is associated with neurological effects as it is readily absorbed in the intestinal tract and can cross the blood brain barrier accumulating in brain tissue (Klaassen et al., 1986).

Elemental mercury is primarily a health threat for humans via inhalation of vapors. Exposures can result in neurological effects and kidney damage (Klassen et al., 1986).

Inorganic mercury salts in sediment may be divalent (mercuric) or monovalent (mercurous). Toxicity of these salts is associated, in general, with their solubility (U.S. Environmental Protection Agency, 1985; Klassen et al., 1986) or rather their propensity to dissociate to the mercuric or mercurous ion. The mercuric ion (Hg^{2+}) is the toxic species and can be readily accumulated by aquatic organisms. Mercurous compounds are less toxic than mercuric forms (presumably because they are less soluble) (Eisler, 1987; U.S. Environmental Protection Agency, 1985). Ingestion of mercurous or mercuric compounds can result in gastrointestinal problems and kidney damage (Klaassen et al., 1986) for humans.

Inorganic mercury complexes are less toxic than organomercury forms (i.e. methyl mercury).

For aquatic life, inorganic forms (with the exception of mercurous nitrate) are 4 to 31 times less acutely toxic than organomercury compounds (U.S. Environmental Protection Agency, 1985).

Knowledge of the relative toxicity of mercury species and the distribution of those species in Site 8 sediments will aid in the evaluation of remedial alternatives and selection of treatability studies.

If the predominant form of mercury is found to be in the elemental form, a remedial alternative involving volatilization will be considered and a volatilization treatability study implemented.

Remedial alternatives for ionic or inorganic mercury contamination will involve reducing their solubility. Such alternatives may include in-situ reduction of mercury to mercuric sulfide by amending sediments/soils with calcium sulfate and lactate. Removal of the mercuric sulfide may not be necessary.

Remediation of organic mercury contamination (methyl mercury) would have to consider soil removal and treatment. Organic species of mercury are, as before stated, highly toxic and mobile and require removal from the aquatic environment.

1.2.2 Scope

This Work Plan describes the scope of work and methodology which will be employed by Jordan to conduct a Mercury Speciation Study at Site 8. This study will be conducted in compliance with Subtask 11.2 - Implementation of the Speciation Study, of Task Order No. X-09, Amendment No. 2. This subtask is divided into the following four work elements.

- Element 1: Soil and Sediment Sampling
- Element 2: Laboratory Speciation Analysis
- Element 3: Preparation of Draft Speciation Study Report
- Element 4: Preparation of Final Speciation Study Report

It is intended that the work described in this Work Plan will be in compliance with CERCLA, SARA, and RCRA regulations where applicable. The IRP program parallels the CERCLA program conducted by the USEPA. Because of the impact of SARA on CERCLA, the USEPA guidance documents, Guidance and Remediation Investigations under CERCLA (EPA, 1985) shall be considered during the implementation of this Work Plan.

1.3 Project Logistics

This section describes the logistics of on-site field work to be carried out at NOS Indian Head. Prior to conducting field work activities, Jordan will submit a letter to NOS security which contains the names and social security numbers of all personnel who will potentially be involved with on-base activities. These personnel will receive a security clearance prior to proceeding on base.

Upon arrival at NOS, Jordan personnel will meet with the Senior Environmental Engineer and the Branch Manager in order to inform them of Jordan's plan of action during field sampling activities. In accordance with arrangements made during previous Jordan field trips, the following logistical considerations will be implemented.

- Communications. On-base communications will be conducted from telephones located in the Nitroglycerin (NG) plant office. Off-base telephones will be utilized should the need arise to contact parties located off base.

- Potable Water and Sanitation. Potable water and sanitation facilities located in the NG plant office will be utilized.

- Safety Inspections. All vehicles and equipment will undergo a safety inspection at the safety office prior to proceeding on base.

- Emergencies. In the event of an extreme medical emergency, injured personnel will be taken to the base medical facility. Less serious injuries requiring medical attention will be handled at Physician's Memorial Hospital. Pertinent emergency phone numbers and routes to hospitals are presented in the Health and Safety Plan, Appendix A.

2.0 TECHNICAL APPROACH AND TASKS

This Speciation Study Work Plan has been developed based on Jordan's review of the Initial Assessment Study Report for NOS Indian Head; the Confirmation Study Report; available background information; the results of a supplemental site characterization, ecological risk assessment, and feasibility study conducted by Jordan; and discussions with U.S. Navy, Energy Systems, and personnel of ORRI.

2.1 Results of Previous Investigations

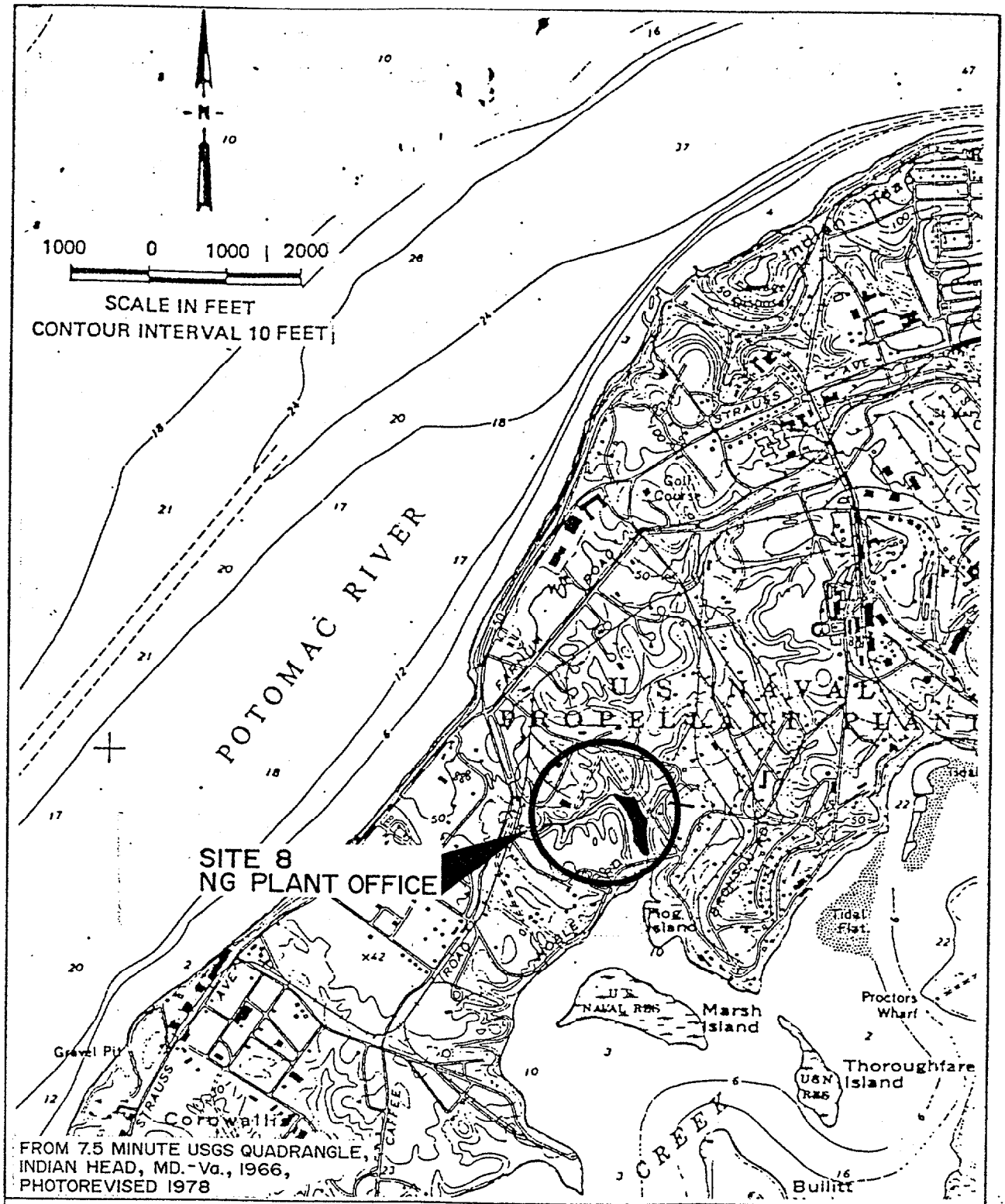
Mercury is used as a catalyst at NOS to perform a purity analysis of nitroglycerin. The nitrate-ester analysis is conducted at a laboratory in the NG Plant Laboratory Office (Building 766). For more than 20 years before 1981 wastewater contaminated with small amounts of mercury was discharged down floor drains into a storm drainage manhole outside the building and then into an open stream. The floor drain has been sealed off and these practices have been discontinued. The stream flows across 0.75 miles of Navy property into a tidal pond which drains into Mattawoman Creek. The drainage has a total relief of 36 feet and discharges into a tidally influenced area of Mattawoman Creek. This ditch and tidal pond comprise Site 8, the location of which is presented on Figure 2.

Sediment samples collected during the Confirmation Study in 1985 from the stream just below Building 766 contained mercury concentrations as high as 1,100 mg/kg. Lower concentrations were found in sediments as far as 2,500 feet down gradient from the NG Plant Laboratory Office. Concentrations in sediments at the outfall into Mattawoman Creek (3.5 mg/kg) were still above background levels (0.45-1.5 mg/kg). Seven water samples from within the creek also revealed elevated total mercury concentrations, up to 0.17 mg/l. Of the seven water samples collected in January 1984, the three from the stream exceeded the USEPA drinking water limit of 0.002 mg/l; concentrations of the remaining four samples collected from tidal pond were below this limit. These water samples were all collected during cold water conditions (January 1984).

Estimates from the Confirmation Study suggested that 200 to 500 pounds of mercury may reside in the soils along the drainage area and in sediments of the stream and pond. The study also suggested that the tidal pond probably traps much of the sediment from the drainage and that more than 95 percent of the mercury at Site 8 is located in the tidal pond sediments.

A supplemental site investigation was conducted at Site 8 in October 1987 as part of Jordan's Feasibility Study. The purpose of that investigation was to improve the knowledge about chemical and physical conditions at the site. The results of this investigation indicate that approximately 23,100 cubic yards of sediment exist in the stream bed and tidal pond. A relatively steep topographical gradient and a narrow stream bed exist in the upper reaches of the stream. Therefore, relatively little sediment exists in the upper reaches of the stream bed.

Results of Jordan's site investigation suggest that approximately 55 pounds of mercury remain in sediments at the site. The volume of sediments containing a



FROM 7.5 MINUTE USGS QUADRANGLE, INDIAN HEAD, MD.-Va., 1966, PHOTOREVISED 1978

FIGURE 2

SITE 8 LOCATION

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mercury concentration of ≥1 mg/kg and ≥5 mg/kg is estimated to be 7,555 and 2,276 cubic yards, respectively. Comparison of these results with the Confirmation Study indicate that the concentration of mercury in the sediments is decreasing with time or may have been inaccurately estimated in previous studies due to insufficient data.

2.2 Task 11 - Speciation of Mercury

In response to Task Order Authorization No. X-09, Amendment No. 2 of the SOW, this Speciation Study Work Plan was developed. The purpose of the Mercury Speciation Investigation is to evaluate the chemical species of mercury present in the sediments of the stream and tidal pond. Presented in the following sections is a detailed technical plan of action for the field sampling, laboratory analytical program, and evaluation of laboratory analytical results. Also appended are project plans for quality assurance, quality control, and health and safety.

2.2.1 Sediment and Soil Sampling

A field sampling trip will be conducted to collect four sediment samples and one soil sample for mercury speciation analyses. The location of the soil and sediment samples is presented on Figure 3. The rationale for each of the sampling locations is as follows:

- Sediment sample station SSD-1 is located in the upper reach of the stream in an area containing the highest concentration of total mercury;

- Sediment sample station SSD-2 is located at the mouth of stream where it enters the tidal pond. Sediments here contained relatively high concentrations of mercury and the form of mercury may be different from that detected in the upper reach of the stream;

- Sediment sample station SSD-3 is located in Mattawoman Creek and will serve to confirm that the mercury has not migrated out of the tidal pond or if it has, then in what form it is present; and

- Sediment sample station SSD-4 is located in the central portion of the tidal pond. The tidal pond is believed to act as a sink for the majority of the mercury contaminants present at the site.

- Soil sample station SSS-1 is located in the floodplain of the lower reach of the stream which has been flooded and the soils of which contain mercury as discovered in the Site Characterization Study conducted in October 1987.

At each sediment and soil sampling location, three separate sediment samples will be collected. Multiple samples are collected in order to provide an estimate of variability of results among samples collected from one sampling location. The results may provide some indication as to the spatial variability in mercury speciation at one general sampling location.

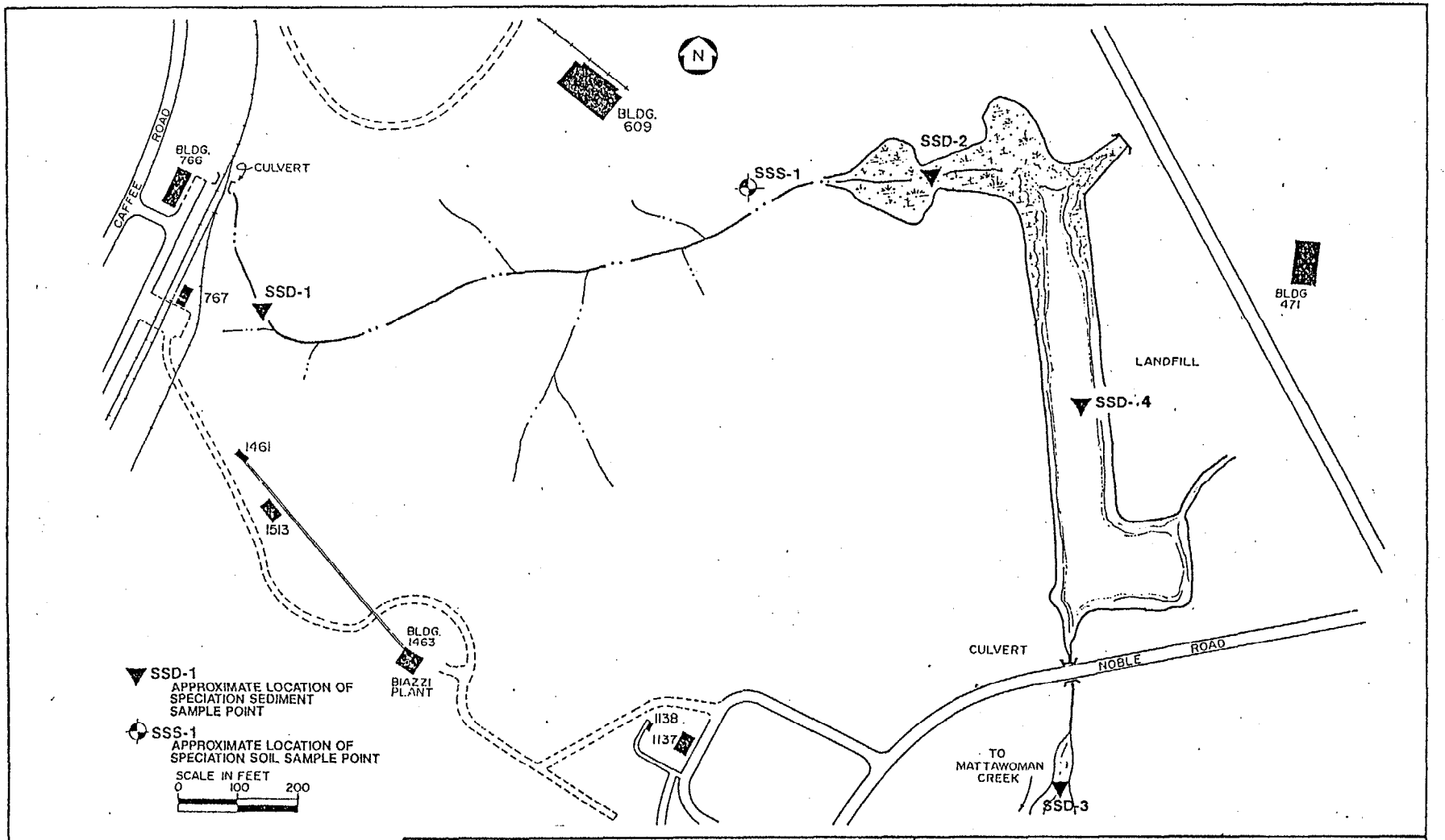

FIGURE 3

SOIL AND SEDIMENT SAMPLING LOCATIONS

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Soil and sediment samples will be collected using a manually driven 24-inch split-spoon sampler containing plastic sampling sleeves to avoid disturbing the sample any more than necessary prior to laboratory analysis. The sleeved split-spoon sampler will be driven 12 inches into the soil or sediment. Upon withdrawal, plastic caps will be placed on the ends of the sleeves and these will be taped in place. The sealed sleeves will be preserved at 4 degrees Celsius and transported, via overnight courier, to ORRI. Dr. Nat Revis will personally supervise the removal of the top 6 inches of each sample for analyses and the laboratory analysis of each sample.

Split-spoon samplers will be cleaned by soap and water wash and rinse, methanol rinse, and distilled water rinse prior to the commencement of sampling operations and before the collection of each sample.

Table 1 presents a summary of the sampling and analytical program for the mercury speciation study. The program includes collection of quality control samples. A sample bottle containing organic/metal free water will accompany all sample containers during all phases of sampling operations. This will serve as the Trip Blank. Rinsate collected from the final distilled water rinse of the split-spoon sampler, prior to collection of the last sample, will serve as the Rinse Blank. One soil sample, collected offsite in an area considered to be unaffected by mercury contamination at Site 8 will serve as a background soil sample. Two of the sediment core samples will be split in the laboratory for separate duplicate analyses and will serve as the field duplicate samples.

Table 1
Sampling and Analytical Program

Mercury Speciation
Naval Ordnance Station, Indian Head, Maryland

	Number of samples Site 8			Number of samples offsite		
Method	Sediment[1]	Soil[2]	Field duplicates	Rinse blank	Trip blank	Soil background
Nitric acid extractable mercury	12	3	2	1	1	1
Methyl mercury	12	3	2	1	1	1
Heat extractable mercury	12	3	2	1	1	1
Nitric, perchloric, and sulfuric acid extractable mercury	12	3	2	1	1	1

[1]Sediment samples equal four samples with three replications per sample.
[2]Soil samples equal a sample with three replications.

2.2.2 Laboratory Speciation Analysis

The laboratory speciation analysis conducted on the soil and sediment samples will include four procedures designed to extract different species of mercury. These four procedures include:

- total mercury as extracted by a combination of perchloric, nitric, and sulfuric acid;
- ionic mercury as extracted by water;
- bound ionic/elemental/organic mercury as extracted by nitric acid;
- mercuric sulfide as extracted by sodium sulfide;
- methyl mercury as extracted by toluene/benzene; and
- elemental mercury as extracted by heat.

The analyses will be performed and the results interpreted by Oak Ridge Research Institute. Unless otherwise specified herein, Jordan's Quality Assurance Project Plan, heretofore approved by CHESDIV for all IRP work, will be followed. Quality Assurance Level E will be established during all phases of sampling and analysis. The procedures for mercury analyses and quality assurance are presented in Appendix B - Task Quality Assurance Plan and Appendix C - Laboratory Quality Assurance Plan (LQAP).

Data will be validated by E.C. Jordan according to energy systems guidelines (August 1988) for metals and Level E data. The data validation including sample and method blank data; spike blanks; matrix spikes; duplicate data and calibration and linearity data will be included as deliverables in the Mercury Speciation Study Report.

2.2.3 Draft Mercury Speciation Study Report

Jordan will evaluate the interpreted analytical results and prepare a Draft Speciation Study Report as an addendum to the Supplemental Site Characterization Report dated December 1987. The addendum will present the sampling and analysis methodology, results and conclusions from the speciation study. Based on the results of this study recommendations will be made to Energy Systems and CHESDIV for proceeding with Task 12 - Treatability Studies on the soils and/or sediments. The report shall include all sample and QA/QC data developed in the investigation as appendices to the report.

2.2.4 Final Mercury Speciation Study Report

Based on review comments received from Energy Systems and CHESDIV, Jordan shall finalize the Draft Speciation Study Report and submit 15 copies of the report to Energy Systems within 10 working days of receipt of all comments.

3.0 PROGRAM MANAGEMENT

3.1 Management Approach

In undertaking the NOS Indian Head Mercury Speciation Investigation as part of the Feasibility Study/Remedial Design Task, Jordan will be responsible for management of the IRP technical program. Senior Jordan personnel will fill the key roles of Corporate Officer, Program Manager, Task Order Manager, staff level Contracts/Administration, Quality Assurance Coordinator, and Health and Safety Coordinator. The quality review board will be staffed by two senior Jordan staff members.

Jordan will take a holistic approach to problem-solving at NOS. None of the tasks/subtasks are "stand alone" activities; instead, they are interrelated. Consequently, the results of one work assignment have the potential to affect other work elements. As such, the activity leaders will work closely with the Task Order Manager and each other to create an appropriate level of synergism. The Task Order Manager for NOS Indian Head will be Tony Allen.

3.2 Personnel

3.2.1 Key Personnel

The designated roles for the IRP work at NOS are given below. The Task Order organization is shown on Figure 4.

- Corporate Officer. The Corporate Officer is William R. Adams, Jr., P.E., a vice president of Jordan. He is responsible for committing the corporate resources necessary to conduct the program work activities, for supplying corporate-level input for problem resolution, and for assisting the Program Manager and Task Order Manager as needed in project implementation.

- Program Manager. The Program Manager, Alan Ikalainen, is responsible for oversight and management of the overall multi-installation Navy IRP program for the Northeast Region. In this position, Mr. Ikalainen is able to perceive overall program needs, to promote technology and other information transfer between various IRP projects, and to direct resources, as appropriate, for effective and timely completion of program activities.

- Task Order Manager. The Task Order Manager for Indian Head NOS Task Order will be Tony Allen. He is responsible for evaluating the appropriateness and adequacy of the technical or engineering services provided for the NOS Task Order and in developing the technical approach and level of effort required to address each of the work plan tasks. He is also responsible for the day-to-day conduct of the work, including the integration of the input of supporting disciplines and subcontractors (i.e., field investigation and laboratory subcontractors). Mr. Allen will be reviewing the ongoing quality control during performance of the work, and the

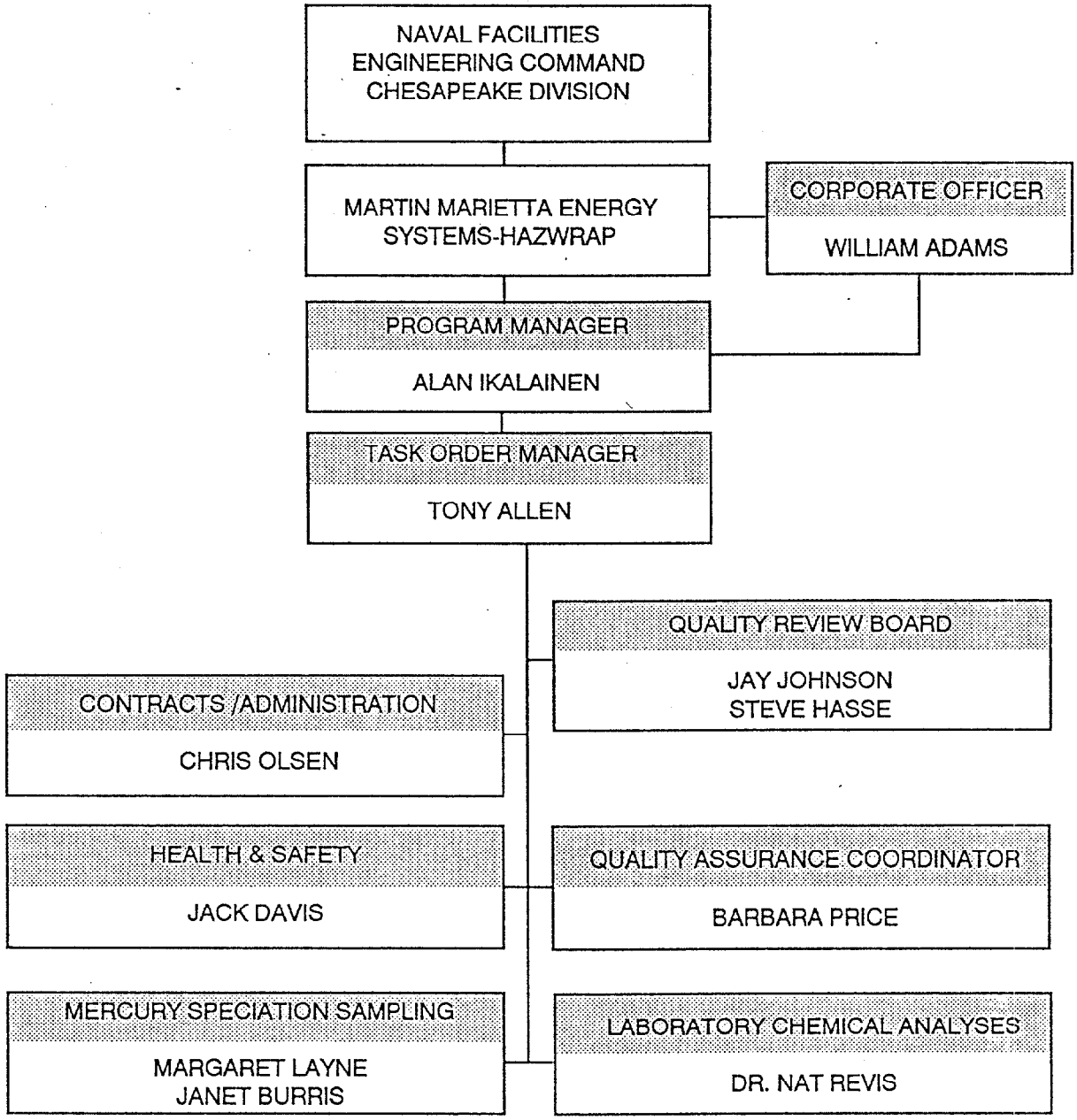

FIGURE 4

PROGRAM ORGANIZATION

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technical integrity of conclusions and recommendations, and the clarity and usefulness of all project work products.

