SITE OPERATIONS PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

May 1992

Prepared for

Town of Oyster Bay

Prepared by

Geraghty & Miller, Inc.
125 East Bethpage Road
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SITE OPERATIONS PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
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May 13, 1991

Geraghty & Miller, Inc. is submitting this work plan to the Town of Oyster Bay for work to be performed at the Syosset Landfill. The work plan was prepared in conformance with Geraghty & Miller's strict quality assurance/quality control procedures to ensure that the work plan meets the highest standards in terms of the methods used and the information presented.

Respectfully submitted,

GERAGHTY & MILLER, INC.

Vincent J. Glasser
Senior Scientist/Project Manager

for AJB
Andrew J. Barber
Senior Consultant/Project Director

Michael F. Wolfert
Vice President/Project QA/QC Manager

VJG/AJB/MFW:mb
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SECTION I

SITE OPERATIONS PLAN OVERVIEW
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

1.0 INTRODUCTION

Geraghty & Miller, Inc. was retained by Lockwood, Kessler and Bartlett, Inc. (LKB) under contract to the Town of Oyster Bay (Town) to prepare this Site Operations Plan (SOP) for conducting the Second Operable Unit (OU-2) Remedial Investigation (RI) of the Syosset Landfill Syosset, New York.

This SOP for the OU-2 (off-site) RI for the Syosset Landfill was prepared to provide a detailed description of the methodologies for conducting the OU-2 RI. The rationale and scope of the OU-2 RI program was described in the April 1991 work plan (Geraghty & Miller, Inc. 1991) that was approved by the United States Environmental Protection Agency (USEPA) in their letter dated July 5, 1991. Specifically, the OU-2 RI will consist of an Off-Site Ground-Water Study to determine the horizontal and vertical extent of a plume of leachate that may be emanating from the landfill and to determine the plume thickness on-site. Leachate-impacted ground water was detected during the On-Site Ground-Water Study that was performed as part of the First Operable Unit (OU-1) RI (Geraghty & Miller, Inc. 1989). The OU-2 RI field program also includes an Off-Site Subsurface Gas Study to determine whether landfill gases are migrating off-site from the landfill.

This document was prepared in accordance with the most recent USEPA guidance document (Interim) (USEPA 1988) for the conducting of a remedial investigation/ feasibility study (RI/FS) and also follows the terms of the Administrative Order signed by the USEPA and the Town of Oyster Bay on June 19, 1986. The completion of the tasks and deliverables described in the SOP will ensure consistency with the National Contingency Plan (NCP).

1.1 PURPOSE AND SCOPE

The OU-2 RI will consist of an Off-Site Ground-Water Study and an Off-Site Subsurface Gas Study. The purposes of the Off-Site Ground-Water Study will be to determine the plume thickness, define the off-site extent of a leachate plume that may be emanating from the landfill, and confirm the direction of ground-water flow. Leachate-impacted ground water was detected beneath the landfill and inside the northern property boundary (downgradient) during the OU-1 RI. The purpose of the Off-Site Subsurface Gas Study is to determine the extent of off-site subsurface gas migration from the landfill where elevated concentrations of methane were detected during the OU-1 RI.

During the Off-Site Ground-Water Study, eleven monitoring wells will be installed at five locations; two of the proposed drilling locations are on-site where a single deep monitoring well will be installed next to existing well clusters (SY-3 and SY-6) (Figure 1). One on-site location is upgradient of the landfill where a deeper well will be installed at existing upgradient Well Cluster SY-6 to provide additional upgradient water-quality data. The second on-site location is downgradient of the landfill where a deeper well will be installed at existing Well Cluster SY-3 to determine the plume thickness on-site. The three proposed off-site drilling locations will each have three wells per location. An exploratory boring will be drilled using a Barber rig (cased boring method) to install the proposed deep well on-site next to existing Well Cluster SY-3 and the deep well at the middle location of the three proposed off-site well cluster locations.

During the Off-Site Subsurface Gas Study, three shallow gas monitoring wells will be installed on or in the vicinity of the Great Eastern Printing Company property, which is located adjacent to the southwestern portion of the landfill but separated from it by the Long Island Rail Road (LIRR). In addition to the three off-site gas monitoring wells, five new gas monitoring well clusters will be installed on-site as part of the OU-1 Remedial

Design Program, in accordance with the amendment to the OU-2 RI work plan (Geraghty & Miller, Inc. 1991) that was submitted by the Town of Oyster Bay in their letter to the USEPA dated June 26, 1991. Although the additional on-site work will be conducted as part of the OU-1 Remedial Design Program, the monitoring results will be presented in the OU-2 RI report.

1.2 FORMAT

This document consists of four distinct parts or sections as follows: Section I: Site Operations Plan (SOP) Overview; Section II: Sampling and Analysis Plan (SAP); Section III: Quality Assurance Project Plan (QAPP), and Section IV: Health and Safety Plan (HASP). All four sections are included as one volume. Because each of these sections serve a separate specific function and may be used individually, they each contain tables of contents, references, and appendices. However, all tables and figures are placed in tabs at the back of this volume. This format facilitates removal of individual sections. The SAP provides detailed descriptions of the methodologies of the OU-2 RI field program. The QAPP ensures that the precision, accuracy, representativeness, comparability, and completeness (the PARCC parameters) of the collected data are known, documented, and adequate to satisfy the data quality objectives of the investigation. The HASP provides assurance that field personnel will conduct their tasks in a manner that will minimize health and safety risks to themselves, the public, and the environment.

1.3 PROJECT ORGANIZATION

The Town is the owner of the Syosset Landfill and will serve as the Project Coordinator. LKB is the RI/FS Program Manager and Geraghty & Miller is the RI Program Manager. Technical and administrative roles for performance of the RI/FS were assigned to staff who have the appropriate qualifications to carry out their respective responsibilities. More details on project responsibilities are provided in Section 8.0 (Project Organization)

of the SAP (Section II) and Section 2.0 (Project Organization and Responsibilities) of the QAPP (Section III). Resumes of key project personnel are provided in Appendix A of the SOP Overview (Section I).

1.4 SITE DESCRIPTION AND HISTORY

The Syosset Landfill is located in central Nassau County in the Town of Oyster Bay, Syosset, New York. The site is roughly rectangular in shape and encompasses approximately 38 acres. The office and facilities of the Town of Oyster Bay Department of Public Works (TOB-DPW) are located adjacent (east) to the landfill and occupy approximately 15 acres; together the landfill and the adjacent facilities total approximately 53 acres. Currently the Town controls access to the site, which is surrounded by a fence. Topographically, the site is relatively flat and at a similar elevation to the surrounding area. As illustrated in Figure 1, the site is bounded by the Long Island Expressway and Miller Road to the southeast, Cerro Wire & Cable Corporation to the southwest, and the LIRR to the northwest. A residential area and the South Grove Elementary School border the site to the northeast. The entire landfill area is enclosed by a 6-foot high cyclone fence. The three proposed off-site ground-water monitoring well clusters are located northeast of the landfill and the off-site gas monitoring wells are located southwest of the landfill (see Figure 1). The OU-2 RI will also present and evaluate gas monitoring results obtained from the on-site gas Cluster Wells CW-2, 4, 5, 6, 7, and 8 that will be installed and monitored during the OU-1 Remedial Design Program (see Figure 1).

2.0 REFERENCES

Federal Register. 1988. National Oil and Hazardous Substances Pollution Contingency Plan, Proposed Rule. December 21, 1988, pp. 51394-51520.