Some specific responsibilities of this role include:

- overall technical responsibility for the project;
- initiating project activities;
- participating in the work plan preparation and staff assignments;
- identifying and fulfilling equipment and other resource requirements;
- monitoring task activities to ensure compliance with established budgets, schedules, and the scope of work; and
- regularly interacting with the IRP Management Team, the Corporate Officer and others, as appropriate, on the status of the project.

- Quality Review Board. A Quality Review Board, made up of senior technical staff from the Jordan team, will assist the Task Order Manager by providing review of the technical aspects of the project to assure that the services reflect the accumulated experience of the firms, that they are produced in accordance with corporate policy, and meet the intended needs of the IRP Management Team. The primary function of this board is to assure the application of technically sound methodologies and the development of interpretations and conclusions.

  Jay Johnson and Steve Haase will comprise the Jordan Technical Quality Review Board and will be actively involved in assuring the technical quality, and appropriateness of the conclusions and recommendations of the Mercury Speciation Investigation.

- Contracts and Administrative Manager. The staff-level position of Contracts and Administrative Manager is established because of the importance of day-to-day scope, schedule and budget monitoring for the NOS Task Order both within Jordan and between Jordan and the IRP Management Team. It is expected that program decisions will be occurring frequently. Therefore, it is necessary to anticipate and immediately implement the administrative actions (initiate internal work orders, follow-up on support needs, amend subcontracts, track cost-charges) to carry out the program plans. To achieve maximum efficiency and insure timeliness of response, Chris Olsen will be responsible for these areas to Jordan through the Task Order Manager and will be the principal communication link to the client for these areas. Three specific tasks for which Mr. Olson will be responsible are the following:

  - establish and oversee all subcontracts for support services;
  - prepare monthly technical/management/cost progress reports; and

- ensure that appropriate financial record and reporting requirements are met.

- Quality Assurance and Health and Safety Coordinator. The Task Order Manager is supported by a Quality Assurance Coordinator and a Health and Safety Coordinator. These staff-level positions will report to the Corporate Officer and the Task Order Manager, respectively. The Quality Assurance Coordinator will assure that appropriate IRP, NEESA and EPA protocols are followed on the NOS Task Order and will be responsible for the development of the quality assurance plan. The coordinator also works with the Program Manager/Task Order Manager to assure that established quality control procedures are implemented. The Health and Safety Coordinator is responsible for assuring that NOS project team complies with the company's Health and Safety Plan when conducting site visits. He is responsible for seeing that the Health and Safety Plan is developed for the implementation of the selected alternative.

Barbara Price of Jordan will serve as the QA Coordinator and Jack Davis will serve as Health and Safety Coordinator for the program respectively.

3.2.2 Team Members

Other key line positions in the NOS Task Order are the technical activity leaders, i.e., senior and/or most-experienced individual in each technical area of the project. These technical activity leaders as shown on the Project Organization Chart are as follows:

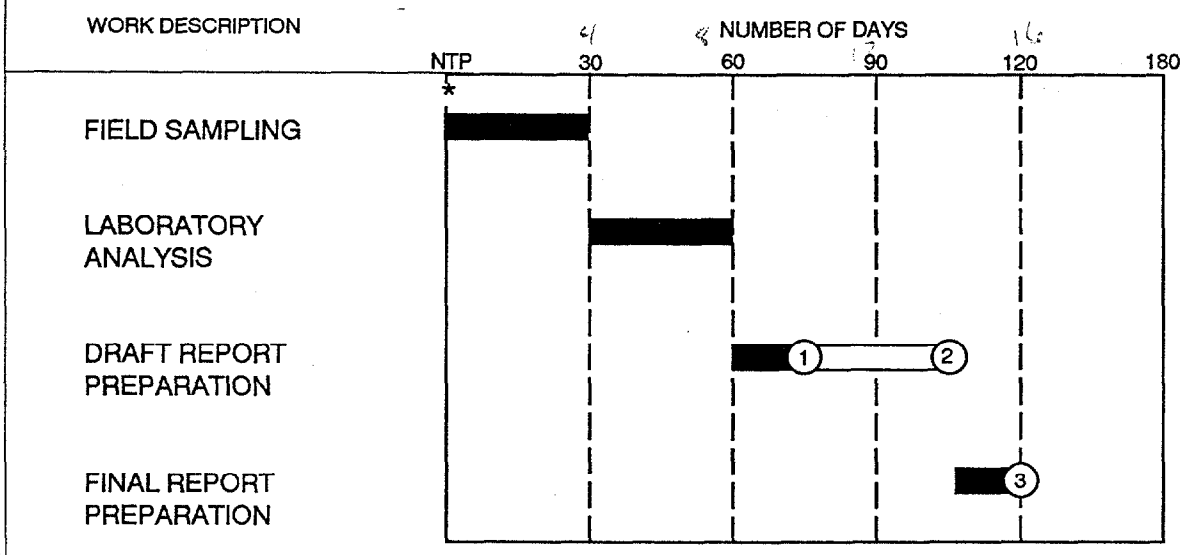
- Mercury Speciation Sampling. Margaret Layne, Professional Engineer, will be the technical leader for the mercury speciation sampling described in Task 11. She will be responsible for the soil/sediment sampling and preparation of Draft and Final Reports.

- Laboratory Chemical Analyses. Dr. Nat Revis will be responsible for all laboratory analytical activities. Dr. Revis will oversee the laboratory analysis to be conducted at ORRI and interpret and validate the results.

4.0 PROJECT SCHEDULE

The schedule for the IRP Mercury Speciation Investigation at NOS as described herein is shown in Figure 5. The schedule is given in terms of duration rather than for specific dates pending establishment of a specific date for Work Plan approval and initiation. The proposed schedule shows an estimated 17 weeks for project completion. This schedule is dependent upon a 2 week review turn around.

A proposed schedule for the completion of Task 12 - Treatability Study will be estimated when the specific Treatability studies are recommended at the completion of the mercury speciation investigation.



* NOTICE TO PROCEED

(1) SUBMITTAL OF DRAFT DELIVERABLES

(2) RECEIPT OF COMMENTS

(3) SUBMITTAL OF FINAL DELIVERABLES

DURATION OF TASK ACTIVITIES

DURATION OF ENERGY SYSTEMS/NAVY REVIEW

FIGURE 5

PROPOSED SCHEDULE

MERCURY SPECIATION STUDY TASK 11

NAVAL ORDINANCE STATION
INDIAN HEAD, MARYLAND

5.0 REFERENCES

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Klaassen, C.D., et al., 1986, Casarett and Doull's toxicology, the basic science of poisons: MacMillian Publishing Co., New York, 3rd Ed., 974 p.

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U.S. Environmental Protection Agency, Ambient Water Quality Criteria for Mercury - 1984, EPA 440/5-84-026, January 1985.

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6.0 LIST OF ACRONYMS

CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (Superfund)
CHESDIV	Chesapeake Division, Naval Facilities Engineering Command
CRZ	Contamination Reduction Zone
DOE	Department of Energy
EPA	U.S. Environmental Protection Agency
HASP	Health and Safety Plan
Hg	Mercury
HgS	Mercuric Sulfide
HSM	Health and Safety Manager
HSO	Health and Safety Officer
HSS	Health and Safety Supervisor
IRP	Installation Restoration Program
LQAP	Laboratory Quality Assurance Plan
NEODTC	Naval Explosive Ordnance Disposal Technology Center
NEESA	Naval Environmental Engineering Support Activity
NG	Nitroglycerin
NOS	Naval Ordnance Station
NSEOD	Naval School Explosive Ordnance Disposal Technology Center
ORRI	Oak Ridge Research Institute
OSHA	Occupational Safety and Health Administration
QA	Quality Assurance
QAP	Task Quality Assurance Plan
QAPP	Quality Assurance Project Plan
QC	Quality Control
ORRI	Oak Ridge Research Institute
RCRA	Resource Conservation and Recovery Act of 1976
SARA	Superfund Amendments and Reauthorization Act of 1986
SOW	Statement of Work
TLV	Threshold Level Value

APPENDIX A

SITE SPECIFIC HEALTH AND SAFETY PLAN

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1.0 GENERAL

SITE: Naval Ordnance Station Indian Head , Site 8

SITE OWNER/CONTACT: U.S. Navy/Thomas Woo (Department of Safety)

LOCATION: Indian Head, Maryland, 25 miles southwest of Washington, D.C.

PREPARED BY: Richard Gonzalez DATE: July 24, 1989

REVISION: _____

APPROVED BY: _____ DATE: _____

PROPOSED DATE(S) OF INVESTIGATION: To be determined.

BACKGROUND REVIEW: COMPLETE: _____ PRELIMINARY: _____

OVERALL HAZARD: SERIOUS: ___ MODERATE: ___ LOW: X UNKNOWN: ___

2.0 SITE CHARACTERIZATION AND ANALYSIS

2.1 SCOPE OF WORK

Field work to be conducted during the Mercury Speciation Study includes the collection of sediment samples and one floodplain soil sample. Soil and sediment samples will be collected using a manually driven split-spoon sampler. Collected samples will be preserved according to the site specific Quality Assurance Plan and submitted to Oak Ridge Research Institute for mercury speciation analysis.

2.2 Site History

For more than 20 years, prior to 1981, small amounts of mercury were rinsed down the lab sinks and floor drains within Building 766, NOS Indian Head. This mercury subsequently drained into a stream/tidal basin system which comprises Site 8. In 1981, these releases were ceased.

Estimates from a Confirmation Study conducted in January 1984 suggested that 200 to 500 pounds of mercury may reside in the soils along the drainage area and in sediments of the stream and pond. A supplemental site investigation conducted by Jordan in December 1987 suggested the existence of only 60 pounds of mercury in this same area. It appears that the mercury concentration in the sediments and soils is decreasing with time.

2.3 Site Risks

2.3.1 Health Hazards.

Personnel conducting field sampling activities associated with the Mercury Speciation Study may encounter mercury vapors and/or free mercury metal. Mercury (Hg) has a high vapor pressure and relatively high atmospheric concentrations can occur from negligible contamination. Inhalation is the prime route of exposure although dermal exposure should be avoided. Eighty percent of Hg vapor inhaled by humans is absorbed in the blood.

Acute poisoning from Hg vapor produces metallic taste, nausea, abdominal pain, vomiting, diarrhea, and headache. After a few days, salivary glands swell, gingivitis develops, teeth may loosen, and ulcers form on lips and gums, and death may result. The brain is the target organ for chronic damage from inhaled Hg vapor. Table A-1 presents some additional physical and toxicological characteristics of Hg.

TABLE A-1
CONTAMINANTS OF CONCERN

	Mercury, Solid	Mercury, Vapor
Approximate Odor Threshold (ppm)	--	--
TLV (ppm)	50 mg/m^3	0.05 mg/m^3
Physical Characteristics	Silvery white heavy liquid	High vapor pressure
Dermal Toxicity	Skin irritant	Skin irritant
Remarks	Cumulative neurotoxin	Cumulative neurotoxin

The TLV for mercury vapor is 0.05 mg/m^3. Personnel conducting the August 1984 sampling trip measured Hg vapor levels in excess of the TLV; if the readings on the Jerome mercury vapor analyzer were correct. Interferences for the Jerome analyzer are H_2 and acidic gases. There are aboveground nitric and sulfuric acid storage tanks within 500 feet of the stream which may produce acid gases through venting.

Air quality measurements conducted during Jordan's December 1987 sampling trip did not indicate the presence of mercury vapor levels approaching the TLV.

2.3.2 Safety Hazards

Personnel participating in the sampling trip will be exposed to a minimal degree of physical hazards. These hazards can most likely be described within one of two broad categories; accidents and weather related stress.

2.3.2.1 Accidents. Minor cuts, bruises, muscle pulls, etc., are inherent hazards associated with any field sampling operation.

The tidal pond at Site 8 is, typically, 8 to 20 inches deep with a soft muck bottom. This bottom will not support a man's weight. Therefore, there is a remote potential for drowning.

Steep, leaf covered slopes adjacent to the tidal stream cause a distinct potential for slips and falls.

2.3.2.2 Weather Related Stress. It is anticipated that field sampling activities will be conducted during the fall/winter months of 1989. Therefore, hypothermia may become a significant factor for site operations.

2.3.3 Conclusion/Risk Assessment.

The TLV for mercury vapor in air was not exceeded during Jordan's field sampling activities conducted in December 1987. Therefore, it is concluded that the potential for exposure to harmful levels of Hg vapor is low. The potential for accidents at Site 8 is low to moderate.

2.4 Initial Site Entry

2.4.1 Initial Levels of Protection.

Task	Minimum Level
Soil/Sediment sampling	Level D. Upgrade to level B if Hg analyzer exceeds 0.05 mg/m^3.

2.4.2 Initial Monitoring

Mercury vapor monitoring will be conducted utilizing a Jerome analyzer. A Hg vapor level of 0.05 mg/m^3 will require evacuation upwind and upgrade to level B.

3.0 SITE CONTROL

3.1 Zonation

The site itself will normally be divided into three zones: the majority of the work area will be considered the Exclusion Zone, with limited areas serving as Support Zone, and an area for decontamination called the Contamination Reduction Zone (CRZ).

4.0 ENGINEERING CONTROLS, WORK PRACTICES, AND PERSONAL PROTECTIVE EQUIPMENT

4.1 Engineering Controls

A Jerome Hg vapor analyzer will monitor the ambient air during all on-site activities. Mercury vapor above 0.05 mg/m^3 will require evacuation upwind and upgrade to level B.

An air monitoring survey will be conducted prior to any site investigation.

4.2 Work Practices

Workers will be expected to adhere to the established safe work practices. Work at the site will be conducted according to established protocol and guidelines for the safety and health of all involved.

- In any unknown situation, always assume the worst conditions and plan responses accordingly.

- Use the buddy system.

- Establish and maintain communication.

- Because no personal protective equipment is 100-percent effective, all personnel must minimize contact with contaminated materials. Do not sit or kneel on the ground in the Exclusion Zone or CRZ. Avoid standing in or walking through puddles or stained soil.

- Smoking, eating, or drinking in the work area and before decontamination will not be allowed.

- Avoid heat and other work stresses related to wearing protective gear. Work breaks should be planned to prevent stress-related accidents or fatigue.

- Personnel must be observant of not only one's own immediate surroundings, but also those of others. A team effort is needed to notice and warn of impending dangerous situations.

- Contact lenses are not allowed to be worn on site; if corrosive or lachrymose substances enter eyes, proper flushing is impeded.

- All facial hair that interferes with the face piece fit, must be removed prior to donning a respirator at all sites requiring level C or B protection.

- Rigorous contingency planning and dissemination of plans to all personnel minimizes the impact of rapidly changing safety protocols in response to changing site conditions.

- Extreme caution should be exercised when proceeding up and down steep inclines and when working on and around the tidal pond.

4.3 Personal Protective Equipment

Coated Tyveks
Chemical-resistant safety boots/shoes
Disposable boot covers
Inner chemical-resistant gloves
Outer chemical-resistant gloves
Hard hat

4.3.1 Other Protective Equipment

Life jackets will be on hand when sampling activities occur on the tidal pond.

5.0 MONITORING

5.1 Air Sampling.

Dosimeter badges

5.2 Other Monitoring Equipment.

Jerome mercury vapor detector

5.3 Contaminant Levels for Modification of Protective Equipment.

A mercury vapor level of 0.05 mg/m^3, as measured by the Jerome Mercury Vapor Analyzer will necessitate evacuation upwind and upgrade to level B.

6.0 DECONTAMINATION/DISPOSAL

All personnel and/or equipment leaving contaminated site areas are subject to decontamination, which occurs in the CRZ.

6.1 Personnel Decontamination.

Decontamination procedures are followed by all personnel leaving hazardous waste sites. Under no circumstances (except emergency evacuation) will personnel be allowed to leave the site prior to decontamination.

Disposable items (i.e., Tyvek coveralls, inner gloves, and latex overboots) will be changed on a daily basis unless there is a reason to change sooner.

Pressurized sprayers or other designated equipment will be available in the decontamination area for wash down and cleaning of personnel, samples, and equipment.

6.2 Small Equipment Decontamination.

A decontamination station for personnel sampling equipment will be established at the site. Between sampling locations, sampling equipment will be brought to this area for washing. Temporary decontamination stations will be established at individual sampling sites to more conveniently accommodate limited decontamination operations. The Hg analyzer will be kept in a plastic bag as much as possible.

6.3 Disposal of Decontaminated Materials.

Decontamination fluids will be water, detergents/water, and methanol. Water and detergent/water mixtures will be disposed of via discharge to the NOS's sanitary wastewater treatment system. Methanol will be containerized and disposed at an appropriate treatment and/or disposal facility.

7.0 EMERGENCY/CONTINGENCY PLAN

This section identifies the emergency contingency planning undertaken for operations at this site. Other sections provide further information to be used under emergency conditions. Refer to Appendix A-3 for emergency telephone numbers, routes to emergency medical facilities and emergency signals.

7.1 Evacuation.

Evacuation upwind and upgrade to level B will be required if the ambient air mercury vapor concentration, as measured by a Jerome Mercury Vapor Analyzer, reaches 0.05 mg/m^3.

8.0 OTHER

8.1 Illumination.

All field sampling activities will be conducted during daylight hours. Downrange operations will halt in time to permit personnel and equipment to exit the Exclusion Zone and proceed through decontamination before dusk.

8.2 Sanitation.

Sanitation facilities located in the NG plant office will be utilized, as necessary, by Jordan personnel.

9.0 ADMINISTRATIVE

Personnel authorized to participate in downrange activities at this site have been reviewed and certified for site operations by the Site Manager and HSS. Certification involved the completion of appropriate training, a medical examination, and a review of this site-specific HASP. All persons entering the site must utilize the buddy system, and check in with the Site Manager and/or HSO before proceeding downrange.

Certified E.C. Jordan Co. Team Personnel:

HSO: Janet Burris _____

Margaret Layne _____

Other Certified Personnel:

_____ _____

_____ _____

* Current First Aid Training
\+ Current CPR Training

9.1 Field Team Review.

I have read and reviewed the HASP, understand the information contained, and agree to comply.

NAME: _____ Date: _____

SITE/PROJECT: _____

9.2 Medical Data Sheet.

This Medical Data Sheet will be completed by all on-site personnel and will be kept in the Support Zone during site operations. It is in no way a substitute for the Medical Surveillance Program requirements consistent with the E.C. Jordan Environmental-Corporate Health and Safety Program for Hazardous Waste Sites. This data sheet will accompany any personnel when medical assistance or transport to hospital facilities is required. If more information is required, use the back of this sheet.

Project: _____

Name: _____

Address: _____

Home Phone: Area Code ( ) _____

In case of emergency, contact: _____

Address: _____

Telephone: Area Code ( ) _____

Do you wear contacts? ( ) Yes ( ) No

Allergies: _____

List medication taken regularly. _____

Particular sensitivities: _____

Previous/recent illnesses or exposures to hazardous chemicals: _____

_____

Name of personal physician: _____

Telephone: Area Code ( ) _____

HASP APPENDIX A-1

CHRIS DATA SHEETS/MATERIAL SAFETY DATA SHEETS
FOR CONTAMINANTS OF CONCERN

MERCURY

MCR

Common Synonyms	Liquid	Silver	Odorless
Quicksilver	Sinks in water.		

AVOID CONTACT WITH LIQUID. Keep people away.
Stop discharge if possible.
Isolate and remove discharged material.
Notify local health and pollution control agencies.

Fire	Not flammable.
Exposure	CALL FOR MEDICAL AID. **LIQUID** Effects of exposure may be delayed.
Water Pollution	HARMFUL TO AQUATIC LIFE IN VERY LOW CONCENTRATIONS. May be dangerous if it enters water intakes. Notify local health and wildlife officials. Notify operators of nearby water intakes.

1. RESPONSE TO DISCHARGE

(See Response Methods Handbook)

Should be removed
Chemical and physical treatment

2. LABEL

2.1 **Category:** None

3. CHEMICAL DESIGNATIONS

3.1 **CG Compatibility Class:** Not listed
3.2 **Formula:** Hg
3.3 **IMO/UN Designation:** Not listed
3.4 **DOT ID No.:** 2809
3.5 **CAS Registry No.:** 7439-97-6

4. OBSERVABLE CHARACTERISTICS

4.1 **Physical State (as shipped):** Liquid
4.2 **Color:** Silvery
4.3 **Odor:** None

5. HEALTH HAZARDS

5.1 **Personal Protective Equipment:** Avoid contact of liquid with skin. For vapor use chemical cartridge (Hopcalite) respirator.
5.2 **Symptoms Following Exposure:** No immediate symptoms. As poisoning becomes established, slight muscular tremor, loss of appetite, nausea, and diarrhea are observed. Psychic, kidney, and cardiovascular disturbances may occur.
5.3 **Treatment of Exposure:** Consult a doctor.
5.4 **Threshold Limit Value:** 0.05 ng/m³
5.5 **Short Term Inhalation Limits:** Data not available
5.6 **Toxicity by Ingestion:** No immediate toxicity
5.7 **Late Toxicity:** Development of mercury poisoning
5.8 **Vapor (Gas) Irritant Characteristics:** None
5.9 **Liquid or Solid Irritant Characteristics:** None
5.10 **Odor Threshold:** Not pertinent
5.11 **IDLH Value:** 28 mg/m³

6. FIRE HAZARDS

6.1 **Flash Point:** Not flammable
6.2 **Flammable Limits in Air:** Not flammable
6.3 **Fire Extinguishing Agents:** Not pertinent
6.4 **Fire Extinguishing Agents Not to be Used:** Not pertinent
6.5 **Special Hazards of Combustion Products:** Not pertinent
6.6 **Behavior in Fire:** Not flammable
6.7 **Ignition Temperature:** Not flammable
6.8 **Electrical Hazard:** Not pertinent
6.9 **Burning Rate:** Not flammable
6.10 **Adiabatic Flame Temperature:** Data not available
6.11 **Stoichiometric Air to Fuel Ratio:** Data not available
6.12 **Flame Temperature:** Data not available

7. CHEMICAL REACTIVITY

7.1 **Reactivity With Water:** No reaction
7.2 **Reactivity with Common Materials:** No reaction
7.3 **Stability During Transport:** Stable
7.4 **Neutralizing Agents for Acids and Caustics:** Not pertinent
7.5 **Polymerization:** Not pertinent
7.6 **Inhibitor of Polymerization:** Not pertinent
7.7 **Molar Ratio (Reactant to Product):** Data not available
7.8 **Reactivity Group:** Data not available

8. WATER POLLUTION

8.1 **Aquatic Toxicity:**
0.5-1 ppm/48 hr/caragius ardium/TL_m/fresh water
0.29 ppm/48 hr/marine fish/TL_m/salt water
8.2 **Waterfowl Toxicity:** Data not available
8.3 **Biological Oxygen Demand (BOD):** None
8.4 **Food Chain Concentration Potential:** Mercury concentrates in liver and kidneys of ducks and geese to levels above FDA limit of 0.5 ppm. Muscle tissue usually well below the limit.