Geraghty & Miller, Inc. 1989. Interim Remedial Investigation Report, Syosset Landfill, Syosset, New York. August 1989.

Geraghty & Miller, Inc. 1991. Work Plan for the Second Operable Unit Remedial Investigation at the Syosset Landfill, Syosset, New York. April 1991.

U.S. Environmental Protection Agency (USEPA) 1988. Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA. Office of Emergency and Remedial Response. USEPA Washington, D.C., October 1988.

APPENDIX A

SECTION I: SITE OPERATIONS PLAN OVERVIEW

APPENDIX A

RESUMES OF KEY PROJECT PERSONNEL

300655

NAME: RICHARD W. LENZ, P.E.
ADDRESS: TOWN OF OYSTER BAY
DEPARTMENT OF PUBLIC WORKS
150 MILLER PLACE
SYOSSET, NEW YORK 11791

QUALIFICATIONS:

- Bachelor's Degree from Polytechnic Institute of New York.
- Professional Engineer's license.
- Worked for Nassau County Dept. of Public Works - Sanitation and Work supply for 16 year lateral Sewer Construction and Design of Sewage Treatment Plants.
- Project Manager for approximately $100 million - Plant Construction in the Design Phase/Construction Phase.
- Liaison between County and D.E.C., responsible for quarterly reports and grant applications.
- Working for Town of Oyster Bay for 2 1/2 years - responsible for finalizing Remedial Investigation Report with EPA for Syosset Landfill.
- Draft and finalization of Feasibility Study.
- Discussed modifications to Consent Decree.
- Responsible for several other types of projects from Design through Construction.

JOB TITLE: PROJECTION MANAGER/CIVIL ENGINEER III

TELEPHONE NO.: (516) 921-7347

THERESA CURRAN HENEVELD, P.E.
Environmental Engineer

LKB CONSULTING ENGINEERS
Since 1889

EDUCATION/REGISTRATION

BCE, Civil Engineering, Manhattan College, 1982
MEE, Environmental Engineering, Manhattan College, 1983

Registered Professional Engineer, State of New York

EXPERIENCE

Ms. Heneveld is an Environmental Engineer with over seven years of experience in solid waste management, the preparation of hazardous waste site remedial investigations, feasibility studies and remedial action designs, the design of water and wastewater collection and treatment systems, the development of hydraulic and hydrologic analyses, and the preparation of environmental impact statements. At LKB, Ms. Heneveld is currently a Project Engineer for several environmental engineering projects involving hazardous waste site remedial investigations and feasibility studies, groundwater monitoring programs, landfill gas system evaluations and environmental impact statements.

As project engineer for the Syosset Landfill Remedial Investigation/Feasibility Study process, Ms. Heneveld developed work plans, reports, remedial action design alternatives and construction cost estimates for this Superfund site in accordance with USEPA, CERCLA and NYSDEC requirements. The remedial action design alternatives involved capping and gas control technologies, in addition to related site work and air and groundwater monitoring programs. In addition, Ms. Heneveld is currently Project Engineer for the Second Operable Unit Remedial Investigation (OU2 RI) process at the former Syosset Landfill. The OU2 RI will identify off-site conditions attributable to the site.

Ms. Heneveld recently acted as design engineer for the Groundwater Remediation Program at the Old Bethpage Solid Waste Disposal Complex (OBSWDC), which was an integral part of the Remedial Action Plan developed for this Superfund site. Her responsibilities included: the design of approximately 1.25 miles of a groundwater transmission line from five groundwater recovery wells; the design of a 1.5 MGD groundwater treatment plant to remove Volatile Organic Compounds, Iron and Manganese; and a discharge system consisting of 2,000 feet of piping and a series of diffusion wells to recharge the treated water back into the underlying aquifer. The treatment processes she designed included an air stripper, four manganese greensand pressure filters, three activated carbon columns, and sludge handling and dewatering equipment.

As Environmental Engineer for a major New York City engineering firm, Ms. Heneveld was principal design engineer for a performance evaluation of a wastewater treatment plant in Suffolk County, NY. She was responsible for investigations, innovative treatment technologies, selecting a treatment process and obtaining approval from the Suffolk County Department of Health.

JOHN P. LEKSTUTIS, P.E.
Vice President - Project Dir./Principal-in-Charge

LKB CONSULTING ENGINEERS Since 1889

EDUCATION/REGISTRATION

B.E., Civil Engineering, Manhattan College, 1965
M.E., Sanitary Engineering, Manhattan College, 1966

Registered Professional Engineer in NY, NJ, CT, MA, RI, ME, VT, WA

EXPERIENCE

Mr. Lekstutis has more than 25 years of experience in the fields of civil and sanitary engineering, environmental assessment and hazardous waste management. During this time, Mr. Lekstutis has developed an expertise in the management of hazardous waste site remedial investigations, feasibility studies and remediation design and construction programs. He has managed more than five such programs.

Mr. Lekstutis has been actively involved with the First Operable Unit remedial investigation/feasibility study process for the Syosset Landfill. This project entailed negotiation of a Consent Decree with USEPA and the development of a remedial investigation and feasibility study for the capping and closure of the Syosset Landfill. As part of his responsibilities, Mr. Lekstutis is also assisting the Town in obtaining EQBA Title 3 funds for implementation of the design and construction phases of this program. In addition, Mr. Lekstutis is currently managing the Second Operable Unit Remedial Investigation for the site.

Mr. Lekstutis's recent project experience involves the successful completion of the remedial investigation and design program at the Old Bethpage Landfill for the Town of Oyster Bay. The program addressed three separate remedial action components, which are either under construction, in the bid phase or in the final design and approval phase. These include: a groundwater remediation a project, which entails the capture and treatment of up to 1.5 million gallons per day of contaminated groundwater; the design of a landfill capping and closure program that addresses severe slope conditions; and the design and installation of an up-graded perimeter landfill gas migration control system, comprised of extraction wells, a header system, a blower facility and a combustion unit for the destruction of captured gases.

As project manager, Mr. Lekstutis's activities involved the management and control of a multi-disciplined team of engineers and scientists. He was also responsible for interfacing with the USEPA, NYSDOL and NYSDEC in the negotiation of the landfill's Consent Decree, in obtaining approvals for both the conceptual and final design plans and specifications, and in obtaining EQBA Title 3 funds for the project. In addition, he had to interface with local community residents to keep them advised of the program's progress and to respond to related areas of concern. Construction for all remedial components is expected to be completed before the end of 1992, at which time the Old Bethpage Landfill will be the first Long Island landfill to be fully remediated under USEPA's Superfund Program.

ANDREW J. BARBER

Senior Project Advisor & Senior Associate -
Corporate Safety Manager

CREDENTIALS/REGISTRATION

B.S. Chemistry, Union College, 1979

PROFESSIONAL AFFILIATIONS

American Chemical Society
American Industrial Hygiene Association
American Society of Safety Engineers
Association of Ground Water Scientists and Engineers (NWWA)

FIELDS OF SPECIALIZATION

- Hazardous waste site remediation
- Field sampling and analysis
- Health and safety
- Analysis of water-quality and geochemical data
- Fate and transport of contaminants in soil and ground-water systems
- Regulatory analysis

EXPERIENCE SUMMARY

Prior to joining Geraghty & Miller, Inc., Mr. Barber was employed by Peabody Coastal Services of Linden, New Jersey, as a member of its Hazardous Material Response Team. While at Peabody, he was directly involved with cleanup activities at several uncontrolled hazardous waste sites. His responsibilities included data and sample collection, supervision of heavy equipment operations, and classification and segregation of waste materials.