9. SHIPPING INFORMATION

9.1 **Grades of Purity:** Pure
9.2 **Storage Temperature:** Ambient
9.3 **Inert Atmosphere:** No requirement
9.4 **Venting:** Open

10. HAZARD ASSESSMENT CODE

(See Hazard Assessment Handbook)

A-X

11. HAZARD CLASSIFICATIONS

11.1 **Code of Federal Regulations:** ORM-B
11.2 **NAS Hazard Rating for Bulk Water Transportation:** Not listed
11.3 **NFPA Hazard Classification:** Not listed

12. PHYSICAL AND CHEMICAL PROPERTIES

12.1 **Physical State at 15°C and 1 atm:** Liquid
12.2 **Molecular Weight:** 200.59
12.3 **Boiling Point at 1 atm:** 675°F = 357°C = 630°K
12.4 **Freezing Point:** −38.0°F = −38.9°C = 234.3°K
12.5 **Critical Temperature:** 2664°F = 1462°C = 1735°K
12.6 **Critical Pressure:** 23,300 psia = 1587 atm = 160.8 MN/m²
12.7 **Specific Gravity:** 13.55 at 20°C (liquid)
12.8 **Liquid Surface Tension:** 470 dynes/cm = 0.470 N/m at 20°C
12.9 **Liquid Water Interfacial Tension:** 375 dynes/cm = 0.375 N/m at 20°C
12.10 **Vapor (Gas) Specific Gravity:** Not pertinent
12.11 **Ratio of Specific Heats of Vapor (Gas):** Not pertinent
12.12 **Latent Heat of Vaporization:** Not pertinent
12.13 **Heat of Combustion:** Not pertinent
12.14 **Heat of Decomposition:** Not pertinent
12.15 **Heat of Solution:** Not pertinent
12.16 **Heat of Polymerization:** Not pertinent
12.25 **Heat of Fusion:** 2.7 cal/g
12.26 **Limiting Value:** Data not available
12.27 **Reid Vapor Pressure:** Data not available

NOTES

HASP APPENDIX A-2

OSHA JOB SAFETY AND HEALTH PROTECTION POSTER

JOB SAFETY & HEALTH PROTECTION

The Occupational Safety and Health Act of 1970 provides job safety and health protection for workers by promoting safe and healthful working conditions throughout the Nation. Requirements of the Act include the following:

Employers

All employers must furnish to employees employment and a place of employment free from recognized hazards that are causing or are likely to cause death or serious harm or employees. Employers must comply with occupational safety and health standards issued under the Act.

Employees

Employees must comply with all occupational safety and health standards, rules, regulations and order issued under the Act that apply to their own actions and conduct on the job

The Occupational Safety and Health Administration (OSHA) of the U.S. Department of Labor has the primary responsibility for administering the Act. OSHA issues occupational safety and health standards, and its Compliance Safety and Health Officers conduct jobsite inspections to help ensure compliance with the Act.

Inspection

The Act requires that a representative of the employer and a representative authorized by the employees be given an opportunity to accompany the OSHA inspector for the purpose of aiding the inspection.

Where there is no authorized employee representative, the OSHA Compliance Officer must consult with a reasonable number of employees concerning safety and health conditions in the workplace.

Complaint

Employees or their representatives have the right to file a complaint with the nearest OSHA office requesting an inspection if they believe unsafe or unhealthful conditions exist in their workplace. OSHA will withhold, on request, names of employees complaining.

The Act provides the employees may not be discharged or discriminated against in any way for filing safety and health complaints or for otherwise exercising their rights under the Act.

Employees who believe they have been discriminated against may file a complaint with their nearest OSHA office within 30 days of the alleged discrimination.

Citation

If upon inspection OSHA believes an employer has violated the Act, a citation alleging such violations will be issued to the employer. Each citation will specify a time period within which the alleged violation must be corrected.

The OSHA citation must be prominently displayed at or near the place of alleged violation for three days, or until it is corrected, whichever is later, to warn employees of dangers that may exist there.

Proposed Penalty

The Act provides for mandatory penalties against employers of up to $1,000 for each serious violation and for optional penalties of up to $1,000 for each nonserious violation. Penalties of up to $1,000 per day may be proposed for failure to correct violations within the proposed time period. Also, any employer who willfully or repeatedly violates the Act may be assessed penalties of up to $10,000 for each such violation.

Criminal penalties are also provided for in the Act. Any willful violation resulting in death of an employee, upon conviction, is punishable by a fine of not more than $10,000, or by imprisonment for not more than six months, or by both. Conviction of an employer after a first conviction doubles these maximum penalties.

Voluntary Activity

While providing penalties for violations, the Act also encourages efforts by labor and management, before an OSHA inspection, to reduce workplace hazards voluntarily and to develop and improve safety and health programs in all workplaces and industries. OSHA's Voluntary Protection Programs recognize outstanding efforts of this nature.

Such voluntary action should initially focus on the identification and elimination of hazards that could cause death, injury, or illness to employees and supervisors. There are many public and private organizations that can provide information and assistance in this effort, if requested. Also, your local OSHA office can provide considerable help and advice on solving safety and health problems or can refer you to other sources for health such as training.

Consultation

Free consultative assistance, without citation or penalty, is available to employers, on request, through OSHA supported programs in most State departments of labor or health.

Augusta Area Office
Federal Bldg. & P.O. Box 40
Western Ave., Rm. 121
Augusta, Maine 04330
Telephone (207) 622-8417

More Information

Additional information and copies of the Act, specific OSHA safety and health standards, and other applicable regulations may be obtained from your employer or from the nearest OSHA Regional Office in the following locations:

Atlanta, Georgia
Boston, Massachusetts
Chicago, Illinois
Dallas, Texas
Denver, Colorado
Kansas City, Missouri
New York, New York
Philadelphia, Pennsylvania
San Francisco, California
Settle, Washington

Telephone numbers for these offices, and additional area office locations, are listed in the telephone directory under the United States Department of Labor in the United States Government listing.

Washington, D.C.
1985
OSHA 2203

William E. Brock, Secretary of Labor

U.S. Department of Labor

Occupational Safety and Health Adminstration

Under provisions of Title 29, Code of Federal Regualtions, Part 1903.2(a)(1) employers must post this notice (or a facsimile) in a conspicuous place where notices to employees are customarily posted.

HASP APPENDIX A-3

EMERGENCY INFORMATION
(to be posted in the trailer)

(This appendix must always be the last one. This is to allow for easy removal in order to post the information in the trailer. If the site does not have a trailer, it also allows the information to be found quickly in an emergency situation.)

EMERGENCY ROUTES

HOSPITAL: From Building 766, proceed on Coffee Road, go right onto Hanlon Road, left onto Strauss Avenue, left onto Jackson Road, right onto West Wilson Road to Building 1600.

SITE EVACUATION: In the remote possibility that the site needs evacuating, Jordan personnel will notify Thomas Woo of base security and evacuate through the nearest gate.

POST IN TRAILER

EMERGENCY TELEPHONE NUMBERS

(Local) Police Department	911 or (301) 934-2214
(Local) Rescue Service	911 or (301) 934-2222
Physician's Memorial Hospital	(301) 645-0100
NOS Clinic	(301) 743-4449
(Local) Fire Department	911 or (301) 934-2214
Off-site Emergency Services	911
National Poison Control Center	(800) 492-2414
Maine Poison Control Center	(207) 871-2950
National Response Center	(800) 424-8802
Regional USEPA Emergency Response	(800) 414-8802
Chemical Manufacturer's Association Chemical Referral Center	(800) 262-8200
Site HSO: Janet Burris	(904) 656-1293
Site Manager: Margaret Layne	(904) 656-1293
Regional HSS: C.E. Sundquist	(207) 775-5401 ext 755
C.E. Environmental HSM: J.A. Reynolds	(201) 992-2323 ext 264
E.C. Jordan (Maine)	(207) 775-5401
E.C. Jordan (Florida)	(904) 656-1293
E.C. Jordan (Massachusetts)	(617) 245-6606
Envirologic Data	(207) 773-3020
Thomas Woo NOS Safety	(301) 743-4320

APPENDIX B

TASK QUALITY ASSURANCE PLAN

Task: Naval Ordnance Station Indian Head, Mercury Speciation Investigation

Task Objectives:

- Speciate and quantify mercury compounds in sediment and soils at Site 8, NOS Indian Head, Maryland

Subtasks:	Standard Protocol Selected
Sediment Sampling	Energy Systems Position Paper No. 1 (attached) using modified California sampler
Soil Sampling	Energy Systems Position Paper No. 1 (attached) using modified California sampler
Laboratory Analysis	Oak Ridge Research Institute Laboratory Quality Assurance Plan for the Study of Mercury Speciation at Environmental Sites (Appendix C)
Mercury Speciation Data Validation Level E	Requirements for Quality Control of Analytical Data; HAZWRAP, DOE/HWP-65; HW-RAP-102-1, August 1988

Task Organization

Name	Function
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T. Allen	Technical Leader
M. Layne	Field Operations Leader
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Subcontractor	
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Contacts	Function
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ANALYTICAL DATA

Matrix	Parameter	Analytical Method*
Sediment/Soil	Total mercury	Perchloric/nitric/sulfuric acid extraction
	Elemental mercury	Loss upon heating
	Total mercury minus mercuric sulfide species	Nitric acid extraction
	Inorganic mercury	Sodium sulfide extraction
	Methyl mercury	Toluene extraction

*Oak Ridge Research Institute. N.D. Standard Operating Procedures and QA/QC Programs, Isolation and Analysis of the Different Species of Mercury in Environmental Samples.

SAMPLE IDENTIFICATION/CROSS REFERENCE

Sample ID Number	Sample Location
SSD-01-A SSD-01-B SSD-01-C SSD-01-A-DUP	Upper reach of stream, depth interval 0-6 inches
SSD-02-A SSD-02-B SSD-02-C	Confluence of stream and tidal basin, depth interval 0-6 inches
SSD-03-A SSD-03-B SSD-03-C	Mattawoman Creek, depth interval 0-6 inches
SSD-04-A SSD-04-B SSD-04-C	Tidal pond, depth interval 0-6 inches
SSD-05-A SSD-05-B SSD-05-C	Floodplain in lower reach of stream, depth interval 0-6 inches
RB-1	Equipment Blank 1, taken in conjunction with sediment samples 08-SD-X01-X01-01-DX
SSS-06	Background soil sample, collected offsite in area unaffected by mercury contamination
FB-1	Trip blank, to accompany all samples at all times

POSITION PAPER NO. 1

USE OF LINERS IN SUBSURFACE SOIL SAMPLING

1.0 INTRODUCTION

The object of most hazardous waste investigations is to determine the concentration and distribution of contaminants in surface soils, subsurface soils, groundwater, surface water, sediments, and air. This object can be further refined and defined to say that the object of these types of investigations is to determine the concentration and distribution of contaminants in the specific media being investigated. The focus of this position paper is to achieve the latter with regard to subsurface soil sampling, e.e., to determine the actual concentration of contamination in the media in such a manner that the number reflects actual in-situ concentrations and not a number that has been affected by the sampling methodology, decontamination procedures, analytical methodology, packaging, or shipping procedures.

This position paper specifies a recommended methodology for sampling subsurface soils. In addition, a rationale is given, and the conditions under this methodology is selected.

2.0 SUBSURFACE SOIL SAMPLING METHODOLOGY

The standard, nationally accepted methodology for the sampling of subsurface soils is through the use of a device referred to as a split spoon sampler. This device has other names depending upon the area of the country or manufacturer. For example, or CME sampler. Although all of these instruments are not exactly the same, they perform the same function.

The methodology for subsurface soil sampling that is most widely accepted is listed as ASTM D-1586-84, "standard Method for Soil Penetration Testing and Split Barrel Sampling". This methodology was originally developed by engineers for accomplishing the following goals:

Through the use of this methodology, a measurement could be taken to approximate the load carrying capabilities of the subsurface soils. This is accomplished through the use of a 140-lb hammer, dropped 30 inches which drives the split barrel sampler into the subsurface. By counting the number of blows it takes to progress over a six inch distance, these measurements could be related to subsurface soil loading capability, soil competency, cementation, etc. In addition, the sampler itself could retrieve a sample of the soil horizon being tested so that engineering tests could be performed such as soil classification, sieve analysis, liquid and plastic index determinations, etc. In conjunction with these tests, the split barrel samplers were also equipped with liners so that an in-situ sample could be taken of the subsurface. This sample, remaining in the liner, could then be sent to the soil laboratory so that rising, falling, or constant head infiltration tests (3-void Ratio Infiltration test) could be run on the "undistriburbed" sample.

With the advent of RCRA (1976) and Superfund (1980), an immediate regulatory need precipitated the requirement to collect subsurface soil samples for analytical purposes. Since none had been developed to specifically address this issue, the above ASTM method was adapted and modified to accomplish this goal since it had already been utilized as a proven technology by engineers. However, the adaption and modification of this methodology to the hazardous waste industry is not without problem areas.

3.0 PROBLEM AREA IN SUBSURFACE SOIL SAMPLING FOR ANALYTICAL PURPOSES

One of the major problem area in collecting subsurface soil samples via a split spoon sampler is the collection of these samples for the purposes of analyzing for volatile organics. The problem arises once the sampler is retrieved from the borehole. To restate the goal of the sample, it is the intent of the sampling effort to obtain a sample of the subsurface soil and analyze it to ascertain the concentration of the contaminant(s) which reflects actual field contamination levels.

A common procedure has been as follows: once the sampler is retrieved, the top half of the split spoon sampler is removed and a portion of the sample is extracted and placed in a 40 ml VOA vial. The placement of this sample in the vial has been with a spoon, spatula, or other like device. It has been shown by many analytical groups, including our own ORNL, that exposure of the sample during this procedure will cause most of the volatile organics to be lost to the atmosphere. Therefore, the results of the chemical analysis will show a concentration of volatile organics in the sample which will not represent actual field conditions, in point of fact it will show a significantly lower concentration. The actual amount of volatile organics lost due to this procedure differs greatly, depending upon the amount of disturbance of the sample by placing it into the 40-ml vial.

4.0 RECOMMENDED PROCEDURE FOR SUBSURFACE SOIL SAMPLING FOR VOA ANALYSIS

Based on the above information it then becomes very important to develop a method for field use to reduce the potential of losing volatile organics in these subsurface soil samples. The methodology that has been successful in the past few years, and the one that is being recommended here, has been through the use of liners in split spoon samples. These liners are decontaminated and placed into the split spoon samplers. When the split spoons are retrieved from the borehole, the sample is not extracted from the liner but; instead, the ends of the liners are sealed with Teflon end caps, wrapped in aluminum foil, and taped closed. The liner with contained sample is then ready to be sent to the lab for a chemical analysis.

Liners are made of several different materials and all are currently available. Some of these materials are aluminum, stainless steel, brass, cardboard, and two or three varieties or organic compounds, such as polybuterate or lexan. The material must commonly used for volatile organic analysis is brass.

The following is an example of the use of liners in subsurface soil sampling.

Normally when subsurface soil samples are collected, more than one suite of compounds will be analyzed. For example, you may require a VOA metals, and BNA sample. In addition, you may require a stratigraphic sample. Split spoon samples come in several different lengths and several different diameters. For the sake of this example a 2-foot long, 2-inch O.D. split spoon will be used. Brass liners would then be placed inside the split spoon will be used. Brass liners would then be placed inside the split spoon. The number and lengths of the liners are dependent upon the analytical requirements. In this example, four 6-inch liners are selected. The liners are labeled A through D with Liner A representing the top of the split spoon (sampling the top of the two foot sampling horizon) and Liner D representing the bottom of the split spoon nearest the cutting end. Once retrieved from the borehole, Liner C is immediately sealed on both ends with Teflon end caps. This represents the VOA sample. The material in liners A, B, and D are extruded. Stratigraphic information is obtained from these extruded liners. The sample from Liner D is used for metals analysis since it has had the least contact with the brass liners, and Liner B is used for the BNA samples and Liner A is used for lithology or stratigraphy. (NOTE: Liner A should not be used for analytical purposes since it may contain cave-in material).

If low level metals analysis is a requirement and the use of brass liners is questionable, then the bottom liner could be of a nonmetal material. However, it is believed that the activity of collecting metals samples in brass liners is not a significant problem since in most cases the level of metals contamination we would be concerned with in the field would be greater than 5- - 100 ppm and the amount contributed by the brass liners would not significantly alter the total metals concentration.

In some instances, whether it is for technical reasons or to satisfy regulatory requirements, it may be preferential to composite samples (with the exception of samples for VOAs). This technique can be accommodated in the above scenario by removing the samples from the A, B, and D liners, compositing these samples, and splitting them to form the metals samples, BNA samples, or any other analytical samples you may require. The decision to composite or not to composite is one which must be made by the hydrogeologist based on the above conditions (ie.e., technical or regulatory).

There may also be a requirement to split samples. This task cannot be accomplished, as a general rule, with 2-inch O.D. split spoons. This task can normally only occur with 3-inch O.D. split spoons or larger. Only with these size spoons can a volume of material be obtained to sufficiently fill all of the appropriate sampling receptacles. For split samples, the materials must always be composited before there are within the liners, therefore, two successive liners may be used to approximate split samples for VOAs.

In general, careful selection of liners is warranted based on the analytical requirements of the investigation. In addition, all of the metallic liners can be decontaminated and reused. In general, the organic-based liners cannot be reused due to the uncertainty of the decontamination process.

A common argument against the use of liners for VOA sampling is that the chemist will lose a certain amount of the volatile organics. This argument is not entirely valid since the sample is in tact from the field to the laboratory and, therefore potentially, the sample will only be distributed once rather than twice. Second, the chemist, due the fact that they are located in a laboratory, can better minimize the loss of volatiles since the extraction of the sample from the vial or liner to the chemist procedure is immediate. Thirdly, the chemist, by using coring devices, can extract an undisturbed sample form the liners and the undisturbed can be placed into the chemical extractant.

5.0 UTILIZATION OF LINERS

The question arises, "Are liners required to be used for all subsurface soil sampling?". The answer to that question is NO. It is the purpose of the liters to effectively capture contamination in the subsurface soils, in particular volatile organics, and thus assure, that the number derived from the analytical effort is closest to actual field condition concentrations.

In general, if the purpose of the subsurface soil sampling effort is to screen the subsurface soils (i.e., determine gross levels of VOA contamination in the subsurface soils) then liners are probably not necessary. However, if the intent of the investigation is to "write-off" a site, i.e., to show that there is not significant level of subsurface contamination, then the above method is not appropriate. To accomplish this goal, the Best Available Technology (BAT) is required. In this instance, the BAT is to utilize liners to show there is no significant contamination.

Within the HAZWRAP program, we investigate both NPL and non-NPL sites. In some of the non-NPL sites, the use of liners may not be required; however, the decision to use them or not comes from a recommendation by the hydrogeologist and concurrence by the Project Manager. For all NPL sites where VOAs are in analyte, liners will be used.

Gas Chromatographic Determination of Methyl Mercury in Fish, Sediment, and Water

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Methyl mercury is extracted as the bromide salt from fish and sediment and as the chloride salt from water samples. All extracts are treated with a common cleanup procedure that results in the conversion of methyl mercury to the iodide salt for electron capture gas chromatographic analysis. Recoveries ranged from an average of 88.5% for water samples to averages of 95.5 and 96.3% for perch and sediments. Methods for controlling contaminants and interferences are discussed for all phases of the method. Particular problems encountered were column poisoning and detector poisoning. When the method was applied to sediment samples collected from a polluted river, a correlation could be established between total mercury and methyl mercury when the concentration of total mercury was in the 0–10 μg/g region. For samples of very high inorganic mercury, the correlation failed.

When it became clear that mercury existed in many streams of the United States (1), and that naturally occurring methylation processes were causing the formation of the highly toxic methyl mercury, widespread efforts were undertaken to find ways to evaluate the full impact of the situation. At the National Environmental Research Center in Cincinnati the objectives were to develop rapid and accurate procedures for the determination of total mercury and methyl mercury in fish, sediment, and water. The method selected by the Analytical Quality Control Laboratory for the Environmental Protection Agency for the determination of total mercury in these matrices has been reported (2). This paper is a companion report on the method proposed for the

Westöö (3–6) published the first practical method for the determination of methyl mercury and adapted it to a variety of materials. Since the time of her first work, several variations of this basic method have evolved. All consist of an extraction of methyl mercury as a halide with solvent, followed by a cleanup procedure to prepare the extract for gas chromatography. Although other approaches are available, such as the extraction of methyl mercury as a dithizonate complex (7), the formation and extraction of the chloride (8, 9), bromide (10), or iodide (11) salts have found most widespread use.

The cleanup described by Westöö (5) involved the formation of a water-soluble adduct of methyl mercury and cysteine and its extraction into water. This was followed by acidification and extraction of the now-released methyl mercury with solvent. Aqueous solutions of other sulfur compounds—sodium thiosulfate (11), for example—have also been successfully used in place of cysteine. With this approach methyl mercury can be re-extracted from the thiosulfate solution after the addition of excess iodide salt.

Variations of these methods were tested for their application to environmental samples and were found to give satisfactory results, with high sensitivity and excellent recoveries.

METHOD

Apparatus

(a) *Gas chromatograph.*—Operated with oven temperature of 140°C and equipped for on-column injection with inlet temperature of 140°C; 6′ × ¼″ od Pyrex column containing 3% HI-EFF 4 BP (purified butane-1,4-diol succinate) on 80–100 mesh

tritium source detector can be used, but it is subject to foil poisoning and subsequent loss of sensitivity. If contaminated, tritium foil can be cleaned with abrasive.

(b) *Centrifuge tubes.*—50 ml with ground-glass stoppers or with screw caps with Teflon cap liners. Tubes should be calibrated or marked at 15 ml.

(c) *Tissue grinder.*—15 ml (Kontes Glass Co., Vineland, N.J., No. K-885459).

(d) *Kuderna-Danish concentrator.*—Set consisting of 3-ball Snyder column, 500 ml flask, and 10 ml ampoule.

Reagents

(a) *Methyl mercury iodide.*—(*1*) *Stock solution.*—Dissolve 0.163 g methyl mercury iodide in benzene and dilute to 100 ml. (Although standards prepared in this manner in our laboratory were stable for months, other researchers have reported methyl mercury iodide to be unstable in solution.) (*2*) *Standard solution.*—0.05 ng/μl. Dilute 5.0 μl stock solution to 100 ml with benzene.

(b) *Solvents.*—Benzene and toluene; pesticide quality.

(c) *Copper sulfate.*—Dissolve 50 g $CuSO_4.5H_2O$ in 200 ml water.

(d) *Bromide reagent.*—Mix 110 ml concentrated H_2SO_4 with 100 ml water, and cool. Dissolve 360 g KBr in 700 ml water. Combine solutions in 1 L volumetric flask and dilute to mark with water.

(e) *Sodium thiosulfate.*—0.005*M* in water.

(f) *Potassium iodide.*—Dissolve 100 g KI in 200 ml freshly boiled distilled water. Reagent must be stored in brown bottle and refrigerated. Formation of iodine in solution causes interferences in chromatographic analysis.

Extraction

(a) *Fish.*—Remove 1–2 g portion from frozen specimen. Add to tared aluminum weighing boat and weigh to nearest 1 mg. Quantitatively transfer to tissue homogenizer. Rinse pan with 5 ml water and add rinse to homogenizer. Grind until no chunks remain (2–15 min). Transfer sample to 50 ml centrifuge tube, using enough water to bring final volume to 15 ml. Add 2 ml copper sulfate reagent and shake. Add 8 ml bromide reagent, shake, and let stand 5 min. Add 20.0 ml toluene; stopper or cap and shake vigorously 2 min. Centrifuge to separate layers. Pipet 15.0 ml toluene layer into 125 ml separatory funnel and continue as described under *Cleanup*.

(b) *Sediment.*—Samples usually require some pretreatment to insure that homogeneous portions are

each sample. Shake, stir, or blend sample to
nize and place 25–50 g in tared evaporati
Weigh to nearest 10 mg and evaporate to
on water bath. Dry 1 hr at 103°C in oven
desiccator, and weigh. Percentage of residu
in calculations below.

Transfer additional 10 g portion of sa
tared, plastic disposable weighing boat an
to nearest 1 mg. Using minimum of water,
sample to 50 ml centrifuge tube and dilute
with water. Add 2 ml copper sulfate reag
shake. Add 8 ml bromide reagent, shake,
stand 5 min. Add 20.0 ml toluene; stoppe
and shake vigorously 2 min. Centrifuge to
layers. If substantial amount of sediment
in toluene layer, use glass rod to stir layer,
fuge, and decant solvent layer into 25 ml gr
cylinder. Record volume and transfer to
separatory funnel. Continue as describec
Cleanup.

(c) *Water.*—Adjust measured volume (50
sample to pH 1.1–1.3 in beaker with conce
HCl and transfer it to 1 L separatory fun
tract water with 100 ml benzene by shakin
ously 2 min. Let benzene layer separate from
then remove water by draining it back into
Collect organic layer in 300 ml Erlenmeye
Repeat extraction twice more with additiona
aliquots of benzene and combine organic laye
benzene through 20 mm diameter chromato
column packed with 2" anhydrous cry
Na_2SO_4 and collect it in Kuderna-Danish fl
ampoule set. Rinse 300 ml Erlenmeyer flas
with 5–10 ml portions of benzene and add r
column after extract has stopped draining.
column has again drained, add Snyder col
collection assembly and carefully concentr
tract to 10 ml on steam bath. Transfer extrac
ml separatory funnel and continue as de
under *Cleanup*.

Cleanup

Add 5.0 ml sodium thiosulfate reagent and
min. After any emulsions have broken, c
drain bottom layer into second 125 ml sep
funnel. Add additional 1.0 ml sodium thiosu
solvent fraction and shake 1 min. Again drai
ous layer into second separatory funnel and
solvent layer. Add 3.0 ml potassium iodide
to aqueous layer and mix. Add 3.0 ml benze
shake 2 min. Draw off aqueous layer and d
Drain benzene layer into 15 ml centrifuge tu
taining ca 1 ml Na_2SO_4 and mix. Decant

ingly if extract volume changes before gas chromatography. Although usually not required, maximum sensitivity can be achieved by carefully concentrating extract, using 60°C water bath with gentle flow of air directed at surface of solvent. Under no circumstances should solvent volume be reduced to <1.0 ml.

Gas Chromatography

Set nitrogen carrier gas at 60 ml/min, and provide additional 30 ml nitrogen/min as purge gas. Operate detector in dc mode with input of 60×10^{-11} amp. Alternatively, set conditions so methyl mercury iodide is fully resolved from solvent peak and will give full scale response for mercury equivalence of 0.3–0.5 ng.

Prepare calibration curve from series of injections of methyl mercury iodide. Measure areas under curve with electronic integrator or by calculating areas in sq mm, using product of peak height and peak width at half-height.

Inject portion of extract and quantitate any methyl mercury iodide peak giving 2% scale deflection. Perform calculations, using following formula:

Methyl mercury (μg as mercury) = (ng/μl from curve $\times V_a \times F \times 3.0)/(V_b \times V_c)$

where V_a = final volume of extract at time of gas chromatography, in ml; V_b = volume recovered after Na_2SO_4 drying, in ml; V_c = volume of toluene recovered from original extraction, in ml; F = 20 for fish and sediment, 1 for water. To convert to concentration units:

Fish—Divide by total weight in g and report as μg/g.

Sediment—Divide by dry weight in g and report as μg/g.

Water—Divide by sample volume in L and report as μg/L.

Discussion

Sample Preservation

Because a complex of biological and chemical activity, such as methylation and demethylation processes and the uptake and release of mercury compounds by microorganisms, can occur in an aqueous environment, it is necessary to preserve samples if analyses are not to be performed within a few hours. The use of 1 g copper sulfate/L will preserve the integrity of mercury compounds in aqueous solution if the copper sulfate is introduced at the time of collection. For example, a sample of Ohio River water dosed with 100 μg methyl mercury chloride/L and stored without sample treated with 1 g copper sulfate/L time of dosing contained 87 μg/L after 1 under the same conditions. Freezing wate ples has been similarly successful in pre: methyl mercury.

Fish and sediment are frozen upon coll Sediments are thawed shortly before analy the moisture content is determined at th so results can be calculated on a dry weigh Samples are taken from frozen fish to avoid caused by decay and rapid dehydration.

Extraction of Aqueous Samples

There are 2 requirements for the success traction of any of the methyl mercury l from water. There must be a large excess halide anion in solution available to the mercury cation. Also, there must usua rather rigid pH control. Methyl mercury is the easiest to extract from water becau compound is insoluble at neutral pH leve requires only an abundance of an iodide sal forced into the solvent layer. Extraction of n mercury chloride is best achieved at about p The anion is provided by acidifying with H about 0.1*M*. At lower pH levels methyl me chloride becomes increasingly soluble in acic When methyl mercury is extracted as the br salt the pH is adjusted to below 0.5 with I and an excess of bromide salt is used to ac the coupling.

Either benzene or toluene can be used success in the above extractions. The deg success achieved in the partitioning betwee salt-acid aqueous solutions and benzene or ene depends upon the anion selected. The tion constant (*p*-value) for methyl mercury ride from pH 1.2 water to benzene is only (10), which means that the successful extra of this halide requires large amounts of sol For example, to achieve a 96% recovery in tractions requires a 1:5 solvent-to-water Extraction of the iodide or bromide ca achieved with less solvent, although the vo of solvent required to extract 1 L water sa would still be large enough to require conce tion of the extract to achieve adequate sensiti The use of selective partitioning to achieve centration of extracts with solvent volum

a Kuderna-Danish concentrator was successful only for the chloride salt of methyl mercury. Total loss of the bromide and iodide salts occurred when 200 ml benzene solutions of these salts were concentrated to 10 ml, using the Kuderna-Danish apparatus. Recoveries as high as 94% for the chloride salt were obtained when dosed extracts were concentrated with this device. No evaporation tests were performed with toluene because of its high boiling point.

The Kuderna-Danish concentration of an extract to 10 ml must be done with extreme care and the solvent must not be permitted to completely boil away in the ampoule. High variance in recoveries can always be traced back to this step in the analysis of water samples.

In summary, the experiments with liquid-liquid partition of methyl mercury from water to solvent show: (*a*) to extract the halides requires pH control and abundance of the desired anion; (*b*) methyl mercury is best extracted from small volumes as the iodide or bromide; (*c*) methyl mercury is best extracted from large volumes as the chloride, which is not susceptible to volatility losses during solvent evaporation; and (*d*) benzene and toluene are both suitable for extraction of the halides, although benzene must be used for methyl mercury chloride because toluene boils at a prohibitively high temperature.

Extraction of Sediment and Fish

Methyl mercury in some sediments and all fish will form strong bonds with organic and inorganic sulfur. Direct extraction with solvent is not capable of overcoming these bonds and chemical treatment of fish and sediments is required before the extraction. Mercuric chloride has been used to displace methyl mercury from the sulfur and make it available for extraction (4). Because it seems ill advised to add mercury in any form to a sample being analyzed for mercury compounds, copper sulfate has been substituted in this role. The copper salt has been used for a variety of fish and sediment with success (11). It is partly because of this ability to displace mercury in sulfur bonds that copper sulfate is used for water samples as a preservative.

It must be noted that the acid hydrolysis method for methyl mercury can cause the degradation of these materials may appear as positive ferences in the methyl mercury determinati

Cleanup

The benzene or toluene extracts of fish or cannot be injected directly into the gas matograph. Apparently, the extracts contai fur compounds, possibly mercaptans, that d chromatograph. These materials, deposited front of the column, can form bonds with m mercury and may retard or even prevent th tion of methyl mercury. The cleanup ste included to eliminate such compounds.

The quality of the cleanup often depends cleanliness of the glassware. It has been that if the cleanup partitions are not pure when a portion of an emulsion is carried th a separation), the chances of column pois become significant. Efficient extractions wi carryover of emulsions can be obtained if techniques are employed to clean the sepa funnels used in the partitions. It is importan they not be washed with detergent. Instead use, they should be rinsed with tap water, ch acid, tap water, and then with distilled wa

Water analyses are affected by interferen the low levels of detection (20 ng/L) and also include cleanup. Aroclor® 1232, for exa if present in a sample at a concentration o μg/L would interfere with the gas chron raphy of methyl mercury unless it were removed by partitioning the methyl mercur an aqueous phase and then back to clean so It is also desirable to limit all chromatogra the determination of one particular methyl cury halide, for reasons discussed below.

The cleanup consists of the formation water-soluble adduct between methyl m and a suitable sulfur-containing compoun this way the methyl mercury can be re from the extracting solvent with high effic Once the aqueous layer is isolated it c treated somewhat like a water sample. Firs pH and salt content are adjusted for a fav extraction of a methyl mercury halide. S an extraction with benzene or toluene i formed to yield the methyl mercury in an e clean enough for chromatographic analysis an electron capture detector

solve and also decomposes rapidly upon standing. In addition, all lots of HCl in our laboratories introduced interfering peaks into the electron capture chromatogram. On the other hand, sodium thiosulfate is a dilute, relatively stable solution. The strong solution of potassium iodide is unstable but can be kept for several months if proper care is taken in its preparation. By using freshly boiled distilled water and by storing in a brown bottle in the refrigerator, one may prevent the formation of free iodine which causes interfering electron capture response.

The sodium thiosulfate-potassium iodide cleanup technique selected for use was modified to provide the smallest practical final volume and, therefore, the greatest sensitivity. The cleanup steps themselves provided recoveries in excess of 96%. With such efficiency, on the rare occasions when samples are still contaminated after a cleanup, they may be treated a second time with the cleanup without substantially affecting the validity of the results.

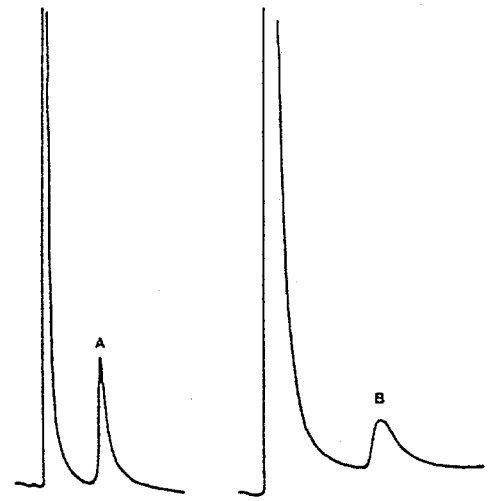


FIG. 1—Chromatograms of 0.10 ng methyl mercury iodide: A, typical of good column, retention time, 2.4 min; B, example of poisoned column, retention time, 4.7 min.

Gas Chromatography

The gas chromatographic determination of methyl mercury salts is complex because of the high activity of mercurials and the ionic nature of the salts. An on-column injection into a glass column was found to be the most successful way of chromatographing methyl mercury.

Several polyester type columns were tested in an effort to find one on which the tailing of the methyl mercury was minimized. HI-EFF 4 BP appeared to give the best results. A 6′ column with a 3% loading on Gas-Chrom Q, operated at 140°C, eluted the halides of methyl mercury in about 2.5 min with a minimum of tailing (Fig. 1). Some columns were used for 6 months or longer with no apparent loss of efficiency, although they required periodic reconditioning with microgram injections of methyl mercury iodide. The reconditioning is necessary occasionally even though the extracts are all subjected to the cleanup. The column can alternatively be restored after poisoning by injections of mercuric iodide solution (1 μg/μl in acetone), although the GLC system takes longer to recover from the injection of the inorganic salt.

Because the column is susceptible to poison-

alone may not be valid. However, area measurements (product of the peak height times the width at half-height) give satisfactory results.

Still another problem with the gas chromatography of methyl mercury is the tendency toward anion interchange. All halides will freely convert to one another, depending upon which of the anions is present in excess as residual system contamination. Because the retention times of the halides are nearly identical the apparent effect of any interchange is a broad peak, but in reality, because the halides have different sensitivities to the detector, even the response to injections of standards becomes erratic. The change in apparent retention time when the interchange occurs is small but measurable. Constant monitoring of this parameter is helpful in detecting either column poisoning or anion interchange.

The problem of anion interchange can be controlled by maintaining an excess of residual halide corresponding to that halide selected for the methyl mercury analysis. Whenever a halide of methyl mercury is to be determined with a column that has been used for other halides or other types of extracts it must first be conditioned for the specific halide by injecting microgram amounts of the appropriate methyl mercuric salt.

Table 1. Recovery of methyl mercury from dosed samples

Sample	Source	No. of analyses	Dose[a]	Recovery, %
White perch	local market	4	0.200 μg/g	95.5±4.6
Sediments	Anderscoggin River	10	0.010 μg/g	96.3±8.7
River water	Ohio River	4	0.10 μg/L	88.6±10.1

[a] The perch contained a background level of 0.370 μg/g (n = 7). The sediments and water did not contain a detectable natural level.

how to cure it have been well described (12). The ^{63}Ni detector, operated at 280°C, is apparently not subject to this effect and is the detector recommended for use. The sensitivity is sufficient so that 0.2–0.3 ng methyl mercury iodide gives a full-scale response with a stable baseline.

Accuracy and Precision

Except in unusual cases, the methyl mercury peak should be the only sample peak seen in the chromatogram of an extract that has been subjected to the cleanup. All other peaks can be traced to impurities in the reagents and can be eliminated. Once the chromatographic conditions are stabilized, few problems are encountered as the method is designed to avoid column and detector poisoning.

Recoveries from dosed samples of fish, sediment, and water analyzed by this method are reported in Table 1. The lower recoveries for water reflect the losses of methyl mercury during concentration of the extract. The sensitivity of the method is as follows: fish, 0.01 μg/g; sediment, 0.001 μg/g; water, 0.02 μg/L.

When 7 portions of a fillet of a large white perch were analyzed by this method, a mean value of 0.370 μg/g was obtained, with a standard deviation of 0.034 μg/g. Distribution of methyl mercury throughout the fillet appears to be uniform enough to permit small portions to be used for the analysis without homogenizing the whole fillet. The methyl mercury concentration in other parts of the fish was not determined.

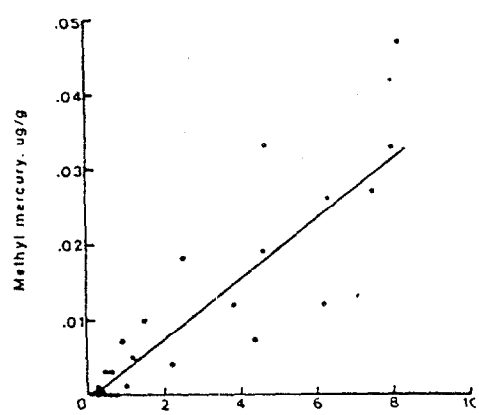
Application to Sediment Samples

The method was tested for its application to a set of sediment samples taken at regular intervals along a 25 mile stretch of a river known to be samples was in the 0–10 μg/g range, a good relation existed between the 2 parameters shown graphically in Fig. 2. The correlation efficient for these data was 0.879 and the equa for the line of best fit was:

$$Y \text{ (methyl mercury)} = 0.0040 \times X \text{ (total mercury)} - 0$$

indicating the total mercury content to be al 250 times greater than the methyl mercury.

When the total mercury was very large, a the case of 2 samples taken directly belo chlor-alkali plant outfall, the comparison fai The first sample was found to contain 195 total mercury/g and only 0.067 μg methyl n cury/g. The second sample was taken do stream a short distance and contained 111 total mercury/g and only 0.008 μg methyl n cury/g. The results of the analyses of thes samples tentatively indicate the rate of format of methyl mercury is not a function of total n cury when very high levels of inorganic merc are present.



APPENDIX C

OAK RIDGE RESEARCH INSTITUTE LABORATORY QUALITY ASSURANCE PLAN

OAK RIDGE RESEARCH INSTITUTE
113 UNION VALLEY ROAD
OAK RIDGE, TENNESSEE 37830

LABORATORY QUALITY ASSURANCE PLAN

FOR THE

STUDY OF MERCURY SPECIATION AT ENVIRONMENTAL SITES

NAVAL ORDNANCE STATION, INDIAN HEAD, MARYLAND

April 1990
(replacing November 1989)

Dr. N. W. Revis (Director) _____

Ms. B. J. Gille (LQAC) Laurie J. Moulton (for Barbara Gille)

Sponsor's Project Officer _____

Sponsor's QA Officer Janet Burns (for Mike Nugent)

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1.0 INTRODUCTION

This document describes a series of measures which constitute a plan to ensure that the analytical results obtained at the Oak Ridge Research Institute (ORRI) are reliable. At the core of the plan are the quality control procedures to establish and demonstrate the accuracy of the analytical data, by the inclusion of control samples containing accurately known amounts of the analyte under investigation. Control samples will consist of blanks, standard solutions, matrix spikes, and reference samples, which will demonstrate that the analytical method, in any given run, is in control. By inference, the results of unknowns determined in duplicate in a run which is in control may be expected to be accurate.

Supporting quality assurance procedures are also described. These comprise a series of steps which, taken together, demonstrate (1) that the equipment and personnel are adequate to carry out the analytical work, (2) that the samples will be handled, labelled, and documented in a manner which can ensure that the reported analytical results refer to the correct samples, and (3) that the integrity of the studies has not been compromised in any way.

The plan contains a provision for internal audits by the Laboratory Quality Assurance Coordinator (LQAC), during which all aspects of the data gathering and reduction process, as well as compliance of the facility, equipment, and documentation, will be evaluated. Reports of such audits will be made to management and will become part of the permanent quality assurance record of the study. These reports will be included as an appendix in the monthly reports submitted by ORRI to the study sponsor.

2.0 PROJECT DESCRIPTION

The purpose of this project is to determine the extent of mercury contamination at various sites at the Naval Ordnance Station, at Indian Head, Maryland. The level of contamination will be assessed by removing samples of sediment and soil from the sites and measuring the concentration of mercury therein. This will be achieved by acid digestion of the samples followed by atomic absorption spectrophotometry, using the cold vapor technique. (EPA 245.1, 1979; Std. Methods 16th Ed. #303F, pp 171-173).

A novel goal of this project is to determine the concentrations of the different chemical species of mercury which make up the total. To achieve this end, specific differential solubilization procedures, developed at ORRI, will be used. Thus, a saturated solution of sodium sulfide will be used to extract mercuric sulfide, with the concentration of mercury in the extract, again measured by the cold vapor technique, serving as a measure of the contribution of mercuric sulfide to the total. (Revis et al. (1989), Analyst 114: 823-825.)

Ionic mercury will be differentiated into unbound and bound components by sequential extraction into water, then dilute nitric acid. In each case, the extracts will be measured for mercury using the cold vapor technique.

Elemental mercury will be measured following acid digestion of the soil/sediment residue after heating at 115°C for 5 days to quantitatively remove the elemental

mercury by volatilization. The difference between the residual mercury and the previously determined total mercury will be used to obtain a measure of the elemental mercury originally in the samples. Finally, methyl mercury will be measured in the soil and sediment samples by gas chromatography, following quantitative extraction of the analyte into benzene, as described by Longbottom et al.

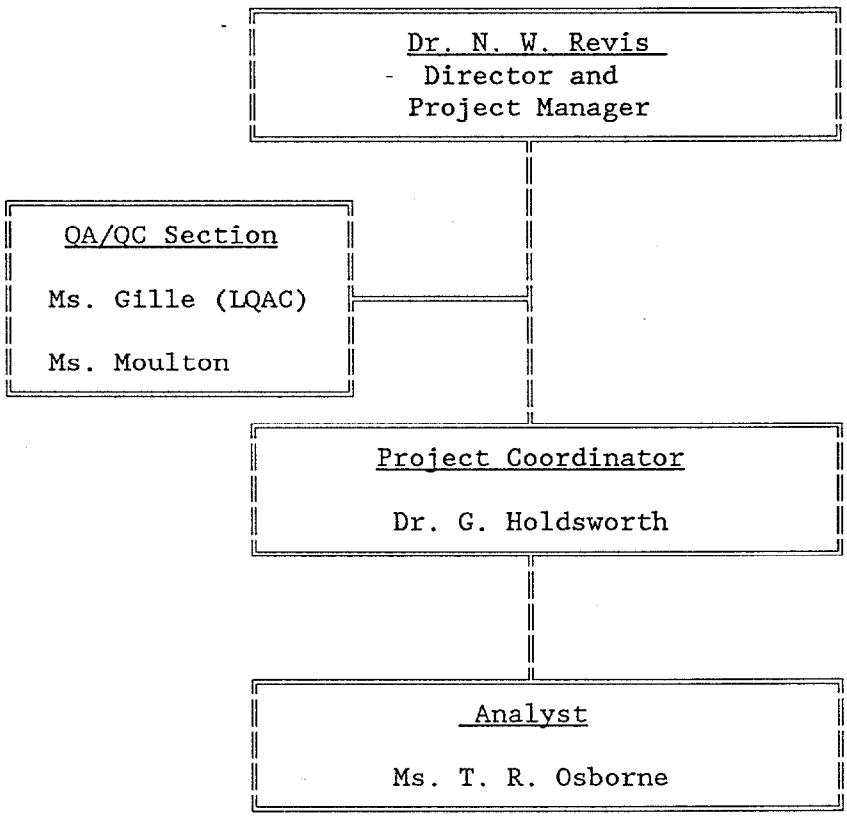
3.0 QA PERSONNEL

Ms. B. J. Gille is the LQAC at ORRI. Her curriculum vitae is contained in Appendix A, together with those of other key personnel. Ms. Gille is a technical writer and information scientist by training, with over 20 years experience. She is the LQAC for the ORRI toxicology program and for all the company's analytical services. Ms. L. Moulton will also serve to fulfill the quality assurance requirements for this project, deputy to Ms. Gille. By training Ms. Moulton is a computer database specialist, with further training in statistics. Her particular responsibility will be to carry out the data validation sections of the quality control program. These will be carried out according to the ORRI SOP for data validation, using the checklist format described in C-6.

4.0 LABORATORY ORGANIZATION

Table 1 presents the organization of the ORRI key personnel in the laboratory analytical services department. As indicated, the LQAC has direct access to the project manager, and has the authority to audit all aspects of the company's analytical activities to determine compliance of procedures and equipment, and that the data have been properly gathered from analytical runs which are demonstrably in control. In the event of a finding of non-compliance, or of an out-of-control run, the LQAC will require that corrective action be taken before any additional samples are analyzed. The finding and the nature of the corrective action will be recorded.

TABLE C-1 ORRI ANALYTICAL SERVICES KEY PERSONNEL



5.0 PERSONNEL TRAINING

5.1 Laboratory Methods

It is the policy of the Oak Ridge Research Institute that all analytical procedures and supporting activities will be fully described by standard operating procedures (SOPs). The SOPs provide a readily available resource to which new personnel or other employees unfamiliar with the procedure may be directed.

In practice, staff who are unfamiliar with a method or piece of equipment are instructed in its use by demonstration and guidance of the project coordinator and the research associate.

When a new laboratory technician is employed at the facility, it is the role of the research associate, Ms. Osborne, and the project coordinator, Dr. Holdsworth, to institute a series of on-the-job training exercises aimed at developing an awareness of basic laboratory procedures. Items in the series include, familiarity and competence with automatic pipettors and other measuring devices, the use of spectrophotometers, methods for carrying out simple biochemical and

chemical assays, construction of a simple standard curve, records keeping, etc. When these skills have been satisfactorily attained, a dated memo by the institute's director signifying that fact is included in the workers personnel file.

With the development of these basic "hands on" skills, the laboratory technician then receives instruction in the use of some of the major items of analytical equipment in the facility such as the gas chromatograph, and the atomic absorption spectrophotometer. Instruction in the use of these devices is integrated with an introduction to simple quality control concepts such as the use of reference samples, spikes, standard curves, method detection limit, blanks, etc. At no point is the new technician allowed to assay client's samples independently until cleared to do so by the demonstration of competence, and of an understanding of all the requirements for generating reliable results. As before, achievement of this level will be documented in the worker's personnel file.

The training protocols outlined above are instituted for each new employee as required, and have the aim of assuring that noone is called upon to carry out a task for which they have not received training, or for which their level of expertise is not determined.