Mr. Barber joined the firm in 1980; his responsibilities include geochemistry, project management, and management of the company's Health & Safety Program. As a geochemist with the firm, he has worked on numerous ground-water investigations to characterize the transport and fate of chemical contaminants. He has extensive experience in the development and implementation of subsurface investigations and monitoring plans.

As a project officer, Mr. Barber has directed investigative and remedial efforts at numerous sites in the northeast. Projects have been conducted under RCRA and CERCLA, as well as under various state or local regulations, and have included discharge permit issues, underground storage tank removal, hazardous waste storage, property transfer and closure plans. Activities include: negotiation with Federal, state and/or local regulatory agencies; preparation of work plans and reports; management of field activities; scheduling; regulatory compliance; quality assurance/quality control, and contract administration. Mr. Barber is also the manager of the Albany, New York office, which opened in November, 1989.

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As Corporate Safety Manager for the firm, Mr. Barber has developed and managed the company health and safety program. This program includes medical monitoring and extensive training, in compliance with OSHA hazardous waste regulations. He is also responsible for the preparation, review, and implementation of site-specific health and safety plans.

KEY PROJECTS

- Supervised remediation of disposal pit containing residual solvents and PCB oil in upstate New York.

- Developed and applied field techniques to analyze volatile organic contaminants in soil and water samples using a portable gas chromatograph.

- Continually involved in analysis and presentation of water quality and geochemical data and the determination of contaminant fate and transport mechanisms.

- Project officer for the Remedial Investigation/Feasibility Study for a CERCLA landfill site on Long Island.

- Co-authored three volume technical report as part of on-going litigation support for a major environmental lawsuit.

- Project officer for investigation and remedial activities at a major petroleum transfer facility.

- Continually involved in developing work plans for taking representative samples for contaminants in soil, air, and water.

- Project officer for feasibility study, ground-water monitoring and remediation for major CERCLA site on Long Island.

- Negotiated, and subsequently directed, investigation and remediation of contaminated soils at an industrial property on Long Island.

- Provided technical oversight and review of USEPA contractor performance at a municipally-owned CERCLA site.

- Currently negotiating Remedial Investigation/Feasibility Studies on behalf of two separate industrial clients listed on the New York State Superfund list.

- Provided expert testimony on behalf of an industrial client in relation to alleged discharge permit violations.

KEY PROJECTS (Continued)

- Continually involved as project officer on environmental assessments of commercial and industrial real estate prior to purchase or refinancing.

- Officer for project involving the evaluation of the hydrogeologic aspects of candidate landfill sites for a two-county region in upstate New York.

SELECTED PUBLICATIONS

Barber, A.J., and O.C. Braids, Application of a Portable Organic Vapor Analyzer in Ground-Water Contamination Investigations, "Proceedings of the Second National Symposium on Aquifer Restoration and Ground-Water Rehabilitation," National Water Well Association, Worthington, Ohio, 1982 (paper presented May 27, 1982).

Barber, A.J., Case Histories in Safety Programs for Hazardous Waste Site Investigations, Paper presented to the National Water Well Association, "Aquifer Restoration and Ground-Water Monitoring," Third National Symposium, Columbus, Ohio, May 25-27, 1983.

Lobasso, T. and A.J. Barber, A Monitoring and Removal Plan for Leaked Propane in the Vadose Zone, "Proceedings of Characterization and Monitoring of the Vadose (Unsaturated) Zone," National Well Water Association, Worthington, Ohio (Paper presented December 9, 1982, Las Vegas, Nevada).

Barber, A.J. and B.V. Moran, Health and Safety Considerations for Working with Hazardous Chemical Wastes - A Consultant's Perspective, Paper presented to the American Chemical Society, Twentieth Middle Atlantic Regional Meeting, Baltimore, Maryland (paper presented September 4, 1986).

Barber, A.J., Human Error and Groundwater Sampling, Waste Age, Volume 17, No. 6, pp. 183-184.

Barber, A.J. and R.D. Raskin, Application of a Portable Gas Chromatograph to Subsurface Contamination Investigations, "Proceedings of the First Annual Hazardous Materials Management Conference/Central," Tower Conference Management, Glen Eleyn, IL, 1988 (paper presented March 15, 1988, Chicago, IL)

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TOM C. ENG

Senior Engineer/Industrial Hygiene
Northeast Regional Health & Safety

CREDENTIALS/REGISTRATION

B.S. Chemical Engineering, City College, 1984
Industrial Hygienist in Training (IHIT), American Board of Industrial Hygiene, 1989

PROFESSIONAL AFFILIATIONS

American Industrial Hygiene Association, National and Local Chapter Member
American Board of Industrial Hygiene, Member

FIELD OF SPECIALIZATION

- Occupational Health & Safety
- Identification, Evaluation and Control of Occupational Disease Exposures
- Development of Comprehensive Control Strategy
- Field sampling and data analysis

EXPERIENCE SUMMARY

Prior to joining Geraghty & Miller Environmental Services, Inc., Mr. Eng was the New York Division Industrial Hygienist/Pollution Control Coordinator for Liberty Mutual Insurance Group, Inc. His responsibilities included consulting with clients on industrial hygiene and pollution liability issues, designing, implementing and teaching in training seminars for both clients and internal personnel, field surveys to identify potential health hazards, and sampling and data analysis.

Associated with Liberty Mutual for 5 plus years, Mr. Eng has extensive industrial hygiene experience in a major cross section of industries and has appreciable knowledge of current occupational health and safety standards and guidelines. He has accompanied and represented clients in regulatory meetings as a technical expert in the negotiation process to determine adequate control measures.

Joining the firm in 1990, his responsibilities include managing portions of the company's Health and Safety Program, preparing and reviewing Site Safety Programs, providing technical expertise in the field of industrial hygiene, and participating in training programs design for clients.

KEY PROJECTS

- Formaldehyde and ethylene oxide surveys for New York area health facilities.
- Indoor air quality surveys for commercial and industry facilities.
- Laser hazard surveys for medical and high tech equipment manufacturers.

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KEY PROJECTS (Continued)

- Noise surveys for a variety of industries, from food to industrial clients, punch press operations, bakeries, print rooms.., etc.

- Lead surveys in various industries, foundries, indoor firing ranges, electronics ... etc.

- Organic solvent surveys, degreasing operations, spray painting, printing press, electronics..., etc.

- Ionizing radiation survey for a watch manufacturing client.

- Hazard communication program development and implementation for various industries.

- Assisting clients in implementing monitoring programs to address occupational disease exposures.

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VINCENT J. GLASSER

Senior Scientist

CREDENTIALS/REGISTRATION

B.A. Environmental Studies, S.U.N.Y.-Binghampton, 1980

PROFESSIONAL AFFILIATIONS

Association of Ground-Water Scientists and Engineers (NWWA)
North Jersey Water Conference

FIELDS OF SPECIALIZATION

- Coordinating field drilling and sampling programs
- Utilizing and maintaining data - collection instruments
- Contouring of ground-water flow regimes and contaminant plumes
- Constructing hydrogeologic cross sections
- Coordinating and carrying out pumping tests and analyzing pumping test data

EXPERIENCE SUMMARY

In addition to his duties as a field hydrogeologist, Mr. Glasser has been a member of Geraghty & Miller's sampling van team, collecting ground-water samples in accordance with federal and state protocols. Prior to joining Geraghty & Miller in 1981, Mr. Glasser was employed as a well driller's assistant by Habernicht Water Well Systems in Bellmore, New York.