Retraining may be required if the quality of work for which an employee is responsible falls significantly. This would be determined if the assays run by a technician were to consistently fail to meet quality control acceptance criteria in circumstances where the equipment is found to be functioning adequately. Instances of employee retraining, the reason for it, and the outcome, will be likewise recorded in the appropriate personnel file.

5.2 Quality Control Procedures

ORRI has certain basic requirements for the inclusion of calibration standards, control samples, reference samples, blanks, matrix spikes, and medium spikes for each analytical run which is carried out in this, or any of the other analysis programs at this institution. Instruction in quality control concepts and methods is an integral part of instruction in the method itself. At ORRI, it is policy that the analysts should calculate and document their own results. These then pass to the project coordinator to be checked. If the results are correct, they then pass to the quality assurance unit. This policy ensures that the results from each analytical run are checked against the raw data by as many people as possible, thereby providing an additional safeguard against any transcription or calculation error passing undetected into the final report.

5.3 Safety Policies

A number of safety procedures have been established for all analysts and other laboratory staff working at ORRI. These have been developed with the safety of the worker in mind, as well as fire prevention. Details of these requirements are contained in the appropriate SOP, and conveyed to all new employees when they commence work.

The project coordinator has the overall responsibility for ensuring that all employees are informed about the details of the company's safety policies. This is achieved by an informal in-service workshop.

Key features of the policy are that all workers should wear protective clothing, such as laboratory coats or smocks and safety glasses, while at their work station. They are required to refrain from eating, smoking, or applying cosmetics while in work areas. Such activities are to be confined to designated break areas.

In compliance with State of Tennessee "right to know" legislation, workers are instructed as to the level of precaution they are required to take for all chemicals with which they come into contact. These precautions may include, but are not limited to, the use of extractor fans or hoods, the need for protective gloves, respirators, and requirements for disposal. New employees are also shown the location of emergency exits and fire extinguishers and are instructed in their use.

Documentation of understanding and training in handling hazardous chemicals, and of the overall responsibilities of the new employee in laboratory safety, are kept in the employees personnel records.

6.0 SAMPLE HANDLING PRACTICES

In the mercury speciation project, the protocol requires the soil and sediment samples to be collected by the E. C. Jordan Company, followed by transportation to ORRI for analysis. Because of the disparate nature of the extractions and analyses to be performed, it is recommended that no preservative be used for the samples at the site of collection, but that the samples be stored on ice in clean glass bottles which can be sealed with a teflon-lined screw cap. The samples are then to be transported overnight to the analysis laboratory in a refrigerated container.

6.1 Sample Tracking/Chain of Custody Documentation

The glass bottles to be used to hold the specimens during transport will be provided to the E. C. Jordan Company by ORRI. Each will have been thoroughly washed with hot water containing non-phosphate detergent, rinsed several times with deionized water, then rinsed with 1:1 metals-grade nitric acid, water again, and finally pesticide-grade methylene chloride, before being oven-dried at 125°C. Each bottle will be accompanied by a sample chain-of-custody sheet, and have a label affixed to it. The pre-prepared sample label, which will contain all the information necessary for effective sample identification, is shown in Appendix C-2. It lists the following essential components:

Project #
Date of collection
Time of collection
Sample ID
Specific site
Location
Medium

Amount collected
How collected
Sampled by
Additional information

Personnel who collect the samples will sign and date the sample label and the chain-of-custody sheets (Appendix C-3) which will then accompany the samples to the laboratory.

6.2 Receipt of Samples

All samples which arrive at ORRI for analysis will be received by the project coordinator, Dr. G. Holdsworth, whose responsibility it is to record the receipt in the Sample Receipt Book, assign the sample an ORRI number, and designate a storage site for the sample within the laboratory. These data will be documented in the Receipt Book and on the sample itself, together with its gross weight.

Thus, for all soil samples which enter the facility in the mercury analysis program, the following items will be entered in the sample receipt book:

Project #
ORRI sample ID number
Test sponsor
Sponsor sample ID number/check standard number
Sample receipt date
Gross weight on receipt
Sample storage location
Sample color
Sample appearance

The project coordinator will have the responsibility for checking that all the samples which are listed on the chain of custody document have actually arrived at the facility. He will do this by checking off each received sample against the list on the chain of custody form. If there are no discrepancies, he will sign the form to acknowledge receipt of the samples. A photocopy of the signed form will then return to the E.C. Jordan company. If there are discrepancies, he will record this on the chain of custody document, and inform the contact person at E.C. Jordan, by telephone, with a letter to follow.

The project coordinator will also assure himself of the integrity of each batch of samples which arrive at the facility. Thus, he will check that the samples are still cool, and that none are broken. If any samples are broken, this fact will be conveyed to the E.C. Jordan Company, by telephone and letter, as described. Similarly, if all the refrigerant in the packing container is spent, and the samples have begun to warm up, the project coordinator will record these details in the sample receipt book, and contact the sampling company as before.

Written records of all discrepancies, and of the agreed measures taken to remedy them, will be kept by the project coordinator in a file, and as a dated comment in the sample receipt book. These findings become a part of the permanent record of the study.

6.3 Dispersal for Analysis

Within 2 hours of their receipt, the soil and sediment samples will be thoroughly mixed to assure complete homogeneity, then accurately subdivided into the various aliquots necessary for further processing. Thus, for total mercury, duplicate 1-g aliquots, measured accurately to the nearest 10 mg, will be weighed out into clean glass containers, labelled with the ORRI number, project number, treatment code, and date. These containers will be filled with enough nitric acid, as a preservative, to maintain the pH below 2.0 and will be sealed.

Duplicate 25-g aliquots, measured accurately to the nearest 10 mg, will be weighed out into sealable glass containers, labelled with the ORRI number, project number, treatment code, and the date, then stored sealed, at 4°C, prior to methyl mercury extraction. For extraction of elemental mercury, duplicate 5-g aliquots, measured to the nearest 10 mg, will be weighed out onto shallow aluminum trays, which will be marked in indelible ink with the ORRI number, project number, treatment code, and date. These samples will then be placed in the oven at 115°C to begin the process of elemental mercury volatilization.

For sequential extraction of the various ionic species of mercury, duplicate 1-g samples, measured accurately to the nearest 10 mg, will be weighed into glass screw-cap test tubes, which will be labelled with the ORRI number, project number, date, and treatment code. These will be stored sealed, at 4°C, prior to processing.

Any residual soil samples will be treated with enough nitric acid to lower the pH of the sample to pH 2.0 or below, prior to storage.

6.4 Sample Storage and Disposal

All samples arriving at ORRI will be stored at 4°C after being logged into the facility and dispersed for analysis as described above. Residual amounts of sample not initially taken for analysis will be stored in the original collection bottle after nitric acid treatment in the refrigerator until the holding time for mercury analysis has expired.

For disposal, all soil and sediment samples will be stored in a 55-gallon drum for future delivery to a designated landfill site. All aqueous waste will be diluted at least 20-fold with tap water and poured into the facility drainage system. All organic waste will be stored in heavy duty plastic storage containers, prior to disposal at an approved landfill site.

6.5 Holding times

No samples will be assayed in the mercury speciation project after the maximum holding time established for the analyte by the EPA. A key feature of the QA audits, carried out by the LQAC, will be to verify that the holding times have not been exceeded for any samples. Maximum holding times for the analysis of the different chemical species of mercury are as follows, total mercury, mercuric sulfide, elemental mercury, and acid soluble mercury, 28 days, and 14 days for methyl mercury.

7.0 MATERIAL PROCUREMENT AND CONTROL

Chemicals, consumables, and other materials and equipment purchased by the company for use in the mercury speciation project will be subject to the existing institution rules and procedures governing material acquisitions.

Thus, for each purchase, a duplicate order form will be made out, detailing the date, the purchase order #, the supplier, the end user, the supplier's stock number, a description of the article and the number of pieces, and the net cost. This purchase order will then pass to the project manager for approval and, if approved, will be telephoned to the supplier. All copies of purchase orders will be stored in numerical sequence in a loose leaf file, and also listed in sequential order in a log book, which will document the ordering date and the date of receipt. Shipping documents and supplier packing slips will also be kept on file.

When purchased materials arrive at the facility, all articles will be inspected by the project coordinator to ensure that they conform in amount, type, and quality, to that which was ordered. The supplier will be notified as quickly as possible in the event of any discrepancy. The shipping documents, packing slips, and the ORRI copy of the purchase order, will be used to verify that the order is complete. The receipt will be recorded in the ordering log book and the copies of the order forms filed. Inventories of all chemical and other consumables will be maintained by the project coordinator and upgraded with each new purchase.

All solvents, acids, and other chemicals which enter the facility will be labelled with their date of receipt, then stored in designated storage areas appropriate for that chemical. Thus, acids will be stored in a cupboard designated for the storage of acids alone. Solvents will be stored in one of two metal flame-proof storage cupboards. When a chemical is used, the date of its initial opening will be written on the label also, to facilitate inventory control. Separate disposal bins will be maintained for aqueous and organic wastes. These bins can be closed and are acid and solvent resistant.

In the mercury speciation project, ORRI will maintain its policy of using only grades of chemicals which are appropriate for the type of analysis intended. For organic extractions, solvents will be EM Sciences "Onnisolve" grade, which is recommended by the supplier for gas chromatography. The concentrations of residues, phosphorus and sulfur reported for this grade of solvent are lower than those specified for pesticide-grade solvents from other suppliers. For acid digestion prior to metals analysis, "AR Select" acids supplied by the Mallinckrodt company will be used. All other chemicals used in these studies will be reagent grade or better. Compliance with these requirements will be verified by the LQAC during the regular facility compliance audits. An audit check list of all points in the facility and procedures associated with this program is included in appendix C-7.

Prior to their use in extractions of mercury analytes, all solvents and acids which come into the department will be screened to determine that they give a suitably low background response to methyl mercury or mercury measurements in the ORRI analytical systems. Examples of all lot numbers will be tested and those which give satisfactory performance will be specified in future orders. The

outcome of all solvent and acid screens will be documented, as a signed and dated record in the chemical inventory log book.

The conductivity of the distilled water used in sample processing will be checked daily using a conductivity meter. Records of each check will be kept by the project coordinator in a log book set aside for this purpose. The target cut off point for acceptable water quality will be 2 umhos per cm.

8.0 FACILITIES AND EQUIPMENT

The research laboratory at ORRI consists of 12,500 square feet of office space, built on a single story. The building has 10 rooms, two of which are devoted to office space. The other rooms contain laboratories dedicated to organic chemistry analysis, biochemistry, microbiology (2), inorganic chemistry analysis, electron microscopy, histology, and radiochemistry.

The major items of equipment for organic chemistry analysis are the Hewlett Packard 5880A and the Tracor 550 gas chromatographs. The Hewlett Packard gas chromatograph, which was bought in 1984, is configured for both capillary and packed column operation and is fitted with flame ionization, electron capture, and nitrogen phosphorus detectors, whose signals are decoded by a Level 4 controller. This GC is also fitted with an HP 7672A autosampler. This system, configured for manual sample injection, will be used to measure methyl mercury in the mercury speciation project. The column will be a 6-ft Pyrex column containing 3% HI-EFF 4 BP on 100-120 mesh Gas Chrom II. The oven and injector will be set at 140°C, with the detector set at 300°C. The carrier gas will be 95/5% argon/methane at a flow rate of 30 cc/min. The ECD detector will be used to measure the methyl mercury eluted from the column. The Tracor GC, bought in 1981, is configured for packed columns only and is fitted with a flame ionization detector.

The other major item of equipment in the organic chemistry laboratory is a Waters high performance liquid chromatograph. This is fitted with fluorescence and ultraviolet detectors and a WISP autosampler.

The inorganic chemistry laboratory's principal item of analytical equipment for mercury analysis is a Perkin Elmer atomic absorption spectrophotometer model PE 603, which is fitted with a mercury/hydride generator model MHS-20 and a background correction unit. This equipment was bought in 1981. In our laboratory, mercury, arsenic, selenium, and antimony are measured by the hydride generation technique using this equipment. We also have a Perkin Elmer Inductively Coupled Plasma (ICP) and an Atomic Absorption spectrophotometer. The ICP is model PE ICP/6500 and the spectrophotometer is model PE 5000. The ICP/6500, bought in 1985, has two-way communication with the Perkin Elmer's data system, DS-10. The manufacturer's-supplied software is used to set up and control the spectrophotometers functions for automatic multielement analyses and the gathering and storage of the data generated. The PE 5000 atomic absorption spectrophotometer, which was bought in 1981, can also be used in the conventional flame mode utilizing an air/acetylene or a nitrous oxide/acetylene flame. In the ORRI laboratory, it is more often used with the heated graphite atomizer (HGA) system. The Perkin Elmer HGA system, model PE HGA-300 with an autosampler PE AS-40, may also be used.

9.0 EQUIPMENT MAINTENANCE AND CALIBRATION

Items of laboratory equipment whose performance is critical to the degree of compliance which is achieved with procedural SOPs will be monitored on a daily basis to determine that they are performing according to required specifications. For example, temperature checks of refrigerators and ovens will be made each day by the Research Associate, Ms. Osborne, who will make a signed and dated entry in the instrument maintenance log book, which will be kept with the machine at all times. The performance of balances will be monitored each day using a set of standard weights supplied to ORRI by the Baird and Tatlock company. These checks will be recorded in the appropriate balance log book. Deviations of greater than 0.5% will be considered sufficient for the balance to be recalibrated. The recalibration will be documented in the log book. Records of all routine services calls for balance maintenance will be kept in the log books. These services will be carried out on a yearly basis.

Calibration standards are supplied by appropriate companies or authorities. For example, our set of standard weights was supplied by the Baird and Tatlock conpany, the lindane/aldrin mixture was obtained from Hewlett Packard, and the inorganic mercury standard is an NSB standard. The dilution of this standard to 250 ng/ml will be documented on each occasion that it is carried out (daily whenever analyses are being run).

All major items of equipment will continue to be maintained on an as-required basis by service engineers employed by the respective manufacturers. Routine maintenance such as changes of injection septa, detector cleaning, etc., will continue to be carried out according to a schedule recommended by the manufacturer, and already in place at ORRI, see appendix C-9. Records of routine maintenance procedures will be available for QA audit.

Routine maintenance for the atomic absorption spectrophotometer will be carried out as recommended by the manufacturer, and documented in the instrument log book which will be kept with the machine. See Appendix C-10.

The atomic absorption spectrophotometer PE 603 will be calibrated and standardized each time analyses are carried out. Calibration will consist of the injection of a volume of accurately diluted NBS standard mercuric nitrate containing 250 ng of mercury. In line with the manufacturer's instructions, this amount of mercury should give a response of 0.2 units in the mercury hydride system. Accordingly, the slit control will be adjusted until this level of response is achieved for this amount of mercury. When the level of machine response is adequate, the standardization phase, described in Section 11.1, will begin. All glassware used in the processing of samples for atomic spectrophotometric analysis will be washed in hot water, using non-phosphate detergent, followed by several rinses in deionized water. After that the glassware will be rinsed with 1:1 nitric acid in water, then inverted and allowed to drain.
Glassware for use in methyl mercury extractions will be washed as described for atomic absorption spectrophotometry, followed by several rinses in distilled water, then pesticide grade methylene chloride. The solvent will be allowed to drain, then the glassware will be allowed to dry in an oven at 125°C.

The methods used for calibrating the gas chromatograph are similar in concept to those described for the atomic absorption spectrophotometer. They initially involve the injection of an accurately known amount of a mixture of 33 pg/μl each of lindane and aldrin in iso-octane. This mixture is a performance evaluation sample for EC detectors supplied by the manufacturers. The instrument attenuation and other parameters will be adjusted until the response is at or within 10% of that specified by the manufacturer. Routine maintenance and calibration of supporting equipment such as balances, pH meters, ovens, etc., will be carried out according to the manufacturer's recommended schedule and the appropriate in-house SOP. For example, all balances will be calibrated using a reference set of weights; pH meters will be calibrated using reference buffer solutions, and ovens will be checked for correct settings using a thermometer.

Limits of acceptability which will apply to the performance of refrigerators are 0°C-6°C. If a refrigerator reaches a temperature outside these limits, it will be readjusted to a more appropriate temperature setting or taken out of service until its performance can be stabilized.

All such maintenance and calibration activities will be documented in an instrument maintenance book, which is kept separately for each instrument. These records will be subject to routine compliance audits by the LQAC and staff.

10.0 ANALYTICAL PROCEDURES

In addition to the analyses which are outlined in the proposal accompanying this QA project plan, ORRI offers the following chemical analysis capabilities:

INORGANIC ANALYSES

(a) Elements

Routine assays		Routine assays	Ref.
Acid digestion of samples	then	ICP scan (up to 10 elements)	200.7

Individual Elements

	Ref.		Ref.
Arsenic	206.5/3	Aluminum	202.2
Calcium	215.1	Cadmium	213.2
Copper	220.1/2	Chromium	218.2
Lead	239.2	Iron	236.2
Magnesium	243.2	Manganese	243.2
Mercury	245.1	Selenium	270.3
Potassium	258.1		
Sodium	273.1		
Zinc	289.2		

(b) Wet Chemistry

Routine assays	Ref.	Routine assays	Ref.
B.O.D.	405.1	Inorg. phosphate	424F (*)
C.O.D.	410.1	Cyanide	335.1
Phenol	420.1	Fluoride	340.1
Hardness	130.2	Chloride	325.3
Alkalinity	310.1	Nitrite/Nitrate	353.3
Conductivity	120.1	Total phosphate	365.3
pH	423 (*)	Kjeldahl nitrogen	351.3
Ammonia	350.2	Sulfate	375.4

(*) std methods, 16th ed.

ORGANIC ANALYSIS

PCBs	608
Methyl mercury	Longbottom J.E. et al., NERC,EPA,Cincinnati, OH

11.0 QUALITY CONTROL PROCEDURES

The quality control procedures outlined below are a list of measures which will be carried out to determine and demonstrate that all analyses and associated laboratory preparation procedures in the mercury speciation study are working properly. The demonstration will be achieved in five ways.

(1) By obtaining standard curves in every run, in which the response can be mathematically demonstrated to be linear. These standardization procedures are described in detail in Section 11.1.

(2) By the repeated analysis, in 20 or more separate runs, of control and reference samples (such as EPA check and matrix standards) the results of which are sufficiently similar to the true values to assure accuracy. This process of primary standardization will take place prior to the analysis of any field samples from the mercury speciation project, and will be undertaken to show that the analyses carried out at ORRI can achieve satisfactory precision. The degree of precision will be assessed with the aid of continually upgraded control charts, a detailed description of which is included in Section 11.4. These charts facilitate considerations such as the level of overall accuracy, and the degree of within and between-run variation.

(3) By inclusion in the sample track of "spiked" samples. These consist of samples to which are added carefully measured amounts of the analyte in question. The spike can be added either to the assay or sample matrix. In the former case, the spike gives a measure of the system's capacity for measuring true amounts of a sample without interference. In the second case, it indicates the efficiency of the extraction procedure by the measurement of the percentage recovery of the spike.

(4) By the measurement of all unknown field samples in duplicate, as an added assurance that precision is being maintained for the field samples in addition to that obtained for the control, reference, and spiked samples.

(5) By determination of the method detection limit, to give an assurance that low levels of the analyte will be reported with an established degree of confidence that the values are greater than zero.

These five considerations, along with details of documentation and its passage through the organization, and how decisions will be made concerning out-of-control events and corrective actions and reporting thereof, are described in detail in the following paragraphs.

11.1 Standardization

In this phase, duplicate water blanks, followed by a range of five duplicated dilutions of an NBS mercuric nitrate standard, will be injected in turn into the hydride generating system in order to measure the mean level of response obtained for the different amounts of mercury used. These mean levels, as well as the mean of the response obtained for the water blank, will be plotted against the amounts of mercury injected, and the "goodness of fit" assessed using linear regression analysis by the method of least squares. A correlation coefficient of >= 0.995 is assumed to be an acceptable test of linearity. Standard curves that meet these criteria will be used to determine the concentration of QC standards and of unknowns, with the results being determined directly from the linear regression. For methyl mercury, accurately made up dilutions of methyl mercury in benzene, prepared from a stock methyl mercury solution which will

itself be made up fresh each day on which analyses are to be carried out, will be injected onto the column in 2-μl aliquots. The mean peak height of the methyl mercury responses, as well as that of a benzene blank, if any, will be plotted against the concentration or amount of analyte injected. Linear regression analysis will be used to assess "goodness-of-fit" as described above. If the graph meets the acceptance criteria of a correlation coefficient of >=0.995, the run will be continued for the determination of quality control standards and of unknowns, with the standard curve used to read off concentrations from the peak heights.

11.2 Limits of Detection

The procedure used for measuring the method detection limit (MDL) of the mercury and methyl mercury analysis will be that described by Glaser et al. (Environ. Sci. Technol. (1981), 15: 1426-1435).

It will be carried out once per month immediately following machine calibration and standardization, throughout the course of the mercury speciation program.

For inorganic mercury, using the cold vapor technique, a dilution of the NBS mercury standard will be made in water, in order to inject 40-ng amounts of mercury into the hydride system. This amount of mercury has been chosen because it meets the criterion of being no more than five times the theoretical minimum detection limit of the system. Seven analyses of this volume of mercury solution will be made, with a water blank interspersed between each. The response for each mercury injection will be measured on the standard curve to obtain a reading for the amount of mercury injected. A mean and standard deviation for these values will then be determined.

The MDL, defined as the lowest amount of the analyte which can be determined to be greater than zero with 99% confidence, will then be calculated according to the following equation:

$$MDL=(t_{n-1}^{99\%}) \times S$$

where $t_{n-1}^{99\%}$ is the value of the one-tailed distribution of t for n-1 degrees of freedom at 99% confidence, and S is the standard deviation. The MDL will also be calculated in a same way for mercury dissolved in saturated sodium sulfide. For this determination, however, all standard solutions, blanks, and the low concentration of analyte used will be made up in saturated sodium sulfide.

Calculation of the MDL for methyl mercury will be preceded by calibration of the EC detector, and by standardization of the methyl mercury analysis, as previously described.

A 10-mg/ml stock solution of methyl mercury iodide in benzene will then be made up, from which serial dilutions in benzene will be made to obtain a final concentration of 25 ng/ml. Two-μl injections of this solution onto the column will deliver 50 pg of methyl mercury, which is a level of methyl mercury not more than 5 times that of the expected region for the MDL. Seven analyses of this volume of methyl mercury dilution will be made, with a benzene blank interspersed between each. The peak height obtained for each injection, will be measured on the standard curve to obtain a reading for the amount of methyl mercury injected.

A mean and standard deviation for these values then will be determined, and the MDL calculated as described above.

11.3 Control Samples

Control samples are materials and solutions in which the concentration of the analyte is accurately known. They will be included in all analytical runs to monitor performance and to provide an indication that the analytical and preparation systems are working properly. The following types of control samples will be routinely used, and are here described: reagent blanks, method blanks, assay spikes, matrix spikes, reference (check) standards, and low standards for MDL determination.

Reagent blanks - consist of the solvent or medium in which the analyte is routinely dissolved for analysis. For inorganic mercury, the reagent blanks will be dilute nitric acid or saturated sodium sulfide, depending on whether acid or sodium sulfide extracts are to be measured. For methyl mercury, the reagent blank will be benzene.

Volumes of reagent blanks equal to those of the samples will be assayed in each analysis system, to give a measure of the background response of the measuring the absence of analyte. The level of response for the reagent blanks will be plotted on each standard curve as the zero concentration parameter (x=0), and entered into the linear regression analysis, to determine linearity of the standard curve, as the lowest response data pair. The coordinates of this point would be x=0, y=0.005, where the mean of the duplicated reagent blanks is 0.005. All other machine responses will be plotted directly against the dose, without subtraction of the reagent blank machine response.

Duplicate determinations of the reagent blanks will be carried out at the beginning of the standardization phase. (Further reagent blanks will be assayed (seven in all) during the establishment of the MDL).

Method blanks - are solutions of acid, sodium sulfide, or benzene, which are the end products of extraction processes carried out on soil known to be free of the analyte being measured. Such extractions will be carried out in parallel with those of the field samples processed in the mercury speciation study. Because of the difficulty of obtaining soil samples which can be certified free of mercury, a NBS standard of urban particulates #1648, which is free of mercury, will be used for this reference material. Extracts of this material will be assayed at 5% frequency or once per analytical run, whichever is higher.