KEY PROJECTS

- Conducted field investigation for a Superfund project involving contamination of municipal and private water-supply wells. Responsible for supervising the installation of monitoring wells and coordinating water and soil sampling protocols with EPA representatives.

- Supervised monitoring well installation at a chemical storage facility and collected water samples after completion. Prepared report that was submitted to the county as required under county directive.

- Analyzed pumping test data from an investigation of a gas spill affecting a complex fractured-rock aquifer system. The results of this test were used to determine the zone of capture under variable pumping conditions and to design schemes for containment of the spill.

- Supervised the redevelopment of abatement wells at a hazardous waste disposal facility.

- Carried out field investigation at a chemical plant in order to determine if a waste lagoon liner was leaking. Supervised the installation of a series of monitoring wells and sampled them for tracer compounds introduced into the lagoon. Contributing author to the report.

- Measured volatile organic compounds in soils with an Organic Volatile Analyzer at a former dump site. Field program was designed to confirm the presence of a "hot spot" of volatile contamination as predicted by a computer model of the ground-water system. In addition, soil samples were collected and analyzed for volatile organic contamination.

CASEY SUSAN SCHMIDT

Staff Scientist

CREDENTIALS/REGISTRATION

M.S. Geology, University of Nevada, Las Vegas, 1987
B.S. Geology, University of Idaho, 1980
Registered Geologist, State of Arkansas

PROFESSIONAL AFFILIATIONS

Geological Society of America
Association of Ground Water Scientists and Engineers (NWWA)
American Geophysical Union
Sigma Xi (The Scientific Research Society)

FIELDS OF SPECIALIZATION

- Supervision of field projects relating to the investigation and evaluation of sediment and ground-water contamination at municipal landfills and industrial facilities.
- Design and supervision of drilling programs utilizing a variety of drilling methods in unconsolidated rock and bedrock.
- Geophysical well logging and interpretation.
- Field sampling.
- Geologic mapping and remote sensing interpretation.
- Evaluating and organizing data using Excel, D-base, Golden Graphics, word processing and other computer software.

EXPERIENCE SUMMARY

Ms. Schmidt has been with Geraghty & Miller since 1988. She participates in hydrogeologic studies of hazardous-waste sites where she is responsible for installing monitoring wells, collecting water samples, preparing geologic logs of bore holes, and preparing maps and cross sections of the hydrogeologic data. She has helped write workplans, proposals and final reports for a wide range of projects relating to municipal landfills and industrial sites, site assessments, and hydrocarbon remediation.

Prior to joining Geraghty & Miller, Inc. in 1988, Ms. Schmidt participated in the geologic evaluation of sites used for underground nuclear weapons testing for the University of California, Lawrence Livermore Laboratory. She has supervised the drilling and geophysical logging of bore holes and the quality assurance of the data obtained. She has applied experience using a wide variety of geophysical tools to interpret the structural geology and stratigraphy of geologic settings. She has done extensive geologic mapping in a variety of geologic terrains for the U.S. Geological Survey and for the Center for Volcanic and Tectonic Studies, University of Nevada, Las Vegas.

KEY PROJECTS

- Supervised several field investigations to delineate the lateral and vertical extent of ground-water contaminated with chlorinated solvents and hydrocarbons in New Jersey and Connecticut.

- Prepared a hydrogeologic report and data interpretation for a municipal landfill in Nassau County, New York in compliance with 6NYCRR, Part 360 monitoring.

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KEY PROJECTS (Continued)

- Prepared budgets, technical specifications and drilling contracts for RCRA and CERCLA sites.

- Responsible for scheduling of ground-water sampling and investigations at a municipal landfill in Nassau County, New York.

- Research on the recent volcanism in Crater Flat, Nye County, Nevada and how it might affect the adjacent proposed nuclear-waste storage facility at Yucca Mountain.

- Responsible for scheduling and field supervision of drilling and geophysical work performed on drill holes at the Nevada Test Site.

PUBLICATIONS

Schmidt, C.S., Smith, E.I., 1985, The Tertiary volcanic stratigraphy of the southern McCullough Range, Clark County, Nevada: Abstracts of the Symposium on Southwestern Geology and Paleontology, Dept. of Geology, Museum of Northern Arizona, Flagstaff, Arizona.

Anderson, J.L., Young, E.D., Clarke, H.S., Orrel, S.E., Winn, M., Schmidt, C.S., Smith E.I., 1985, The geology of the McCullough Range Wilderness Area, Clark County, Nevada: University of Southern California Technical Report submitted to the U.S. Geological Survey, 45 p.

Smith, E.I., Schmidt, C.S., 1986, Mid-Tertiary volcanic rocks of the McCullough Range, Clark County, Nevada: Geological Society of America Abstracts with Programs, v. 18, No. 2, p. 187.

Schmidt, C.S., Smith, E.I., 1987, The McCullough Pass Caldera: a mid-Miocene caldera in the central McCullough Mountains, Clark County, Nevada: Geological Society of America Abstracts with Programs, v. 19, No. 6, p. 447.

Smith, E.I., Schmidt, C.S., Mills, J.G., 1988, Mid-Miocene volcanoes in the Lake Mead area of southern Nevada and Northwestern Arizona, in Weide, D.L. Faber, M.L., eds., This Extended Land, Geological Journeys in the Southern Basin and Range: Geological Society of America, Cordilleran Section, Field Trip Guidebook, pp. 107-122.

3/91

GREGORY K. SHKUDA, Ph.D.

Senior Project Advisor
and Associate

CREDENTIALS/REGISTRATION

Ph.D. Organic Chemistry, New York University, 1976
M.S. Organic Chemistry, New York University, 1973
B.A. Chemistry, New York University, 1968

PROFESSIONAL AFFILIATIONS

American Chemical Society
Association of Ground Water Scientists and Engineers (NWWA)

FIELDS OF SPECIALIZATION

- In-depth knowledge of federal and state environmental regulations
- Evaluation of complex ground-water quality problems
- Knowledge of biodegradation of complex organics in ground water
- Provides expert testimony and reports on hazardous waste compliance issues
- Review of QA/QC plans and development of analytical protocols for litigation purposes

EXPERIENCE SUMMARY

Dr. Shkuda, a Senior Project Advisor for Geraghty & Miller's New York office, evaluates complex problems relating to the chemical contamination of ground water and soil. Dr. Shkuda participates in every aspect of the firm's work relating to ground-water contamination and provides expert testimony on behalf of the firm's clients.

Prior to joining the firm in 1986, Dr. Shkuda was an environmental scientist at the New York State Department of Law, Environmental Protection Bureau where he participated in major environmental litigation as a member of a unique staff of attorneys and scientists. From 1980 to 1982, Dr. Shkuda was employed by a consulting engineering firm where he developed sampling strategies to characterize hazardous waste sites located in two EPA regions as part of a national contract. As part of this work, Dr. Shkuda supervised the chemical assessments needed to characterize hazardous waste sites and was responsible for collecting enforcement data. During the period 1976-1980, Dr. Shkuda was a Research Associate in the Department of Microbiology and Immunology, Albert Einstein College of Medicine.

KEY PROJECTS

- Directed the Remedial Investigation/Feasibility Studies at two municipal landfills on Long Island. Dr. Shkuda was responsible for negotiating the scope of the work plans for these programs with the New York State Department of Environmental Conservation. Implementing the studies, coordination with the client, state and local community.