Inclusion of method blanks in the sample track allows an assessment of whether there is another extractable feature of the matrix which is likely to obscure the system's response to the analyte. This possibility will be assessed statistically by comparing the machine responses of the reagent and method blanks using the Student's t test for unpaired data. If statistically significant differences are found between reagent and method blanks, the processing of field samples for analysis will be suspended, and the cause of this anomaly investigated.
All findings relating to method blank determinations and their comparison to reagent blanks will be documented on the analysis data sheets, and, in the event of significant differences, as a signed and dated record in the quality control

file for that run. This discrepancy will then becomes subject to quality control corrective action procedures as outlined in sections 11.9 and 11.10.

Assay spikes - are extracts of field samples in the mercury speciation study, in either dilute nitric acid, saturated sodium sulfide, or benzene, to which have been added accurately known amounts of either mercuric nitrate, mercuric sulfide, or methyl mercury iodide, respectively, at no more than five times the concentration of the MDL. The spiked solutions will then be assayed and compared to their unspiked counterparts to provide a measure of the incremental response due to the spike. This incremental concentration will be expressed as a percentage of the spike to indicate the ability of the assay system to accurately respond to known concentrations of the analytes without interference from components in the assay system. Assay spikes will also be included in the sample stream at 5% frequency, or at least once per assay run.

Acceptance criteria for the percentage recovery of assay spikes are between 85-115% of the true value. If the percentage recovery falls outside these limits, the method of standard additions (MSA) will be instituted as described in section 11.4.

Matrix spikes - consist of accurately measured amounts of an analyte which is added to soil or sediment samples. Matrix spikes are then extracted in parallel to field samples and assayed as part of the sample track. The rationale for the inclusion of matrix spikes as part of the analysis scheme is to determine that the extraction procedure is sufficiently quantitative.

Because of the difficulty of preparing matrix spike pools in which the added analyte is homogeneously dispersed, fresh matrix spikes will be prepared each time analyses are carried out. Thus, for total mercury, duplicate 1-g aliquots of an unknown soil sample will be weighed out in addition to those unspiked replicates used to determine the endogenous level. To the additional aliquots will be added an accurately measured volume of the suitably diluted NBS mercuric nitrate standard solution, and the entire aliquot will then be acid-digested prior to analysis. The incremental amount of mercury determined in the spiked sample compared to its non-spiked counterpart will be used to determine the percentage recovery of the spike, and thus the efficiency of the extraction.

In a similar way, replicate 1-g aliquots of soil will be spiked with carefully measured volumes of standard solutions of mercuric sulfide in saturated sodium sulfide, prior to extraction for mercuric sulfide determination. Also, 25-g replicate amounts of some soil samples will be spiked with standard solutions of methyl mercury iodide in benzene, prior to extraction into benzene for methyl mercury analysis.

As with assay spikes, matrix spikes will be included in the sample track at a frequency of 5%, or at least one duplicate per analytical run, whichever is higher.
Acceptance criteria for matrix spikes will be in the range 60-140% of the "true value". Any deviation from this range will constitute an out-of-control condition, for which a corrective action study and report will be required. This will be coordinated by the LQAC.

Reference (check) standards - will be used in the quality control of these analyses to determine precision and accuracy. The reference standards will be accurately known solutions of mercury nitrate in dilute nitric acid, mercuric sulfide in saturated sodium sulfide, or methyl mercury iodide in benzene, as appropriate. The mercuric nitrate and mercuric sulfide and methyl mercury reference solutions will be prepared at the beginning of the study in sufficiently large pool amounts to ensure that enough of the material will be present to run the same reference standards in every run throughout the lifetime of the study. After preparation, the solution pools will be subdivided into small aliquots, in vials which will be sealed with a teflon-lined cap. These vials will then be stored in vacuum desiccators, at -20°C for the mercuric nitrate and methyl mercury pools, and at room temperature for the mercuric sulfide pool.

The reference samples will be included in each assay run at a 10% frequency, and at least in duplicate for each assay run. Accuracy will be determined by comparison of the measured concentration with the reference (true) value. Precision will be determined by monitoring the results obtained for the reference standards throughout the study, with the aid of control charts.

A key feature of the quality assurance plan for this project is that, prior to the analysis of unknown samples, the precision of the method will be determined by the preliminary establishment of a data bank from which the successive results of analyses of reference samples will be plotted against run number. At least 20 runs will be used in this primary standardization program. A "grand mean" value will thus be determined for the reference sample, and will be continually upgraded and revised as more data are added to the data bank. The control plots which will be obtained, described in detail in Section 11.4, will give information on the degree of precision obtained in the assay and provide the statistical basis for assessing whether this degree of precision is high enough for each assay run to be considered to be in control. Similarly, plots of the range between duplicate assays, and of the successive range, in which the difference in mean values between successive runs are plotted, will be used to provide important statistical information concerning within and between-run variation, respectively.

The level of accuracy of the assay will be obtained by comparing the value of the accumulating grand mean with the "true" value of a reference sample. Allowable deviation of the measured mean from the reference value will depend on the degree of accuracy which the method of choice is capable of attaining. For mercury measured by the cold vapor technique, acceptable deviation from the reference value may be up to 5%, with an acceptable standard deviation of 5% or less. For methyl mercury measured by gas chromatography, allowable deviations of up to 10% may be envisaged.

11.4 Method of Standard Additions

The method of standard additions (MSA) is a procedure which will be carried out whenever the mean of an assay spike deviates from the theoretical spiked value by more than 15%. The decision to institute this procedure will rest with the project coordinator who will decide how many samples will undergo the procedure and which ones. The reasons for instituting the MSA will be noted in the run file.

The MSA will be carried out by the spiking of several aliquots of a sample digest or extract with an incremental series of accurately known spikes of analyte. The samples in this spiked series will be assayed in duplicate and the responses plotted against the concentration of the spike.

The data will be subjected to linear regression analysis by the method of least squares, and the best fit line drawn through the points based on the regression analysis. The y value (for the response parameter) equivalent to a spike concentration of 0 (plotted on the x axis), represents the response due to the sample alone. This value will then be used for calculating the concentration of analyte in the unknown by comparison to the initial standard curve.

11.5 Control charts

Grand mean charts - will be the primary means of establishing that the analyses carried out in this program are accurate and precise. Prior to the commencement of analysis, a series of at least 20 assay runs will be carried out for each analyte in which, after calibration and standardization of the analytical system, a previously unopened vial of reference sample will be used to provide duplicate aliquots for analysis.

The concentration of the reference material will be calculated for each replicate, and the mean will be plotted as the y parameter, with run number plotted on the x axis. The means of duplicates determined in subsequent runs will be added to the same plot, and the points joined to plot the progression of the mean with time. A grand mean will be calculated from the daily means of the replicates and drawn on the graph as a straight line parallel to the x axis. It is the deviation of this grand mean from the reference value which provides an index of the accuracy of the analytical method.

In addition to the grand mean, warning and control limits will be established for the assay of the reference material. These will also be plotted as lines on either side of the grand mean, parallel to the x axis. To establish these limits, the standard deviation S of the grand mean will be determined. Warning limits will then be the mean $\pm 1.96S$, and the control limits will be the mean $\pm 2.58S$.

The data obtained in this primary standardization phase will be entered into a programmable spreadsheet, and the grand mean chart will be drawn using a computer-aided graphics package. By this method, each additional point on the graph will alter the mean and the warning limits automatically, with subsequent direct visualization of the upgraded control chart. An example of this computer-plotted grand mean chart is shown in Appendix C-4.

When the preliminary 20 runs of the primary standardization phase are complete, and the degree of accuracy of the method has been found to be acceptable, the analysis of unknowns will begin. During this phase, aliquots of the pooled reference materials will continue to be assayed, at least in duplicate for each run, and with a minimum frequency of 10%. Means of the pool duplicates will be added to the grand mean chart for each new run in the series. If the mean of a reference pool is sufficiently close to the reference value, and if it falls within the upper and lower control limits, the assay run will be judged to be in control, providing linearity and recovery criteria are also met. In this case, the values of the unknowns in the analytical series will be used as a measure of the levels of analyte in the field samples.

As stated above, the mean of each reference pool will be evaluated as it is plotted on the chart, as an index of whether that particular run is in control. On the average, even if the process is in control, one point in 20 will exceed the 95% warning limits and one point in 100 will exceed the 99% control limits. The latter probability is considered sufficiently unusual to require that the analytical process be stopped. Each point will also be evaluated serially, to determine whether there is drift in the system. This will be evident in a tendency for each successive point to be higher or lower than the previous one. In practice, if any of the following criteria exist for an analysis method, the process will be considered to be out-of-control.

1) Any one point outside the control limits
2) Any three consecutive points outside the warning limits
3) Any eight consecutive points on the same side of the mean
4) Any six consecutive points in which each is larger (or smaller) than its immediate predecessor
5) Any obvious cyclical pattern in the point

<u>Range charts</u> - will also be plotted for each analyte in the mercury speciation program. For these charts, the difference between the duplicate values of a run (R) will be plotted sequentially against run number, thereby providing a progressive record of the amount of within-run variation. The mean of the range for the various runs in the series will be calculated and drawn on the chart as a line parallel to the x axis. It will be continually upgraded as described for the grand mean charts and warning and control limits will be established. Using the criteria listed for the grand mean, each analytical run will be evaluated for acceptable within-run precision by the position of R for the reference samples compared to the warning and control limits.

The warning limit will be considered to be the mean x 2.51, and the control limit will be the mean x 3.27.

Between-run variation will be evaluated by the construction of <u>successive range charts</u>. For these charts, the mean of the duplicate analysis of the reference sample in a run will be subtracted from the mean of the same sample in the previous run. This will yield a value R_s which will be plotted against run number, for runs numbered 2 through n. The mean of the R_s values will be calculated and continuously upgraded as more runs are added to the series. This mean will be included on the chart as a line parallel to the x axis. Also included on the chart will be warning and control limits which will be determined by the mean x 2.51 and x 3.27 respectively.

The R_s value obtained for the reference samples in each analytic run in the series will be used to evaluated acceptable between-run variation by its position relative to the mean and to the warning and control limits. The criteria listed above for the grand mean will be used to evaluate whether the run will be acceptable in terms of between-run precision.

11.6 Duplicates

All blanks, matrix and assay spikes, reference samples, and field samples which make up an analysis run, will be assayed in duplicate. If, the range of the duplicates gives a Relative Mean Difference (RMD) of greater than 20%, the measurements will be considered to be out-of-control and the sample will be reassayed in duplicate.

The RMD will be calculated according to the following formula:

$$RMD = \frac{V1-V2}{mean \times 2} \times 100$$

where V^1 and V^2 are the duplicate values

The sample track will be constructed so that each pair of replicates is as widely separated in the analysis sequence as possible. By this means, any tendency of the assay to drift during the course of a run will be assessed. Where necessary, the run will be restandardized. The restandardization and the reason for it will be fully documented.

11.7 Calculation of Results

The standardization of each assay will include a standard curve drawn using duplicated analyses for at least five standard concentrations and a reagent blank. To assess linearity, the data pairs of machine responses versus concentrations will be entered into a computer program to carry out a linear regression analysis by the method of least squares. A correlation coefficient of >=0.995 will be used as a criterion of acceptable linearity and "goodness-of-fit." Concentrations of control samples and unknowns will then be determined directly from the linear regression.

Calculations subsequent to the determination of the analyte concentration in the final extract will involve simple proportionality principles, taking into account the amount of soil extracted, the total volume of extractant, and the amount of extractant or its dilution used in the assay procedure.

For mercury measured by the cold vapor technique, the following equation may be used;

$$K = \frac{C \times 1000}{V \times 4} \text{ mg/kg}$$

where C = conc. of unknown (mg/l)
V = vol. of unknown (ml)
K = soil concentration in mg/kg (wet weight)

The value C is the concentration (in mg/l) obtained by reading the response for the unknown off the standard curve. These values are obtained by mixing accurately measured 1-ml aliquots of the different standard dilutions with 10 ml of borohydride solution, to quantitatively measure evolved mercury hydride.

Because 1 g of starting soil or sediment will be digested in a volume of acid finally diluted to 250 ml, the soil concentration must be factored C x 1000/4, to convert the concentration to mg/kg of soil. Finally, because 1 ml of the standard dilutions will be used to construct the standard curve, the value obtained above must be divided by the volume of digest used for mercury hydride generation to obtain the true concentration of the mercury in the soil or sediment in mg/kg (ppm).

In the methyl mercury assay, for each unknown, the height of the peak on the gas chromatograph chart will be compared to that of the standard solutions plotted as a standard curve. Using the linear regression procedure described above, this will yield a value for the concentration in benzene (C).

For the Longbottom procedure for measuring methyl mercury, a starting amount of 25 g of soil will be used in the extraction procedure, and 20 ml of toluene. The volume of toluene recovered after carefully separating the phases will vary, but will be measured accurately in each case (y). The final extraction volume of benzene will be 3 ml, thus the concentration of the methyl mercury in the soil sample (K) may be calculated (in mg/kg) using the following equation:

$$K = \frac{C \times 3 \times 20 \times 40}{1000 \times y} \quad \text{mg/kg}$$

K = soil concentration (mg/kg wet weight)
C = soil concentration in benzene extract (ug/ml)
y = volume of toluene processed (ml)

11.8 Documentation

Central to the establishment of adequate quality control of an analytical project is the maintenance of complete written records which cover all phases of the work. To achieve this goal, guidelines covering documentation of the analyses in the mercury speciation project have been established. These are described in the following paragraphs.

The mercury speciation project and its various analytical subsections will each be given a number for ready reference and classification. Each analytical run within these subsections will be given a reference number, and all preparative, analytical, and quality control records generated in this run will be kept in a separate file encoded by this run number. Thus, results of analyses in the run, and written records of extractions of samples which will be analyzed in the run, will be included in that particular run file. Worksheets on which the raw data of analyses, extractions, and standardizations may be recorded have been designed to simplify record keeping. An example of a mercury analysis worksheet is shown in Appendix C-5. Along with the analytical and extraction records, each file will contain hard copies of all instrument calibration records, control sample data, QC plots, standard curves, and all calculations of unknowns and control samples.

When the documentation of a run is complete, the file will pass to the project coordinator to be checked and then to the quality assurance unit. At this point, a quality assurance file will be created for that particular run in which all

findings from a complete data validation exercise will be contained. The various steps in this process are described in Section 12.2.

If the run is found to be in control, and all audited features of the analytical process are found to be in compliance, the results derived from the run will be accepted and entered into a computer database especially designed for the project. Hard copies of the default report forms will also be kept for each analytical run in the appropriate QA file. The ultimate responsibility for accepting or rejecting the results of an analytical run rests with the LQAC.

11.9 Out of Control Events

This section contains details of the types of out-of-control events which may occur, how these occurrences will be documented, and who will be responsible for correction and documentation.

If initial attempts to standardize the assay, on a given day, yield dose responses which fail to meet linearity criteria, the analyst will continue the standardization process until the acceptable degree of "goodness-of-fit" is achieved. These multiple standardization attempts will be documented in the run file by the analyst. When a linear standard curve is obtained, unknown and control samples will be analyzed in the run as planned.

Non-routine deviations from quantitative recoveries in the spiked samples will be initially assessed by the project manager. If the assay spikes and reference samples are in control for the run, the results of the unknowns may be accepted, although the out-of-control recovery will be documented in the run file.

If a sample container is broken when it arrives at the facility, the receiving officer will note this in the sample receipt book, and document whether or not more sample is available. If no additional sample is available, the supplier will be informed.

If the results of analysis of a reference sample fall outside the 99% confidence limit, or if any of the other criteria for rejection listed in Section 11.4 are met, whether from the grand mean, range, or successive range charts, the run will be deemed to be out-of-control, and the results contained therein may not be used or reported. This finding is documented in the run file, and in the quality assurance file for the particular run. The process of analyzing unknowns will be discontinued, and efforts made to find the reason for the discrepancy. The responsibility for making this decision, and for ensuring that the results determined in the suspect run are not reported to the sponsor will rest with the LQAC. It is the LQAC's responsibility also to ensure that the analytical process is back in control before further samples in the program can be analyzed. Documentation of such a statistical out-of-control event will be made in the quality control and run files, and contained in the regular monthly reports to the sponsor. If the analysis procedure appears likely to remain out of control for an extended period, the study sponsor will be informed to ensure that the supply of samples will be curtailed. This will be necessary so that holding time requirements are not compromised.

An integral part of the laboratory's effort to correct an out-of-control event will be the preparation of a corrective action report. This report, signed by the project manager and the LQAC, will be included in the project monthly report, and will contain a description of the out-of-control event, its date, and who discovered and verified it. Importantly, it will detail the nature of the corrective action and the outcome in terms of return to in-control performance. A copy of the control charts and of other data describing the out-of-control condition and its correction will be included in the report. Copies of this report will be sent to the sponsor of the studies, and included in the quality assurance file for incident documentation.

11.10 Corrective Action

1) Finding

In this section, it is hypothesized that the LQAC and her staff, during a routine data validation exercise, may find an example of poor quality control, for example, in the form of an analytical sample with a reference mean outside the control limits. These outliers have a low probability (<1:100) of belonging to the population, and are therefore indicative of an error in the analytical run for that particular day. Firstly, the discrepancy will cause all the determinations which are done in the same run to be rejected. Secondly, analysis of further samples in the same series will be suspended until the cause of the out-of-control event is determined.

2) Correction

When the flow of samples through the analytical system is suspended, the possible causes of the error will be systematically investigated. In the first instance, because of the possibility that the error is not due to a systematic error in the system, but merely to an operator inconsistency such as careless pipetting, a run will be set up containing at least two aliquots of the reference sample, measured in duplicate. If the analysis of both of these aliquots indicates that control has been restored, the LQAC may conclude that the out-of-control event was not due to a systematic error. However, if the results of the analyses indicate that the system is still out of control, more wide-ranging changes may be contemplated. One or more of the following parameters may be involved, and will need to be evaluated:

- detector calibration.
- accuracy and age of standard solutions.
- comparison of detector response for new standard solutions v the old standard solutions.
- linearity of the new standard curve.
- check of water purity.
- eg. conductivity within defined limits.
- detector response to reagent blank within expected limits.
- calculation check.
- correct weights and volumes used.
- balances functioning normally; -recalibrated.
- service and maintenance of analytical equipment by qualified technician.

These evaluations will be carried out by the research associate, Ms. Osborne, in consultation with the project coordinator, Dr. Holdsworth, and the project manager, Dr. Revis. It will be the project coordinator's duty to maintain full documentation of the corrective actions and their outcome.

In each analytical run which is set up to evaluate the effects of the various changes to the system, aliquots of the reference samples will be run in duplicate, together with method blanks, a full range of standards, and matrix spikes.

When the machine responses regain equivalence to concentrations of analyte in the normal range, the LQAC will carry out a evaluation exercise on the raw data to check the validity of the determination.

Before it can unequivocally be concluded that the system has returned to control, the LQAC will require a further seven runs to be carried out, containing duplicate reference samples, plus blanks, standards, and matrix spikes. Each reference mean in all seven of these analytical runs must be between the upper and lower warning limits, for the method to be considered to be back in control. When this happens, the LQAC will fill out a corrective action report summarizing all the findings, and corroborating the return to control.

With the issue of a corrective action report, an example of which is given in Appendix C-8, routine analysis of samples in the sample track will resume.

All out-of-control incidents will be documented, and copies of the corrective action reports will be sent to the Analytical QC Specialist. Copies of the reports will be placed in the LQAC's file of incidents documentation, and referenced briefly in the monthly progress report.

12.0 INTERNAL LABORATORY AUDITS

12.1 Facility and Procedural Compliance

The LQAC and her deputy will monitor, at regular intervals, all key features of the equipment and facilities of the research laboratory, as they pertain to the efficient functioning of this project. Included are the efficient functioning of the analytical equipment, calibration of balances, date monitoring of chemicals, and ensuring compliance with safety programs, such as the use of masks and other protective equipment where necessary.

Failure to comply with any of the prescribed conditions, or in any other aspect of the study under the surveillance of the QA/QC unit, will be reported by memo to the Project Manager. A duplicate copy of the memo will become part of the permanent QA/QC record, copies and summaries of which are available to the study sponsor in the monthly reports.

At regular designated intervals, the QA/QC unit also will monitor the complete analytical processes for procedural compliance. These audits, which will be carried out unannounced, will be to determine the extent of adherence to the SOPs which form the basis of this study. All matters of concern arising out of this audit will be reported to the Project Manager and become part of the permanent QA/QC record.

Timing and phasing of the compliance audits for facility and procedural compliance will be based on a monthly rotation, but will not be set to a repetitive timetable. However, all features of the analytical operation will be audited at least once during any given month.

12.2 Data Evaluation and Reduction

As described in Section 11.6, the raw data generated in an analysis run will be used by the analyst to calculate the results of unknowns and to upgrade the control charts described in Section 11.4. From these considerations will be generated a series of interim results whose calculation will be documented, and whose values will be tabulated in the file. When the file passes to the quality assurance unit, a complete data validation exercise will begin. All aspects of the documented calibration, standardization, and analysis of control, spiked, and reference samples will be checked, including all calculations and data reductions. The quality control charts will be evaluated with particular reference to the question of whether the values obtained for the reference samples were within the control limits. All findings concerning the accuracy of calibration, the linearity of standardization, the method detection limit, the efficiency of extraction, the level of interference, and the degree of analytical accuracy and precision, will be summarized in writing in a file which will be kept in the quality assurance unit for each analytical run.

If all the described features are in compliance, the interim results from an analytical run will be accepted by the LQAC, and become part of the accepted results of the study. The results will then be entered into the database. Acceptance of the results from an analytical run will be indicated by a written note in the analysis file, and in the quality assurance file.

12.3 Document Control

The documentation associated with the mercury speciation study falls into four categories: (1) the analysis files, (2) the sample integrity files, (3) the quality control files, and (4) the quality assurance files. Each category fulfills a different role in the study, although all documentation is ultimately under the aegis of the quality control section, whose duty it is to determine that the documentation provides a complete record of the study, which would allow outside auditors to reconstruct the study and verify its integrity.

Thus, the analysis files, which will contain all the raw data, plots, and calculations necessary to determine the concentration of mercury species in the unknown, will be audited by the quality assurance section, then returned to the analytical laboratory for storage.

The sample integrity files include the receipt book, chain-of-custody sheets, shipping bills, and all such information necessary to accurately record the passage of specimen through the analytical system. As with all other documentation, these files are subject to routine audit by the quality assurance unit, which will verify their completeness.

The quality control files will be maintained in the quality assurance unit for each analytical run which is carried out in
the study. These files contain the records made during the routine validation of the data of each individual analytical run as described in Section 12.2.

The quality assurance files contain all the records made of the internal audits carried out by the quality assurance unit in monitoring facility and procedural compliance.

12.4 QA Reports

Quality assurance reports will be made on a monthly basis to management by the quality assurance unit and signed by the LQAU. Each report will contain a summary of the audits and data evaluation activities carried out by the unit in the previous month. Particularly highlighted will be evidences of out-of-control analyses, or of non-compliance of procedure or facilities. In these cases, the reports will identify what corrective action was taken, and document the return to the control condition.

Summaries of the major findings contained in the quality assurance reports to management will also be contained in a section of the monthly report submitted by ORRI to the study sponsor.

13.0 DELIVERABLES

The data submitted to the sponsor will be based on the requirements of Level E quality control, as designated by the sponsor. Thus, the data package will include the sample data, the method and reagent blank data, the control charts, calibration records, documentation of linearity and method detection limit, and the quality control data validation records. The final data deliverables will be presented to the sponsor at least 3 weeks prior to issuing the draft of the final report.

APPENDIX C-1

NAME	TITLE
Dr. Nathaniel Revis	Director & Senior Scientist

INSTITUTION AND LOCATION	DEGREE	YEAR	FIELD OF STUDY
Fairleigh Dickinson Univ., Rutherford, NJ	B.Sc.	1962	Chemistry
University Louvain Louvain, Belgium		1963-69	Medicine
Univ. of Glasgow, Scotland	Ph.D.	1972	Biochemistry

RESEARCH AND/OR PROFESSIONAL EXPERIENCE

Assistant Professor Univ. of Glasgow Dept. Cardiology Glasgow, Scotland	1972-1974
Professor and Scientist Department of Biomedical Sciences Univ. of Tennessee and Oak Ridge National Laboratory Oak Ridge, TN	1975-1981
Director and Senior Scientist Oak Ridge Research Institute Oak Ridge, TN	1981-present

AWARDS

Carnegie Research Fellow Award for graduate studies, University of Glasgow, Scotland	1969-1972
Fogarty Award, National Institutes of Health	1980
Small Business Administration, Minority Person of Year	1985

MEMBERSHIPS, SOCIETIES

Society of Toxicology	New York Academy of Sciences
Society of Biochemistry	National Academy of Sciences
American Heart Association	

PUBLIC SERVICE ACTIVITIES

1987-Present	Board member, Methodist Medical Center, Oak Ridge, TN
1978-Present	Member of Health Brain Trust, Black Congressional Caucus
1979-Present	Lecturer for the Talladega College - National Science Foundation Visiting Scientist Program
1978-1983	Coordinator, NIH Summer Hypertensive Training Program for Minorities from HBCU's.

PUBLICATIONS IN SCIENTIFIC JOURNALS: 58

NAME: Dr. George Holdsworth

TITLE: Senior Scientist

INSTITUTION AND LOCATION	DEGREE	YEAR	FIELD OF STUDY
Luton College Tech., GB.	H.N.C.	1968	Biology
Barking College Tech., GB.	M.I. Biol.	1970	Biochemistry
Univ. of Birmingham, Birmingham, GB.	Ph.D.	1973	Biochemistry

RESEARCH AND/OR PROFESSIONAL EXPERIENCE

Research Assistant (in Electron Microscopy) Unilever Research Laboratories Ltd. The Frythe, Welwyn, Herts, GB.	1964-1969
Research Student Department of Biochemistry University of Birmingham, GB.	1970-1973
Research Fellow Department of Biochemistry University of Birmingham, GB.	1973-1976
Research Biochemist (Senior) Lipid and Diabetes Research Laboratory St. Bartholomew's Hospital London, GB.	1976-1981
Visiting Assistant Professor Department of Pharmacology and Cell Biophysics University of Cincinnati College of Medicine Cincinnati, OH	1981-1982
Senior Scientist Oak Ridge Research Institute Oak Ridge, TN	1983-present

AWARDS

1969-70, Hertfordshire County Council; Major Undergraduate Scholarship
1970-73, Rank Hovis McDougall (Research) Ltd., Research Scholarship
1973-76, Medical Research Council; Postdoctoral Research Fellowship

PUBLICATIONS IN SCIENTIFIC JOURNALS: 42

NAME	TITLE
Tanya R. Osborne	Research Associate

INSTITUTION AND LOCATION	DEGREE	YEAR	FIELD OF STUDY
East Tenn. State Univ. Johnson City, TN	B.S.	1981	Microbiology
Dept. of Biomedical Studies U.T. Graduate School, Knoxville, TN	M.S. in progress		Biochemistry Genetics

RESEARCH and/or PROFESSIONAL EXPERIENCE

Research Associate Oak Ridge Research Institute Oak Ridge, TN	1982-present
Chief Technician Oak Ridge Research Institute Oak Ridge, TN	1988-present

PUBLICATIONS IN SCIENTIFIC JOURNALS: 5

NAME	TITLE
Barbara J. Gille	Quality Assurance Officer

EDUCATION	DEGREE	YEAR	FIELD OF STUDY
Ohio Dominican College	B.A.	1969	English
University of Tennessee,	M.B.A. (in progress)	1989	Business Knoxville

PROFESSIONAL EXPERIENCE

Technical Editor Plenum Press New York, New York	1969 - 1971	
Writer/Editor National Center for Research in Vocational Education Ohio State University Columbus, Ohio	1978	
Project Manager, Documentation Battelle Memorial Institute Columbus, Ohio	1979 - 1981	
Technical Editor Oak Ridge, Tennessee	1981 - 1983	Bechtel National, Inc.
Technical Editor E. I. du Pont de Nemours & Co. Savannah River Plant Aiken, South Carolina	1984	
Publications Supervisor Oak Ridge, Tennessee	1985 - 1988	The MAXIMA Corporation
Quality Assurance Officer Oak Ridge, Tennessee	1988 - present	Oak Ridge Research Institute

PUBLICATIONS 1

NAME	TITLE
Ms. Laurie Moulton	Information Scientist

INSTITUTE AND LOCATION	DEGREE	YEAR	FIELD OF STUDY
Memphis State University	B.S.	1980	Education
Johns Hopkins University	M.S.	1989	Epidemiology

RESEARCH AND/OR PROFESSIONAL EXPERIENCE

Research Assistant Biostatistics Vanderbilt University Nashville, Tennessee	1981 - 1985
Research Assistant	1986 - 1989

Special Studies, Preventive Medicine
Vanderbilt University
Nashville, Tennessee

Data Analyst 1987 - 1989
CCI Computing
Nashville, Tennessee

Programs Coordinator 1986 - 1989
Perinatal Data Management System
Vanderbilt University
Nashville, Tennessee

Information Scientist 1989 - present
Oak Ridge Research Institute
Oak Ridge, Tennessee

PROFESSIONAL DATA

Omicron Delta Kappa
Kappa Delta Pi
Mortar Board
Who's Who Among Colleges and Universities

PUBLICATIONS 1

APPENDIX C-2

Example of Specimen Container Label

OAK RIDGE RESEARCH INSTITUTE
113 UNION VALLEY ROAD
OAK RIDGE, TN 37830

Project #_____
Sample description_____
_____
Site_____ Location_____
Date_____ _____
Time_____ _____
Medium_____ Sample type_____

Sample by _____

ORRI # _____

APPENDIX C-3

OAK RIDGE RESEARCH INSTITUTE-CHAIN OF CUSTODY DOCUMENT

page _____ of _____

Project no. _____ Project name _____
Site _____ Sample type _____

Workers _____
_____

Sample ID	Location	Date/time	No.	Remarks

Relinquished by	date/time	Received by	date/time

APPENDIX C-4

APPENDIX C-5

Daily Data Work Sheet

Project #

Date:_____ Sample Number:
Sponsor_____ ORRI_____

Method of Extraction: Nitric Acid [ ], Sulfuric, Nitric, and Perchloric Acids [ ], Organic Solvents [ ], or Sodium Sulfide [ ].

Analyses Absorbance
Blanks: a_____ b_____ c_____ d_____ e_____ Mean

Standards: Absorbance

10 ng	a_____	b_____	c_____	Mean
50 ng	a_____	b_____	c_____	Mean
100 ng	a_____	b_____	c_____	Mean
200 ng	a_____	b_____	c_____	Mean
300 ng	a_____	b_____	c_____	Mean

Sensitivity Check (250 ng)_____

Absorbance
Spike Sample Extract: a_____ b_____ c_____ Mean

Absorbance Reference Standards
EPA a_____ b_____ c_____ Mean

ORRI a_____ b_____ c_____ Mean

No.	Volume of extract used	Dilution factor	Absorbance		
1	_____	_____	a_____	b_____	Mean
2	_____	_____	a_____	b_____	Mean
3	_____	_____	a_____	b_____	Mean
4	_____	_____	a_____	b_____	Mean
5	_____	_____	a_____	b_____	Mean
6	_____	_____	a_____	b_____	Mean
7	_____	_____	a_____	b_____	Mean
8	_____	_____	a_____	b_____	Mean

APPENDIX C-6

Data validation checklist

OAK RIDGE RESEARCH INSTITUTE
113 UNION VALLEY ROAD
OAK RIDGE, TN 37830

PROJECT # _____
ANALYSIS TYPE_____
RUN # _____
DATE _____

1. Necessary records for each analysis run:

a) [ ] Summary list of all field samples included in the run.

b) [ ] Machine print out for calibration

c) [ ] Calibration record.

d) [ ] Standard curve print out
[ ] Standard curve calculation
[ ] Standard curve plot

e) [ ] Machine print out for MDL
[ ] MDL calculation

f) [ ] Machine print out for assay spike
[ ] Assay spike calculated
[ ] Assay spike duplicated

g) [ ] Machine print out for matrix spike
[ ] Matrix spike calculated
[ ] Matrix spike duplicated

h) [ ] MSA procedure instituted
[ ] if yes
[ ] Machine print out available
[ ] Regression line calculated and plotted
[ ] r>=0.995
[ ] x intercept calculated

i) [ ] Reference samples analyzed
[ ] ORRI pools
[ ] other
[ ] Machine print out available
[ ] Duplicated
[ ] Reference sample calculated
[ ] Grand mean chart upgraded
[ ] R chart upgraded
[ ] Rs chart upgraded

j) [ ] Field samples analyzed

[ ] Machine print out available [ ] Duplicated

[ ] Field samples calculated

2. Calculations

a) Determination of standard curve:

[ ] Concentrations used [ ] Five plus blank

[ ] Duplicated [ ] RMD <20%

[ ] Ac-Abl plotted v conc. [ ] Regression line calculation

[ ] r>=0.995

b) Determination of MDL (where applicable)

[ ] Seven replicates plus blanks [ ] Ac-Abl read off std. curve

[ ] Mean conc+ SD checked [ ] MDL calculation check

c) Determination of assay spike

[ ] RMD <20% [ ] Ac-Abl read off std.curve

[ ] Vsp=Csp-Cs/Tsp x 100 [ ] Vsp = 85-115 (y or n)

N.B. Vsp = percentage recoevery of the spike (mg/l)
Csp = spiked sample concentration
Cs = unspiked sample concentration
Tsp = theoretical spike concentration

d) Determination of matrix spike

[ ] RMD<20% [ ] Ac-Abl read off std.curve

[ ] correct proportionality factors used

[ ] Vsp=Csp-Cs/Tsp x 100 [ ] Vsp = 60-140 (y or n)

final concentrations in mg/kg

e) Determination of unknown by MSA

[ ] sample plus 3 spikes [ ] duplicated

[ ] RMD<20% [ ] Ac-Abl read off std curve

[ ] correct proportionality factors used

[ ] Linear regression line drawn [ ] r>=0.995

[ ] determination of unknown from y where x = 0 spike

f) Determination of reference samples [ ] ORRI [ ] other

[ ] duplicated [ ] RMD<20%

[ ] Ac-Abl read off std.curve [ ] correct prop. factors

[ ] +5% ref value [ ] grand mean chart control

[ ] R chart control [ ] Rs chart control

g) Determination of unknowns

[ ] duplicated [ ] RMD<20%

[ ] Ac-Abl read off std.curve [ ] correct prop. factors

[ ] final values in draft summary if 2a, 2c, 2d, and 2f are in control.

[ ] check list v 1a to ensure that run is complete.

APPENDIX C-7

THE QA/QC INTERNAL AUDIT

Checklist

FACILITY COMPLIANCE

OAK RIDGE RESEARCH INSTITUTE
113 UNION VALLEY ROAD
OAK RIDGE TN 37830

Project #_____
Audit #_____
Date _____
signed_____

a) Chemicals (y or n)

name	correct grade	outdated	listed	site	date opened
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]
	[ ]	[ ]	[ ]	[ ]	[ ]

b) Sample records (y or n)

batch IDs	COC	receipt	labels	site checked
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]	[ ]

c) Equipment Calibration

piece	calibrated	recorded
_____	[ ]	[ ]
_____	[ ]	[ ]
_____	[ ]	[ ]
_____	[ ]	[ ]
_____	[ ]	[ ]
_____	[ ]	[ ]

d) Personnel training

name	Safety	SOPs	QA
_____	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]
_____	[ ]	[ ]	[ ]

PROCEDURAL COMPLIANCE

a) Extraction procedures date_____
signed_____

Acid digestion

glassware washed detergent [ ]
rinse [ ]
acid rinse [ ]

sample wts [ ] balance calibrated [ ]
digestion acid volumes [ ] time [ ]
hot plate temp [ ] rinse and vol. make up [ ]

Other comments _____
_____

Solvent digestion

glassware washed detergent [ ]
rinse [ ]
methylene chloride rinse [ ]

sample wts [ ] balance calibrated [ ]

extraction volumes [ ]
mixing times [ ] centrifuge speeds [ ]
phase separations [ ] centrifuge times [ ]
Other comments_____
_____

Sodium sulfide extraction

glassware washed detergent [ ]
rinse [ ]
methylene chloride rinse [ ]

sample wts [ ] balance calibrated [ ]

extraction volumes [ ] centrifuge speed [ ]
mixing times [ ] centrifuge time [ ]
pools Other [ ]

Comments _____
_____

b) Analytical procedures date_____
signed_____

Atomic Absorption Spectrophotometer

operating solutions	[ ]	standard solutions	[ ]
dates	[ ]	dates	[ ]
dilutions	[ ]	machine warm up	[ ]
calibration response	[ ]		
duplicate blanks	[ ]	standard curve	[ ]
		plotted	[ ]
		linear	[ ]
duplicate assay spikes	[ ]	reference standards	[ ]
duplicate matrix spikes	[ ]	thawed	[ ]
		mixed	[ ]
position in sample track	[ ]		

Other comments_____

Gas chromatography

gas flow rate	[ ]	septa	[ ]
oven temp	[ ]	detector clean	[ ]
injector temp	[ ]	detector wipe	[ ]
detector temp	[ ]	program	[ ]
attenuation	[ ]	wipe	[ ]
operating solutions	[ ]	standard solutions	[ ]
dates	[ ]	dates	[ ]
dilutions	[ ]	machine warm up	[ ]
calibration response	[ ]		
duplicate blanks	[ ]	standard curve	[ ]
		plotted	[ ]
		linear	[ ]
duplicate assay spikes	[ ]	reference standards	[ ]
duplicate matrix spikes	[ ]	thawed	[ ]
		mixed	[ ]
position in sample track	[ ]		

Other comments_____

APPENDIX C-8

OAK RIDGE RESEARCH INSTITUTE
113 UNION VALLEY ROAD
OAK RIDGE TN 37830

CORRECTIVE ACTION REPORT

Project #_____ Analyst_____
date _____ Out-of-control event_____
run #_____ _____
Analyte _____
LQAC _____

Sample IDs in run _____
(data rejected) _____
1st Recheck

Finding _____
date _____
Analyst _____
run # _____

Corrective actions

detector calibration		[ ]	[ ]	[ ]
new standard solutions		[ ]	[ ]	[ ]
(linear)		[ ]	[ ]	
conductivity		[ ]	[ ]	[ ]
method blank		[ ]	[ ]	[ ]
calculations		[ ]	[ ]	[ ]
weights correct		[ ]	[ ]	[ ]
volumes correct		[ ]	[ ]	[ ]
balances recalibrated		[ ]	[ ]	[ ]
equipment service	GC	[ ]	[ ]	[ ]
	AA	[ ]	[ ]	[ ]

2nd recheck

	run #1	run #2	run #3	run #4	run #5
date	_____	_____	_____	_____	_____
analyst	_____	_____	_____	_____	_____
liear stds	_____	_____	_____	_____	_____
reference #1 X	_____	_____	_____	_____	_____
reference #2 X	_____	_____	_____	_____	_____
within warning limits #1	[ ]	[ ]	[ ]	[ ]	[ ]
#2	[ ]	[ ]	[ ]	[ ]	[ ]

Summary_____
_____
date_____ LQAC_____

APPENDIX C-9

Gas chromatograph-Routine Maintenance Schedule

Equipment	Type of Maintenance	Frequency
Mainframe exterior surface	Clean	as required
Air filter	Clean/Replace	6 months
Cartridge tape unit	Clean	1 week
Moisture trap	Conditioning	When gas is changed
	Repacking	2 months, or after 10 conditionings
Chemical filter	Conditioning	2 months, or when gas is changed
Carrier gases	Leak check	as required
Column	Conditioning	as required
	Repacking	as required
Ni Catalyst	Conditioning	6 months, or as required
Septum	Replace	Every 20 injections
ECD	Frequency check	Daily
	Thermal clean	1 month
	NRC wipe test	6 months

A log book will be kept with the machine containing signed and dated records of all routine maintenance activity.

APPENDIX C-10

Mercury hydride system

Routine Mainenance Procedures

Equipment	Type of Maintenance	Frequency
Mainframe	Clean	as required
Immersion tube	Rinse in dilute acid	as required
Transport system	purge with distilled water	daily
Flashback arrestor	Clean in dilute HCl	weekly
Windows	Clean in dilute acid	weekly
Quartz cell	Clean	6 months
Flange gasket	Leak check/Replace	6 months
Transfer hose	Check/Replace	as required

APPENDIX D

RESUMES OF KEY PERSONNEL

WILLIAM R. ADAMS, JR., P.E., Vice President

Qualifications Summary

Mr. Adams has extensive professional experience in the administration of state and federal environmental laws as the result of more than 12 years with the U.S. Environmental Protection Agency (U.S. EPA) as Region I Administrator, and with the Maine Department of Environmental Protection as Commissioner. His experience includes leadership in hazardous waste management and implementation of several new statutes, including the innovative Oil Conveyance and Site Location statutes. As a result of this experience and his continuing relationships with regulatory agencies, Mr. Adams has a thorough understanding of these agencies and their processes. He has served as project officer on over 25 of C-E Environmental's (C-EE) recent contracts to assist federal, state and local agencies and industrial clients in the assessment of hazardous waste problems and the proposal of remedial actions.

Education

B.S./Civil Engineering, 1951, University of South Carolina

Professional Licenses

Professional Engineer - Maine, New Hampshire, Massachusetts, Florida
Land Surveyor - Maine

Relevant Experience

State Experience--Mr. Adams served as Commissioner of the State of Maine Department of Environmental Protection for eight years. In this position, Mr. Adams administered state and federal environmental laws. He drafted, presented, received legislative approval, and implemented the governmental reorganization plan. Additional responsibilities included service as a member of Maine State Pesticides Board, Historic Preservation Commission, Mining Bureau, Coastal Development and Conservation Advisory Committee.

Federal Experience--Mr. Adams served as Regional Administrator, U.S. EPA, Region I. As Regional Administrator, Mr. Adams administered federal environmental laws in the New England region, placing major emphasis on improvement of state/federal relations. He developed working relationships with the six state governors and the state program administrators, and implemented a partnership in the development of the annual state/EPA agreements. He pursued delegation of authority to the states, making the region a leader in this effort.

Mr. Adams worked closely with the industrial community in an attempt to solve the region's environmental problem through cooperation rather than litigation.

Hazardous Waste Management--As Vice President for C-EE, Mr. Adams is responsible for the administration of many of the firm's hazardous waste

projects. In this role he provides professional liaison prior to project startup, during the course of a project and after completion to assure that clients receive services responsive to their needs.

Mr. Adams has served as Project Officer on many of C-EE's larger hazardous waste projects. These include C-EE's contract with Ebasco to conduct Remedial Investigation/Feasibility Studies (RI/FS) and Remedial Design for U.S. EPA's REM III contract in New England. He has served as Project Officer on hazardous waste projects for numerous state agencies including Maine, Massachusetts, Rhode Island, New Jersey, and Florida.

In addition to these projects, Mr. Adams serves as Project Officer for hazardous waste projects C-EE is conducting for such large firms as United Technology, New York State Electric and Gas, New England Power Company, Monsanto Chemical Co. and several projects being administered by committees formed by Potential Responsible Parties.

Military Hazardous Waste Projects

Mr. Adams has worked extensively on C-EE's hazardous waste projects for the military. He serves as Project Officer on C-EE's contract with Martin Marietta's Oak Ridge National Laboratory which provide services for hazardous waste projects to the Air Force, Air National Guard and Navy. This Contract includes major RI/FS-type work at over 8 military installations. In addition to these projects, Mr. Adams is Project Officer for work at three different Naval installations. His extensive knowledge of state and federal regulatory agencies, as well as the Department of Defense's Installation Restoration Program, make him a valuable asset in successful conduct of hazardous waste projects throughout the United States.

Additional Experience

Mr. Adams serves on C-EE's quality assurance team for the firm's remedial action assessment and long-term environmental monitoring at Love Canal in Niagara Falls, New York. He is a frequent speaker on hazardous waste matters and recently served as moderator for a day long program sponsored by the New England Council on SARA Title III and community Right-To-Know legislation.

R. ANTHONY ALLEN III, Technical Guidance and Review

Qualifications Summary

Mr. Allen is the Manager of Florida Operations for C-EE. His areas of expertise include project management of multi-site, multi-disciplinary remedial programs conducted under IRP, CERCLA, RCRA and UST environmental regulatory programs for public and private sector clients and RCRA/CERCLA conflict negotiations with USEPA. He is currently the Program Manager in charge of 5 contracts providing IRP, CERCLA and RCRA services to Southern Division of Naval Facilities Engineering Command for facilities in the Southeastern United States.

Education

B.S./Plant and Soil Sciences, 1982, University of Maine
A.S./Forest Management, 1979, University of Maine

Professional Registration

Certified Professional Soil Scientist, 1987 State of Maine
Certified Professional Soil Scientist, 1987 American Registry of Certified Professionals in Agronomy, Crops and Soils (ARCPACS)

Relevant Experience

IRP, Brunswick Naval Air Station(BNAS); Brunswick, Maine--Mr. Allen directed the Verification phase of the NACIP confirmation study at seven sites at BNAS and presented the findings to the Maine DEP and U.S. EPA. He designed the Characterization phase of study at three BNAS sites and presently negotiated approval of the work plan with the Maine DEP and U.S. EPA.

IRP/CERCLA, Massachusetts Military Reservation (MMR); Cape Cod, Massachusetts--Mr. Allen was responsible for the development and supervision of the Phase I Remedial Investigations at a 100-acre base landfill, current fire training area and petroleum fuel storage area at MMR. Mr. Allen was responsible for all communications and interactions with the Massachusetts DEQE and U.S. EPA Region 1 which occurred as part of the Phase I Remedial Investigation.

IRP/CERCLA/RCRA, Mayport Naval Station; Mayport, Florida--Mr. Allen developed and implemented the IRP Site Investigation at 11 sites at Mayport Naval Station. His responsibilities as Program Manager for the project have also included technical development and presentation of a RCRA Facility Investigation work plan and support to the Navy in negotiating RCRA and CERCLA conflicts with the Florida DER and U.S. EPA Region IV.

IRP/CERCLA/RCRA, Dover Air Force Base; Delaware--As Task Order Manager, Mr. Allen was responsible for the Remedial Investigation/Feasibility Study and a RCRA Part B Closure at Dover Air Force Base. This included the technical supervision of a minority owned business subcontractor conducting the RI Phase of the project and the assembly and supervision of a feasibility study team. Mr. Allen has been responsible for the

development and presentation of the project technical approach and results to Oak Ridge National Laboratory, Hazardous Materials and Testing Center, Military Airlift Command Headquarters, Dover Air Force Base, USAF, Delaware NREC, and U.S. EPA Region III. He was also responsible for assisting Dover AFB negotiate a Secretary's Order with Delaware NREC.

IRP Feasibility Study, Indian Head Naval Ordinance Station; Maryland--Mr. Allen is the Task Order Manager in charge of development of an investigation at a mercury contaminated wetland site. Responsibilities include gathering the remaining chemical and engineering data necessary to perform a feasibility study at the site. Innovative new analytical techniques are being applied in this project to speciate the mercury present and remediate the site.

IRP/RCRA, Panama City Naval Coastal Systems Center, Florida--As IRP Program Manager, Mr. Allen is responsible for allocating the appropriate technical resources for effective and timely completion of the IRP Characterization Study and RCRA Facility Assessment and Investigation in compliance with a RCRA HSWA Permit issued and administered by USEPA Region IV for the Panama City facility. Mr. Allen was responsible for effectively negotiating with Florida DER and USEPA Region IV on the issue of RCRA HSWA and CERCLA conflict at this Naval facility.

IRP/UST, David Taylor Naval Ship Research and Development Center, Maryland--As Task Order Manager, Mr. Allen was responsible for the Confirmation Study, Feasibility Study and Remedial Design of a leaking underground fuel storage tank system. He was responsible for technical direction, project management and administration in coordination with the Project Manager at Martin Marietta Energy Systems, Inc. which manages the contract for Chesapeake Division of Naval Facilities Engineering Command.

UST, Florida Statewide Navy Storage Tank Management Program, Florida--As Program Manager for this contract Mr. Allen is responsible for providing storage tank management and petroleum contamination services for 22 Naval Facilities in the State of Florida involving over 1,000 underground storage tanks. The project has included the development of Tank Inventory Management System (TIMS) database which provides physical, management and planning information on the Navy's entire storgae tank population. This database is being considered for a Navy standard by NEESA. Mr. Allen also was part of the negotiation team which was successful in negotiating alternate procedure requests with FDER which amortized the expenditure of millions of dollars over a period of five years.

CERCLA, Potters Septic Tank Site, Maco, North Carolina-- As Site Manager Mr. Allen is reponsible for developing the technical approach and implementing an RI/FS at this USEPA NPL Site.