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KEY PROJECTS (Continued)

- Directed the hydrogeologic investigation at a Superfund site located in Eastern Tennessee for a Fortune 500 client. This site received large quantities of liquid chemical wastes. Dr. Shkuda was responsible for the evaluation of previously collected data, proposal of additional scopes-of-work and negotiations with the State of Tennessee. He participated in the delineation of the extent of contamination and proposed solutions to the problem of the collection of the non-aqueous phase liquids encountered at the site.

- Directed the environmental investigation at a closed aircraft manufacturing facility on Long Island. Dr. Shkuda's role included negotiations with the State as to the scope of the investigation, evaluation of the monitoring data, supervision of RCRA closure activities and coordination of proposals for cleanup activities.

- Directed hydrogeologic investigations for a major defense contractor at seven sites located in three states. Dr. Shkuda played a key role in the collection and review of historical data, preparation of work plans, supervision of field investigations, report preparation negotiations with Federal and state regulatory agencies, and the implementation of remedial activities at several of the facilities.

SELECTED PUBLICATIONS

Geller, S., Wei, S.C., Shkuda, G.K., Marcus, D.M., Brewer, C.F.: Carbon-13--Enriched Tetra-L-Alanine Hapten to Fab' Fragments of Antipoly(L-Alanine) Antibodies. Biochemistry 1980, 3614-3623.

Shkuda, G.K., Wei, S.C., Geller, S., Brewer, C.F., Marcus, D.M.: A ^{13}C NMR Study. New York University, New York 1976.

Shkuda, G.K.: The Decomposition of Bicyclic Diazo Compounds: A Mechanistic Study. New York University, New York 1976.

Rodgers, J.A., Shkuda, G.K.: Training and Safety Considerations in Using Self-Contained Breathing Apparatus (SCBA) and Tethered Cascade Breathing Apparatus (TCBA) in Hazardous Atmospheres at Uncontrolled Hazardous Waste Sites. American Chemical Society 184th Annual Meeting, Kansas City, Mo., September 184th Annual Meeting, Kansas City, Mo., September 1982.

10/90

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LAUREN E. SJOGREN

Scientist III

CREDENTIALS/REGISTRATION

M.S. Analytical Chemistry, University of Maryland, MD, 1988
B.S. Chemistry, C.W. Post Center of Long Island University, NY, 1983

PROFESSIONAL AFFILIATIONS

American Chemical Society
Association of Ground Water Scientists and Engineers (NWWA)

FIELDS OF SPECIALIZATION

- Field sampling
- Chemical data validation

EXPERIENCE SUMMARY

Prior to joining Geraghty & Miller, Ms. Sjogren was employed by a consulting firm as an environmental chemist. Her responsibilities included validation of chemical data, assisting in the preparation of quality assurance project plans and participation in field sampling programs. The chemical validation of data included the screening of all laboratory data to determine the laboratory compliance with New York State Department of Environmental Conservation (NYSDEC) Contract Laboratory Protocols (CLP). Ms. Sjogren's professional experience also includes laboratory positions in both academic and industrial sectors.

As a research assistant at the University of Maryland, Ms. Sjogren utilized instrumental neutron activation analysis of several types of environmental samples. In addition, aqueous samples were also analyzed for sulfate and nitrate using ion chromatography. During her tenure at the University of Maryland, Ms. Sjogren also served as a summer intern at FMC Corporation's Baltimore, Maryland facility as a process laboratory technician. This position included responsibility for conducting pesticide and herbicide analysis utilizing gas chromatography.

Upon completion of the Master's Program at the University of Maryland, including the preparation of a research thesis, Ms. Sjogren took a position as quality control chemist with a chemical manufacturing firm in New York. Responsibilities included stability testing and release of finished products according to United States Pharmacopeia (USP) specifications utilizing liquid chromatography, gas chromatography and UV spectroscopy.

As a chemist and scientist with Geraghty & Miller, Ms. Sjogren has been responsible for the validation of chemical data by USEPA CLP for Federal Superfund sites in Indiana and New Jersey.

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PUBLICATIONS/PRESENTATIONS

American Chemical Society; Middle Atlantic Regional Meeting "Atmospheric Concentrations and Wet Deposition of Elements Used for Tracing Source Emissions," May 1987.

Thesis Research: Wintertime Composition of Precipitation, Atmospheric Particles and Gases in College Park, MD.

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MICHAEL F. WOLFERT

Vice President/Hydrogeologist

CREDENTIALS/REGISTRATION

B.A. Geology, Hofstra University, 1971
Certified Professional Geologist: American Institute of Professional Geologists, States of Arkansas, Indiana, North Carolina, South Carolina, Tennessee, & Commonwealth of Virginia

PROFESSIONAL AFFILIATIONS

Association of Ground Water Scientists and Engineers (NWWA)
Geological Society of America
American Institute of Professional Geologists

FIELDS OF SPECIALIZATION

- Planning, organizing and managing large-scale projects concerning the evaluation and remediation of ground-water contamination and ground-water resource development
- Planning, organizing, and interpreting complex aquifer testing programs
- Development and evaluation of hydrogeological data

EXPERIENCE SUMMARY

Mr. Wolfert has been a member of the firm since 1971 and has carried out ground-water investigations in Canada, Puerto Rico and throughout the United States. He has been involved with the development of ground-water supplies for industry, municipalities and private water companies. He has carried out ground-water management studies for municipalities and planned, supervised, and analyzed data from many complex aquifer evaluation tests. He has planned and organized many investigations of ground-water contamination covering a wide range of contaminants and hydrogeologic settings including Superfund sites. He has also been involved with evaluation and implementation of remedial measures at hazardous waste sites.

KEY PROJECTS

- Developed a 2 million gallons per day ground-water supply for a new town (proposed 20,000 people) in coastal plain of New Jersey next to a sensitive wildlife area (Brigantine Wildlife Preserve). The study proved the availability of the water supply while also quantifying the effect of the proposed withdrawal on other major ground-water users in the region. Effects on surface water were also evaluated.

- Developed a detailed description of the geology and ground-water system of Middlesex County, New Jersey for the county's 208 study.

- Designed field and analytical program to determine movement and extent of elemental phosphorus in the subsurface from a leak at an industrial site in New Jersey.

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KEY PROJECTS (Continued)

- Planned, organized, and provided technical oversight for a supplemental RI/FS that was conducted at a solvent reclaiming facility (Superfund site) in the coastal plain of New Jersey. The study involved a detailed and critical evaluation of a complex multi-layered aquifer system that resulted in the identification of previously unidentified upgradient contributions to the contamination problem.

- Planned and managed a hydrogeologic investigation program for Amherst, Massachusetts that resulted in the development of an additional 1 million gallons per day ground-water supply and evaluated the impacts of this new diversion on existing wells and wetlands.

- Planned, organized, and provided technical oversight for an RI/FS that was conducted at the Islip municipal landfill on Long Island, New York.

- Planned, organized and currently providing technical oversight for an RI/FS being conducted at the Babylon municipal landfill on Long Island, New York.

- Carried out and evaluated data from a detailed field testing program, including several complex aquifer tests, to evaluate the feasibility of developing a 2,000 gallons per minute ground-water supply from a tract of land in the coastal plain along the Delaware River for a proposed gasification plant.

- Planned and managed a large-scale hydrogeologic field study, including an aquifer testing program to collect hydrogeologic data as part of the Suffolk County Flow Augmentation Needs Study (FANS). The collected data was input to a U.S. Geological Survey ground-water computer flow model of a portion of Suffolk County, Long Island, New York. The model predicted the impacts on the hydrologic system and on stream flow in particular from proposed sewering.

- Provided technical assistance to the USEPA concerning its investigation of a ground-water contamination problem in Iowa.

- Prepared a detailed description of the geology and hydrology of the New Jersey Pine Barrens region as part of the planning effort for the area.

- Planned, organized, and currently providing technical oversight for an RFI (RCRA Facilities Investigation) of a commercial hazardous waste treatment facility in New Jersey.

- Carried out and evaluated data from a detailed field testing program, including several complex aquifer tests, to quantify the effects of developing a 2.7 million gallons per day ground-water supply for a proposed refinery site in the Atlantic coastal plain.

- Developed and managed a test drilling program to determine the extent, severity, and source of ground water contaminated with volatile organic compounds at an industrial site in Connecticut.

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KEY PROJECTS (Continued)

- Planned, organized, and managed a study of ground-water contamination (PCBs and volatile organic compounds) resulting from an abandoned disposal area. Prepared conceptual design for slurry wall that was used to remedy problem at an industrial site in southern New Jersey.

- Designed and implemented a pumping well abatement system to contain and remedy a plume of ground water contaminated with organic compounds at a hazardous waste treatment site in the New Jersey coastal plain.

- Planned, organized, and managed a study that developed a detailed methodology for apportioning responsibility between two parties for investigation and clean-up costs related to ground-water contamination at a large military installation in the western United States.

- Designed and supervised the installation and testing of a high capacity production well for a development in the coastal plain near the Delaware River. Analyzed the data from the well testing and determined a safe yield for the well of 2 million gallons per day taking into account the potential effects on surrounding well users.

- Planned and organized a field program to develop hydrogeologic data that was used on behalf of a client to have his property removed from a State of New Jersey list of candidate sites for a toxic waste incinerator.

- Planned, organized, and managed a regional ground-water sampling program covering nine states to develop data that was used by a major pesticide manufacturer in re-registration procedures with the U.S. Environmental Protection Agency.

- Supervised the installation and testing of a high capacity production well (2 million gallons per day) for a northern New Jersey community.

- Planned and managed a large-scale field hydrogeologic study, including an aquifer testing program to gather data for input to a ground-water computer flow model of a segment of the North Fork of Long Island. The computer modeling predicted the effects of large-scale ground-water withdrawals on the local hydrogeologic environment including effects on salt water intrusion. As part of the study, a 2 1/2 million gallons per day test well was developed. The work was carried out for a proposed nuclear power plant project.

- Developed a comprehensive ground-water management plan for the Hop Brook drainage basin located in the towns of Amherst, Belchertown, Granby, and Pelham, Massachusetts. The plan gave recommendations, including land use planning, for maintaining and preserving the quantity and quality of the resource, outlined a detailed sampling and analysis plan for continual monitoring of the resource, assessed, through computer modeling, the vulnerability of the resource to contamination, evaluated the basin ground-water safe yield, and assessed prospects for future ground-water development.

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KEY PROJECTS (Continued)

- Provided an overview of waste disposal practices in the United States as part of a Canadian project to develop a regional waste management facility.

- Provided technical guidance to a national company related to compliance with the New Jersey Environmental Cleanup Responsibility Act (ECRA).

- Planned, organized, and managed a New York State Part 360 hydrogeologic investigation for a construction debris/clean fill landfill site in Melville, Long Island, New York as part of the permitting process.

- Designed and analyzed the testing of a 3 million gallons per day production well for the Town of Southington, Connecticut. The study included an analysis of the potential yield of the ground-water basin.

- Designed a 1 1/2 million gallons per day well for Amherst, Massachusetts as a back up supply.

- Planned, organized, and currently providing technical oversight on a ground-water contamination plume delineation/remediation project in Nassau County, Long Island, New York.

- Assisted in the design of remedial wells for the clean up of the Old Bethpage landfill, Long Island, New York ground-water contamination plume.

- Providing technical oversight to the Town of North Hempstead, Long Island, New York concerning their Port Washington Municipal Landfill.

TESTIMONY

- Appeared at an administrative law hearing and before the Water Policy and Supply Council of the New Jersey Department of Environmental Protection on behalf of the Historic Towne of Smithville Development Company. Testimony resulted in the granting of the full ground-water diversion requested in an environmentally sensitive area.

- Testified before a New Jersey Senate Sub-Committee on issues related to the New Jersey Water Supply Master Plan.

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SECTION 2

SECTION II

SAMPLING AND ANALYSIS PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

CONTENTS

SECTION II: SAMPLING AND ANALYSIS PLAN

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APPENDICES

A. Ground-Water Monitoring Well Specifications and Installation Protocols.

B. Ground-Water Sampling Protocols.

C. Ground Water-Level Measurement Protocols.

D. Gas Well Installation and Monitoring Protocols.

E. Field Instrumentation Operating Procedures.

F. Chain-of-Custody Procedures.

G. Borehole Geophysical Logging Protocols.

H. Geraghty & Miller, Inc. Quality Assurance/Quality Control Forms.

SECTION II

SAMPLING AND ANALYSIS PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

1.0 INTRODUCTION

The Sampling and Analysis Plan (SAP) addresses the field portion of the RI data collection activities. The purpose of the SAP is to provide a detailed description of the field program and how it will be organized, managed, and conducted. The methodologies for conducting each field task are described in the Standard Operating Protocols and Procedures (SOPs), which are given in the appendices to this SAP (see Section 4.0 [Standard Operating Procedures] of this SAP). The format and contents of the SAP have been prepared in accordance with the following USEPA guidance documents: "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (USEPA 1988a) and "Compendium of Superfund Field Operations Methods" (USEPA 1988b). The findings of the OU-1 (on-site) RI indicate that the on-site plume thickness, potential off-site extent of the leachate plume, and the potential off-site extent of landfill gas migration must be defined.

2.0 FIELD CONTROL PROCEDURES

2.1 ACCESS

Field activities are to be performed in and around residential areas near the landfill. Access to drilling sites will be controlled by the Geraghty & Miller field hydrogeologist who will oversee the completion of the soil borings and monitoring well installations. Individuals not involved in the RI and not authorized by Geraghty & Miller, LKB, the Town, or regulatory authorities will be denied access to the drilling and sampling sites. Individuals will be required to log in as described in the HASP (Section IV).

2.2 SECURITY

At the completion of each day of drilling, the drilling site will be surrounded by a temporary fence and a caution sign will be posted nearby to warn intruders not to enter the area. For the three off-site drilling locations, a security guard will also be stationed at the completion of each work day until the following morning and during the weekend. All partially completed boreholes will be secured to prevent the introduction of foreign matter into the borehole. Equipment used in monitoring well drilling and installation and for the collection of samples will be secured daily to discourage theft and prevent injury to intruders. After completion, each monitoring well will be capped and a protective locking well head assembly (curb box or protective steel casing) will be installed.

2.3 CLEARANCE

Prior to the start of field operations, Geraghty & Miller will establish the exact drilling locations in the field and ensure that all the necessary clearances are obtained. After site selection, it will be the responsibility of the drilling subcontractor to obtain any necessary state or local permits for monitoring well drilling. The Town will be responsible for

obtaining any work easements that are needed. Geraghty & Miller is responsible for securing a source of potable water and for contacting the appropriate authorities or agencies to locate buried utilities.

2.4 FIELD PROJECT COORDINATION

Responsibilities of field personnel are described in Section 8.0 (Project Organization) of this SAP. Field project coordination will be carried out by the project director, project manager, and field hydrogeologist in consultation with the Town and LKB, as necessary. The responsibilities of each are fully described in Section 2.0 (Project Organization and Responsibilities) of the QAPP (Section III).