IRP/CERCLA, Naval Air Station Whiting Field, Milton, Florida- As Program Manager Mr. Allen is responsible for directing the development of Remedial Investigation and Feasibility Study Planning Documents for Southern Division of Naval Facilities Engineering Command. In addition, Mr. Allen is responsible for Site Investigation activities being conducted at

Outlying Landing Field Barin in Alabama and supporting the Navy in interactions with Florida DER, USEPA Region IV and the local community.

CERCLA/IRP/RCRA/UST/TSCA, Comprehensive Long-Term Environmental Action Navy (CLEAN), Southeastern United States--Mr. Allen is Deputy Program Manager for the CLEAN contract with Southern Division of Naval Facilities Engineering Command. Under this contract Mr. Allen will be responsible for directing the technical resources necessary to conduct environmental response actions at all Navy installations located in 6 southeastern states for the next 10 years. The scope of this work will cover CERCLA/IRP, RCRA, TSCA and UST regulation compliance. Additionally, Mr. Allen will provide support to the Navy as they negotiate Federal Facility Agreements with the USEPA Region IV and State Regulatory agencies for the installations included on the NPL.

Additional Experience

Mr. Allen has direct experience using inovative technologies applied as investigative tools including geophysics, soil gas surveys, field gas chromatograph screening, and mercury vapor analysis. Mr. Allen's disciplined areas of expertise include soil chemistry, soil genesis and morphology, land application of wastes, and soil microbiology.

JANET A. BURRIS, Aquatic Toxicologist

Qualifications Summary

Ms. Burris' areas of expertise include aquatic toxicology, biomonitoring for toxics control, aquatic ecology, and ecological risk assessment. She is responsible for assessing the fate and transport of chemicals in the environment, their bioavailability to aquatic organisms, and the potential for transport in food chains. She is also responsible for biological characterizations and ecotoxicological evaluations at hazardous waste sites as well as for the development of quantitative methods for assessing risk to aquatic organisms exposed to toxic and hazardous materials.

Education

M.S.P.H./Environmental Sciences and Engineering, 1987, University of North Carolina at Chapel Hill
B.A./Zoology, 1984, Depauw University

Affiliations

Society of Environmental Toxicology and Chemistry
North American Benthological Society
American Society for Testing and Materials

Relevant Experience

EPA Region I Ecological Assessment Work Group--Ms. Burris was reponsible for the direction of the work group which included representatives from EPA, NOAA, and the U.S. Fish and Wildlife Service. The group provided EPA Region I with risk assessment support and prepared a risk assessment guidance document: "Guidance for Ecolgical Risk Assessments at Superfund Sites" which was used as part of the EPA Superfund Environmental Evaluation Manual.

Oak Ridge National Laboratory; Oak Ridge, Tennessee--Ms. Burris conducted ecotoxicology research examining the bioavailability and *in situ* toxicity of inorganic contaminants in streams impacted by hazardous waste disposal activities. She was also involved with the development and implementation of biomonitoring techniques for hazardous waste and industrial effluents.

University of North Carolina; Chapel Hill, North Carolina--Ms. Burris was responsible for evaluating human health effects associated with exposure to the by-products of water chlorination.

Publications

Burris, J.A. and Steward, A.J. Snail Dispersal Patterns as a Behavioral Indicator of Ambient Water Toxicity. Society of Environmental Toxicology and Chemistry, Eighth Annual Meeting; Pensacola, FL; November 1987.

Burris, J.A. and Stewart, A.J. Metal Accumulation and Survival of the Snail Elimia clavaeformis in a Contaminated Stream. North American Benthological Society, 36th Annual Meeting; Tuscaloosa, AL; May 1988.

Baldwin, A.H., J.A. Burris, and M.J. Donato. "Wetlands Assessment at Sites Contaminated with Toxic Chemicals." Society of Wetland Scientists, Ninth Annual Meeting; Washington, DC; June 1988.

Burris, J.A., C. Zarba, and J. Downing. "Application of Sediment Quality Criteria in Ecological Risk Assessment." Society of Environmental Toxicology and Chemistry, Ninth Annual Meeting; Washington, DC; June 1988.

Burris, J.A. and A.J. Stewart. Behavioral Responses of Marked Snails as Behavioral Indicators of Water Quality; Environmental Toxicology and Chemistry; In Press.

Additional Experience

As an aquatic toxicologist, Ms. Burris has prepared several ecological risk assessments as part of Remedial Investigation/Feasibility Study activities at hazardous waste sites. These assessments have involved the development of quantitative methodologies for assessing risk to aquatic and terrestrial organisms and modeling of the fate and transport of toxic chemicals in the environment. Other project work has included the development of risk methods for pesticides for the Maine Board of Pesticides Control, wetlands assessments at hazardous waste sites, and the application of statistical methods in RCRA groundwater compliance monitoring. Ms. Burris has received training in exposure modeling and the use of computer models (SARAH2, WASP, EXAMS, and FGETS) in predicting fate and transport of toxic materials.

JACK DAVIS, Environmental Scientist

Qualifications Summary

Mr. Davis has more than nine years of experience in planning and managing environmental investigations for state, federal, and industrial clients. He also has extensive technical experience in developing remedial action programs for cleanup, treatment and/or disposal of hazardous waste materials.

Education

M.P.H./Public Health, 1979, University of Michigan
B.S./Biology, 1974, Baldwin Wallace College

Relevant Experience

As Technical Director on RCRA and CERCLA investigations at uncontrolled hazardous waste sites and hazardous waste disposal facilities, Mr. Davis was responsible for remedial investigation and RCRA facility investigation work plans; directing day-to-day activities necessary to implement investigations such as monitoring well installations, and soil and groundwater sampling; managing and evaluating data; and preparing and presenting the report.

As Technical Environmental Director for the Florida Department of Transportation (FDOT) I-595 project, Mr. Davis was involved with the investigation of numerous right-of-way acquisitions to identify potential hazardous waste sites located on the right-of-way acquisitions. He was instrumental in the development of state policy with regards to right-of-way acquisition involving hazardous waste sites.

As Technical and Project Manager in charge of petroleum contamination projects, Mr. Davis is responsible for the development of site investigation work plans, management of all on-site activities necessary to perform the investigations, data management and evaluation, and report preparation.

ALLEN J. IKALAINEN, Project Manager

Qualifications Summary

Mr. Ikalainen's areas of expertise include civil and environmental engineering and project management. His work experience includes three years of civil engineering with the New England Division, U.S. Army Corps. of Engineers, fifteen years of field investigation, systems analysis and environmental permit development experience with the U.S. Environmental Protection Agency and four years experience in project management and technical direction of hazardous waste site investigations and remedial action feasibility studies. At E.C. Jordan Co., his hazardous waste experience includes management of site surveys for hazardous waste contamination, groundwater contamination investigations and Superfund site remedial investigations, feasibility studies and remedial design.

Education

M.S./Environmental Engineering, 1978, Northeastern University
B.S./Civil Engineering, 1967, Worcester Polytechnic Institute

Professional Licenses

Professional Engineer - Maine, Massachusetts

Relevant Experience

Installation Restoration Program, U.S. Air Force--Mr. Ikalainen is Deputy Program Manager for Jordan's Hazardous Waste Remedial Action projects at Air Force facilities in the Northeast. The projects include RI/FS, remedial design and underground storage tank investigations.

REM III Program, U.S. Environmental Protection Agency--For RI/FS projects at Superfund sites in Region 1, Mr. Ikalainen directed day to day program activities as part of the REM III contract team for EPA - Region 1. This included planning and conducting RI/FS projects at eleven National Priorities List sites and RCRA Compliance Monitoring at operating industrial waste disposal facilities.

Remedial Investigation/Feasibility Study, New Bedford Harbor Superfund Site, MA--Mr. Ikalainen served as Site Manager for the overall RI/FS for this 1000 acre site contaminated with PCBs and metals. He prepared a detailed project management plan and was responsible for management and technical direction of subcontracted physical/chemical and food chain modeling, coordinating with the U.S. Army Corps. of Engineers Waterways Experiment Station laboratory on pilot dredging, disposal studies, and completion of the overall Feasibility Study including a risk assessment, bench tests of treatment technologies and detailed evaluation of remedial alternatives.

Remedial Investigation/Feasibility Study, Pine Street Canal Superfund Site, VT--To enable the State of Vermont Agency of Transportation to decide if they should proceed with plans to construct a highway through a portion of this coal tar site. Mr. Ikalainen directed preparation of a RI/FS Work Plan defining field sampling, data evaluation, risk assessment and remedial alternative evaluations as required under CERCLA.

Assessment of Nature and Extent of Groundwater Contamination for a Confidential Semiconductor Manufacturer--Mr. Ikalainen managed the analysis and evaluation of groundwater monitoring data to define the nature and extent of organic solvent contamination and contaminant transport processes for this large semiconductor manufacturer. The evaluation was done as part of the facilities corrective action plan under the Resource Conservation and Recovery Act (RCRA) permit program.

Assessment of Abandoned Manufacturing Sites for Past Disposal of Hazardous Wastes, ME--On behalf of a commercial developer Mr. Ikalainen managed assessment of historic land uses, and field evaluations of surface and subsurface contamination at two abandoned foundry and metal fabrication sites. Field evaluations included sampling of soil and groundwater for chemicals classified as hazardous by state and federal environmental agencies.

Evaluation of Toxicity of Manufacturing Discharge, ME--Mr. Ikalainen directed a toxicity assessment program utilizing bioassay procedures for an electrical products manufacturer. The program included chemical analysis of process water discharges, receiving waters and selection of appropriate test species for assessment of heavy metal toxicity.

Additional Experience

Mr. Ikalainen has managed wastewater disposal planning and an evaluation of a large fuel storage tank farm for assessment of tank contents and potential development. He is experienced in federal regulatory programs dealing with industrial waste disposal, ocean disposal and wetlands protection.

JARET C. JOHNSON, P.E., Senior Consultant, Hazardous Wastes Treatment

Qualifications Summary

Mr. Johnson's 18 years of engineering experience have emphasized the treatment of industrial wastes. He has provided project management, technical direction, regulatory negotiations, and public presentations leading to the implementation of remedies at hazardous waste sites. He is the author of numerous technical papers on treatment processes for hazardous materials, groundwater and industrial wastewater.

Education

M.S./Environmental Engineering, 1973, University of North Carolina
B.S./Materials Engineering, 1967, Rensselaer Polytechnic Institute

Professional Licenses

Professional Engineer - Massachusetts, Maine

Relevant Experience

Management of Aquifer Restoration/Site Closure Program, W.R. Grace Site, Acton, MA--At a NPL site being remedied by the responsible party, Mr. Johnson managed all phases of remedial studies, design, and construction. An important part of the problem definition and remedial evaluation was a sophisticated numerical model of groundwater flow and contaminant transport. This modeling effort not only aided in the selection of a cost-effective remedy, but also provided clear graphics that aided the understanding of regulators and the public. Aquifer restoration is now being accomplished by a pumping, treatment, and recharge system designed and installed under Mr. Johnson's direction.

Feasibility Study, Former Transformer Salvage Yard, MA--Mr. Johnson identified and compared remedial alternatives for soils contaminated by PCBs for a group of PRPs that had sent transformers to this site. Both innovative and conventional remedies were evaluated; criteria included effectiveness, time to implement, potential future risks/liabilities, and cost. Complicating the environmental setting at this site is a regional plume of organics in groundwater that originates off-site, but which partially underlies this site. Therefore, identification of compounds that could, and could not, be related to transformer handling was performed.

Feasibility Study, Yaworski Lagoon NPL Site, CT--The subject of this study was a former industrial waste lagoon that was filled with industrial trash and covered in the early 1980's. The old lagoon had been used in previous decades for disposal of organic waste liquids, as well as non-flowing materials in drums. Because the lagoon cover materials are permeable, the lagoon continues to be a source of groundwater contamination. Mr. Johnson led a group of engineers and scientists in identifying and evaluating remedial measures for the source of contaminants and for the affected groundwater.

Feasibility Study, Cannon's Engineering Corp. NPL Site, MA--This was one of the first FS's to be preapred after the enactment of the 1986 CERCLA amendments (SARA). The site received, stored, shipped, and treated wastes from various industries during the 1970's. Mr. Johnson lead a team of engineers in identifying and evaluating remedial techniques for soil, groundwater, tanks, and building surfaces. Contaminants included PCBs, solvents, polycyclic aromatic hydrocarbons, and heavy metals.

Litigation, Plastics Manufacturer, MA--Mr. Johnson represented an industrial client in negiations with a neighboring industry which has accused C-EE's client of being the source of contaminants found on and beneath the neighboring industry's property. As a result of these negotiations, a lawsuit against C-EE's client was withdrawn.

Treatment of Coal Tar Wastes, Northeast Utilities Service Co., CT--Mr. Johnson was the project engineer for a field demonstration of microbial treatment of coal tar contaminated soils. In this effort he led a field crew of five scientists and two heavy equipment operators. Twelve tons of tarry soil were excavated and tilled into treatment plots with clean soil. Nutrients and bacterial cultures were added, and biodegradation was monitored. He has also contributed to field investigations at two other coal tar sites. This experience has made him familiar with the analytical chemistry, fate, transport, and remedial options for coal tar. This work conformed with the 1986 CERCLA Amendments (SARA).

Biological Treatment of Chlorinated Hydrocarbons in Water--Working with a group of chemists and microbiologists, Mr. Johnson provided engineering services to develop practical methods for the biodegradation of trichloroethylene, dichloroethylene, and vinyl chloride. His work included conceptual designs of systems for in-situ treatment of groundwater, and for treatment of wastewater or pumped groundwater in bioreactors. He also provided engineering advice on projects to identify the breakdown products, and identified hardware that could be used to carry out biodegradation at field scale. Because the bacteria carrying out the degradation use methane as their carbon source, innovative designs were required to deliver this methane safely and in adequate quantities (despite its low solubility).

Design of Tank Farm and Chemical Distribution Center, Large Semiconductor Manufacturer, VA--This project included design and construction services for six bulk storage tanks for solvents, pumping and piping systems, spill controls and alarms, and a building for the safe storage and distribution of several hundred chemical products, many of which are toxic, flammable, or otherwise hazardous. Mr. Johnson led the efforts of process, instrumentation, and electrical engineers, and coordinated with architects and site layout (civil) engineers. Construction cost was approximately $10 million.

Evaluation of Fixation/Stabilization Processes for Hazardous Wastes, U.S. EPA, Cincinnati, OH--Mr. Johnson was project manager in this effort, in which three types of waste were each treated by fifteen types of fixation

processes to immobilize contaminants. Leaching tests were performed to evaluate the success of each process/waste combination. This project provided Mr. Johnson with knowledge of the vendors/ developers of various processes, as well as the strengths and weaknesses of the available systems for fixation/stabilization.

At Sea Research on Treatment of Oil Spills, American Petroleum institute, Washington, D.C.--Mr. Johnson coordinated the efforts of several organizations in making, treating, and monitoring experiemental oil spills 50 to 70 miles off the coast of New Jersey. He led a successful effort to obtain a research permit from the U.S. EPA to conduct these experimental spills. Treatment was achieved by spraying a chemical dispersant from a helicopter. In addition to the helicopter, he directed the movements of four remote sensing aircraft and three research vessels.

MARGARET E. LAYNE, P.E., Senior Environmental Engineer

Qualifications Summary

Ms. Layne's areas of technical expertise include hazardous waste management, water and wastewater treatment process design, pollutant fate and transport modeling, and exposure assessment. She has seven years of experience in treatment process design, health and environmental assessment, and regulatory support. Currently, Ms. Layne is project manager for a RCRA Facility Investigation at the U.S. Naval Station, Mayport, Florida. Other project management experience includes coordination of reports, reviews, and background documents on hazardous waste characteristics and management practices for EPA's Office of Solid Waste.

Education

M.S./Environmental Engineering, 1984, University of North Carolina
B.E./Environmental Engineering, 1980, Vanderbilt University

Professional Licenses

Professional Engineer - North Carolina

Relevant Experience

RCRA Facility Investigation, U.S. Naval Station, Mayport, Florida--Ms. Layne is currently serving as project manager for the RFI of the 17 identified Solid Waste Management Units at NAVSTA Mayport. A draft work plan will be submitted to USEPA Region IV for approval in January 1990.

Air Toxics Workshop--Ms. Layne coordinated preparation of the companion document for the Conference on Waste Reduction for Industrial Air Toxic Emissions, sponsored by the North Carolina Pollution Prevention Pays Program, in Greensboro, N.C., April 1989.

Preparation of the 3rd Edition of SW-846, "Test Methods for Evaluating Solid Waste"--Ms. Layne was project manager for the 3rd edition of EPA's solid waste testing manual, SW-846. She was responsible for coordination with subcontractors and an EPA work group.

Exposure Assessment Workshop--She prepared and presented a seminar on pollutant fate and transport modeling as part of a two-day workshop on exposure assessment for the Health Assessment Section of EPA's Office of Solid Waste.

PCB Risk Assessment--Ms. Layne performed pollutant fate and transport modeling for a case study assessing the health risk of PCB-contaminated soils to nearby residents.

LAYNE, MARGARET E. (continued)

Additional Experience

Ms. Layne provided technical support for nationwide surveys of hazardous waste generators and treatment, storage and disposal facilities. She has also prepared background documents on the use of the TCLP and the development of health-based regulatory thresholds for proposed revisions to the RCRA Toxicity Characteristic.

Ms. Layne has performed air pollution dispersion modeling for PSD permit applications and developed preliminary designs for wastewater and solid waste management systems at coal gasification and liquefaction facilities. She has also conducted equipment performance testing and prepared operation and maintenance manuals for municipal wastewater treatment plants.

BARBARA SAUNDERS-PRICE, Manager, Engineering Chemistry

Qualifications Summary

As head of the Engineering Chemsitry Department, Dr. Saunders-Price manages and provides liason for all chemistry related matters associated with the firm's operations. The Engineering Chemistry Department is responsible for chemistry related functions in a variety of projects for the EPA, DOD's Installation Restoration Program, and private clients. Major project areas include remedial investigation/feasibility studies at uncontrolled hazardous waste sites, coordination of chemical data interpretation, and technical review of chemistry related sections of environmental projects from sampling plans through remedial designs.

Dr. Saunders-Price's areas of technical expertise include physical, radiation, and nuclear chemistry. She has over twenty years of experience in research, development, and assessment projects in reactions of chemicals and nuclear products in the environment; and in the detection of chemicals and protection of human health and the environment. She has been responsible for training and coordinating uniform technical approaches to many projects and programs.

Education

Ph.D./Physical Chemistry, Harvard University, 1972
M.A./Chemistry, Harvard University, 1968
B.A./Chemistry, Hunter College of the CUNY, 1966

Relevant Experience

Ebasco Envirosphere, Lyndhurst NJ--Dr. Saunders-Price was a Principal Scientist and Risk Assessment Lead for the Environment and Health Division. She worked on a variety of projects for private clients and the EPA. She was responsible for planning, monitoring, and supervising chemical analyses and surveys; reviewing, and validating analytical laboratory work; interpreting laboratory and field data; performing risk assessments and environmental impact analyses for remediation of contaminated sites; and directing the work of environmental scientists and engineers. She provided technical review and quality control for all EPA remedial investigation reports. She was responsible for training younger staff and for planning and reviewing all contaminant fate, transport, and risk assessment portions of environmental projects.

Applied Science and Analysis, ASA, Inc., Slidell, LA--President and Technical Director of consulting firm specializing in chemical protection and decontamination for military and civil defense applications. Responsible for company planning and marketing, interactions with clients, contracts and billings. Supervised all technical projects and reports. Traveled extensively in U.S. and abroad (Europe and Asia) to work with national laboratories and industries developing new techniques/technologies for environmental and human health protection.

Supervised and conducted projects for the Department of Defense (DOD) evaluating new technologies and methods for air purification and detection of chemicals in contaminated environments. Combined environmental threat with life support and protection requirements for individual/personal protection equipment (chemical protective suits, respirators, gloves/boots, monitors).

Conducted projects with private firms in the design, development, and applications of air purification, filtration, and gas separation equipment to meet military and environmental protection requirements. Supervised and conducted evaluations of detectors and methods for field validation of air purification efficacy and protection factors.

Executive Editor and co-founder of professional journal specializing in technical and political aspects of nuclear, biological, and chemical defense, and in the protection of human health and environment from these threats. Reviewed and edited all articles for technical veracity and applicability.

NASA National Space Technology Laboratories, Engineering Laboratory, Computer Science Corporation, NSTL MS--Principal Scientist and Joint Manager of programs assessing DOD capabilities and improvement programs in chemical defense and life support systems.

Supervised and conducted a wide range of evaluations of detector systems from automatic integrated monitors for demonstration of EPA compliance to sophisticated satellite remote sensing and simplistic point samplers for highly toxic gases. Evaluations and assessments of personal protection equipment, decontaminants, and chemical compatibilities plastics and polymers.

Senior scientist for USATHAMA Installation Restoration Program's feasibility study for Ft. Monroe, VA, a national historic site, characterized by inorganic and organic chemical contamination and used and unexploded munitions. Supervised and reviewed chemical analysis and sampling plan, laboratory and field data, and risk, engineering alternatives and economic assessments for site remediation. Also participated in work plans for site investigations of Rocky Mountain Arsenal, Basin F in Colorado.

Assistant Chemist, Chemical Engineering Division, Argonne national Laboratory, Argonne, IL.--Assistant Chemist in nuclear fuel reprocessing and waste management research including radiolytic damage of organic solvents, reprocessing of fuels in molten metals and salts, actinide chemistry, radioactive tracers, and a variety of radiolytic sources. Responsible for development of process flow sheets, equipping and organizing laboratories, writing technical reports, and planning and conducting experiments. Coordinated and worked with scientists involved in assessing and ameliorating the effects of radioactive waste.

Designed reprocessing processes to reduce the amount of emitted fission products in the larger waste streams.

Participated in design, development and tests of the aging of various glasses for calcining radioactive wastes. Assisted in leaching studies of experimental glasses containing fission products.

Reviewed and assessed the fate of released plutonium, americium and cesium resulting from possible accidents. Included studies of the adsorption of various Pu (VI) compounds on soils and its penetration to a shallow aquifer.

Member of DOE team to review the designs for the nuclear fuel reprocessing plant under construction in Barnwell, S.C.

Postdoctoral Appointment, Chemistry Division, Argonne National Laboratory, Argonne, IL.--Postdoctoral appointment in Radiation Chemistry Group performing energy and environmental research. Used pulse radiolysis with a linear accelerator for kinetic analyses and mechanistic studies elucidating the reactions of coal during combustion, oxidation, liquefaction, and hydrogenation.

Initiated a research program in oxidative decomposition of chlorinated hydrocarbons and other pollutants as a model for water purification by radiation and ozonolysis.

Conducted research in gas phase radiation chemistry including reactions of OH radicals with hydroxylamines as part of an air pollution program to expand the existing data base and rate constants using radiolytically produced gas radicals.

Assisted and conducted research in transuranic radiation chemistry of U, Pu, Am, Np using kinetic experiments with linear accelerator.

Chromatographic analyses of radiation products using HPLC, GC-MS, and MS-MS. Responsible for equipping and organizing laboratories, planning, and conducting experiments, and writing technical papers and reports.

Additional Experience

Coordinator and primary instructor, Chemical Safety Course, MidHudson ACS Section, Fall 1986.

Adjunct Faculty Member, State University of New York, Purchase NY, January to June 1988. Taught "Safety for Experimental Scientists".

CHRISTIAN E. OLSON, Project Administrator

Qualifications Summary

M. Olson's responsibilities include contract administration and project control for the Jordan Company. In this capacity he prepares contract proposals, develops schedules and budgets and monitors subcontractor activities.

Education

B.S./Business Administration, 1983, University of Southern Maine

Relevant Experience

Installation Restoration Program--Mr. Olson is reponsible for contracts and financial administration for nine IRP sites currently underway. This includes proposal development, progress reporting, budget monitoring and forecasting. Mr. Olson communicates with subcontractors to obtain Proposal bids and to ensure that terms are being met under existing contracts.

Michigan Department of Natural Resources--Mr. Olson was responsible for monitoring subcontractor activities on this multi-task project. In this role, he communicated regularly with subcontractors and Jordan personnel regarding budgets and schedules. Mr. Olson also worked closely with the Project Manager to develop subcontractor bids and agreements to track, review and process invoices.