3.0 MONITORING PARAMETERS AND FREQUENCY OF SAMPLE COLLECTION AND MONITORING

This section discusses the monitoring parameters and frequency of collection of ground-water samples during the Off-Site Ground-Water Study and frequency of air monitoring during the Off-Site Subsurface Gas Study.

3.1 OFF-SITE GROUND-WATER STUDY

Ground-water samples will be collected during the Off-Site Ground-Water Study as follows:

1. **Nassau County Monitoring Wells and Public Supply Wells.** One round of water samples will be collected from all Nassau County monitoring wells and public supply wells within a 2-mile radius of the site to determine background water quality. Samples will be analyzed in the field for hardness, alkalinity, and ammonia (primary leachate indicators) and for pH, temperature, chloride, and specific conductance (secondary leachate indicators). Twenty percent of the samples collected will be replicated and sent to the laboratory for confirmatory analysis. This information will be used in conjunction with any existing water quality data for the area (e.g. 2-mile radius from the site) to establish action levels of leachate indicator parameters. These action levels will then be used to determine when to terminate the drilling of the exploratory well borings (see Section 2.1.2 of the OU-2 RI work plan [Geraghty & Miller, Inc. 1991]).

2. **Exploratory Borings.** During the drilling of the on- and off-site exploratory borings to determine the vertical water quality profiles, water samples will be collected from each exploratory boring at 20-foot intervals from the water table to the termination depth (see Section 2.1.2 of the OU-2 RI work plan [Geraghty & Miller, Inc. 1991]).

Samples will be analyzed in the field for leachate indicator parameters (hardness, alkalinity, ammonia, pH, temperature, chloride, and specific conductance).

3. **Site Monitoring Wells (On-Site and Off-Site).** Two rounds of samples will be collected from the 15 existing on-site monitoring wells and the 11 proposed monitoring wells to determine on-site and off-site ground-water quality conditions. These samples will be collected at least 1 month apart following the well installation program. Samples will be analyzed for the parameters discussed in detail in the QAPP (Section III).

The monitoring parameters, designated locations, anticipated number, and frequency of sample collection for each of the purposes described above are summarized in Table 1.

3.2 OFF-SITE SUBSURFACE GAS STUDY

The three new gas monitoring and existing on-site gas monitoring wells in the southwestern portion of the site (G-7, G-8, and G-10) will be monitored on 3 days of low barometric pressure for methane and total volatile organic compounds (TVOCs) using an organic vapor analyzer (OVA).

In addition, on-site gas monitoring cluster wells CW-2, 4, 5, 6, 7, and 8 will be monitored for VOCs and methane as part of the OU-1 Remedial Design Program. The monitoring results from these wells will be presented and evaluated in the OU-2 RI report.

4.0 STANDARD OPERATING PROCEDURES

The SOPs for collecting the samples during the field program are described in detail in the appendices for this SAP. Equipment decontamination for each applicable procedure is also included within these appendices.

The SOPs for the RI field program (Off-Site Ground-Water Study and Off-Site Subsurface Gas Study) are included in the appendices indicated below:

SOP	Appendices in this SAP
Ground-Water Monitoring Well Specifications and Installation Protocol (includes formation sampling).	A
Ground-Water Sampling Protocols.	B
Ground-Water Level Measurement Protocols.	C
Gas Well Installation and Monitoring Protocols.	D
Field Instrumentation Operating Procedures.	E
Chain-of-Custody Procedures.	F
Borehole Geophysical Logging Protocols.	G

5.0 SAMPLE HANDLING AND CUSTODY

Samples will be handled in accordance with the chain-of-custody procedures (Appendix F of this SAP). These procedures include instructions for selecting sample containers, sample preservation, and sample security from the time of collection to arrival at the laboratory. Analytical suites for samples are listed in Table 1. The data usage and requirements, and analytical support levels for each type of sample, are described in detail in Section 3.3 (Quality Control Objectives) of the QAPP (Section III).

6.0 DATA VALIDATION PROCEDURES AND RESPONSIBILITIES

The data validation procedures consist of a systematic review of the analytical results and quality control (QC) documentation, and will be performed in accordance with the following two Region II data validation SOPs: (1) SOP: Evaluation of Metals Data for the Contract Laboratory Program (CLP), Revision 11, January 1992, and (2) SOP No. HW-6: CLP Organic Data Review and Preliminary Review, Revision 8, January 1992. Based on that review, the data validator will be responsible for making judgements and expressing concerns and comments on the quality and limitations of specific data, as well as on the validity of the overall data package. The data validator will prepare documentation of that review and provide conclusions using the standard USEPA Inorganic Regional Data Assessment and Organic Regional Data Assessment forms to summarize any overall deficiencies that require attention. The data validator will also evaluate general laboratory performance. These forms will be accompanied by appropriate supplementary documentation, clearly identifying specific problems.

The data validator will inform the project manager of data quality and limitations, and assist the project manager in interacting with the laboratory to correct data omissions and deficiencies. The laboratory may be required to rerun or resubmit data depending on the extent of the deficiencies and their importance in regard to the data quality objectives (DQOs). A detailed discussion of the data reduction and validation procedures is provided in Section 8.0 (Data Reduction, Validation, and Reporting) and Appendix A of the QAPP (Section III).

7.0 SCHEDULE

A schedule of tasks and deliverables, including field activities, is provided on Figure 2.

8.0 PROJECT ORGANIZATION

This project has been organized in a manner that ensures that the field program will be conducted effectively and efficiently. The following is a list of key personnel with their corresponding project titles.

Town of Oyster Bay - Site Owner	
Project Coordinator	Richard W. Lenz, P.E.
LKB - RI/FS Program Manager	
Program Director	John P. Lekstutis, P.E.
Program Manager	Theresa C. Heneveld, P.E.
Geraghty & Miller - RI Project Manager	
Project Director	Andrew J. Barber
Project Manager	Vincent J. Glasser
Field Hydrogeologist	Casey S. Schmidt
Project QA/QC Manager	Michael F. Wolfert
Project Health and Safety Coordinator	Tom C. Eng
Data Validator	Lauren E. Sjogren
RI Project Support Team	
Sampling Team	David Vines
	Brendan Mulholland
Support Functions	
Data Processing	Laurie Klecka
Data Processing QC	Gregory K. Shkuda, Ph.D.

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Subcontractors

Laboratory	Industrial & Environmental Analysts, Inc. (IEA)
Well Driller	To be bid
Waste Disposal Hauler	To be bid

A further description of the responsibilities of the RI personnel is provided in Section 2.0 (Project Organization and Responsibilities) of the QAPP (Section III).

Geraghty & Miller site personnel will consist of the field hydrogeologist. Site subcontractors will include a well driller, a waste disposal hauler, and a land surveyor. The responsibilities of each of these on-site individuals are as follows:

Site Personnel	Responsibilities
Field Hydrogeologist	- Site health and safety (site safety officer). - Coordinate all field and subcontracting activities. - Insure adherence to field QA/QC protocols. - Supervise monitoring well installation. - Collection of soil samples. - Collection of water-level measurements. - Collection of ground-water samples.
Well Driller	- Drilling and installation of monitoring wells under the supervision of the field hydrogeologist.
Waste Disposal Hauler	- Hauling and disposal of RI-generated waste materials.
Land Surveyor (LKB)	- Determine elevation of measuring points and horizontal location of monitoring wells.

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9.0 DOCUMENTATION

Project files containing data and reports generated during the RI including laboratory documentation will be maintained at Geraghty & Miller's Plainview, New York office according to the procedures outlined in this section. Laboratory documentation (e.g., chromatogram and results of spiked samples) will be maintained for purposes of validating analytical data collected during the RI. All summary reports will be kept in the project file in Plainview. All incoming data will be logged and dated. All information generated from field activities will be documented on appropriate log forms listed below.

- Sample/Core Log.
- Water Sampling Log.
- Gas Monitoring Log.
- Chain-of-Custody Record.
- Daily Log.
- Drilling and Installation of Monitoring Wells Daily Checklist.
- Utilities and Structures Checklist.
- Location Sketch.
- Water Level/Pumping Test Record.
- Materials/Cost Log.
- Sample Container Inventory.
- Well Construction Log.
- Sampling of Monitoring Wells Daily Checklist.
- Telephone Conversation Log.

9.1 SAMPLE DESIGNATIONS

This section provides information on the designations for use on the ground-water samples that will be collected for laboratory analysis. Each ground-water sample will be given a unique designation that will be recorded in the field log book on the water sampling

log form, on the label affixed to the sample container, and on the chain-of-custody record. The sample designation will consist of the following four elements:

1. The site code.
2. The matrix code.
3. The location number of the ground-water monitoring well.
4. The consecutive sample number collected from the location.

Incoming documents will be date-stamped and filed. If distribution is required, the appropriate number of copies will be made and distributed to project personnel. In addition, all notes from project meetings and telephone conversations will be filed along with other project documents.

9.2. DATA REDUCTION AND REPORTING

The reduction of field and analytical data will consist of compiling and summarizing field data (water-level measurements, soil boring logs, well logs, field parameters, gas monitoring logs, and laboratory analytical results). Reduction, validation, and reporting of laboratory data are discussed in Section 8.0 (Data, Reduction, Validation, and Reporting) of the QAPP (Section III). These data will be presented as tables, illustrations, and graphs as appropriate. The original data and reduced forms will be maintained at Geraghty & Miller's Plainview, New York office until final reports are accepted.

Chemical and physical data will be stored and managed using a data-management computer system. Data entry will be performed by a designated person (see Section 8.0 [Project Organization] of this SAP), so that access to the data base is limited.

The field measurement data will be similarly reduced into a tabulated format suitable for inclusion in the RI Report and will be designed to facilitate the comparison and evaluation of the data. These tabulations will include but not be limited to the following:

- Field screening (OVA) results.
- Field analyses (leachate-indicator parameters).
- Well construction details.
- Water-level measurements and surveyed measuring point elevations.

Field logs will be transferred into typed formats or will be presented in their original form for inclusion as RI Report appendices. The following logs will be used:

- Sample/Core Logs.
- Well Construction Logs.
- Water Level/Pumping Test Logs.
- Geophysical Logs.
- Water Sampling Logs.
- Gas Monitoring Logs.

The tables will be compiled whenever feasible by the field team member who collected the data. The data compiler will inform the project manager of any problems encountered during data collection and of apparent inconsistencies, and will also provide opinions on the data quality and limitations. The tables and logs will be used as the basis for data interpretation and will be checked against the original field documentation prior to use.

10.0 FIELD QA/QC AND CORRECTIVE ACTION

10.1 PERFORMANCE AND SYSTEMS AUDITS

The project QA/QC manager will audit all field aspects of the implementation of the RI on a regular basis. Any significant QA/QC deficiencies will be reported and identified, and they will be corrected to the extent practicable. Performance and systems audits relevant to the RI as a whole are discussed in detail in Section 10.0 (Performance and System Audits) of the QAPP (Section III). Reviews will also be performed at the completion of each field activity and will include an assessment of field data quality and the results of system and/or performance audits.

10.2 CORRECTIVE ACTION

If the quality control audit results in the identification of unacceptable conditions, the project QA/QC manager and field hydrogeologist will be responsible for developing and initiating corrective efforts, in consultation with the project manager and project director. The condition or problem will be specifically identified and investigated, and the cause will be determined. Efforts necessary to eliminate the problem will be initiated. Corrective efforts may include the following:

- Repeating field measurements.
- Reanalyzing the samples if the holding-time criteria and sample volume permits.
- Resampling and analyzing.
- Evaluating and amending sampling and analytical procedures.

Upon completion of the corrective effort, its effectiveness will be evaluated, and the elimination of the condition will be verified.

11.0 DELIVERABLES

The data generated during the RI will be provided to the USEPA in data validation reports and also in the RI report.

12.0 REFERENCES

Geraghty & Miller, Inc. 1989. Interim Remedial Investigation Report, Syosset Landfill, Syosset, New York. August 1989.

Geraghty & Miller Inc. 1991. Work Plan for the Second Operable Unit Remedial Investigation at the Syosset Landfill, Syosset, New York. April 1991.

U.S. Environmental Protection Agency (USEPA). 1981. Manual of Ground-Water Sampling Procedures, NWWA/USEPA.

U.S. Environmental Protection Agency (USEPA). 1982. Handbook for Sampling and Sample Preservation of Water and Wastewater.

U.S. Environmental Protection Agency (USEPA). 1988a. Guidance for Conducting Remedial Investigations and Feasibilities Under CERCL.. Office of Emergency and Remedial Response. USEPA, Washington, D.C.

U.S. Environmental Protection Agency (USEPA). 1988b. A Compendium of Superfund Field Operations Methods.

U.S. Environmental Protection Agency (USEPA). 1992a. CLP Organics Data Review and Preliminary Review. SOP No. HW-6, Revision 8.

U.S. Environmental Protection Agency (USEPA). 1992b. Evaluation of Metals Data for the Contract Laboratory Program (CLP), Revision 11.

APPENDIX A

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX A

GROUND-WATER MONITORING WELL SPECIFICATIONS

AND INSTALLATION PROTOCOLS

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX A

GROUND-WATER MONITORING WELL SPECIFICATIONS AND INSTALLATION PROTOCOLS

DRILLING METHODOLOGY

During the Off-Site Ground-Water Study, eleven monitoring wells will be installed at five locations; two of the proposed drilling locations are on-site where a single deep monitoring well will be installed next to existing well clusters (SY-3 and SY-6) (Figure 1). One on-site location is upgradient of the landfill where a deeper well will be installed at existing upgradient Well Cluster SY-6 to provide additional upgradient water-quality data. The second on-site location is downgradient of the landfill where a deeper well will be installed at an existing Well Cluster SY-3 to determine the plume thickness on-site. The three proposed off-site drilling locations will each have three wells per location. An exploratory boring will be drilled using a Barber rig (cased boring method) to install the proposed deep well on-site next to existing Well Cluster SY-3 and the deep well at the middle location of the three proposed off-site well cluster locations. Three drilling methods will be employed during the Off-Site Ground-Water Study: (1) the cased boring method (air rotary), (2) the modified mud rotary method (which includes the reverse rotary method), and (3) the hollow-stem auger method. The cased boring method (air rotary) will be used for drilling the two exploratory borings (one on-site and one off-site) and installing a deep well at these locations, while the mud rotary method (modified) will be used to drill and install the remaining wells except the shallow wells, which will be installed by the hollow-stem auger method. The reasons for the selection of these drilling methods are outlined in Sections 2.1.3.1, 2.1.3.2, and 2.1.3.3 of the OU-2 RI work plan (Geraghty & Miller, Inc. 1991), which was approved by the USEPA on July 5, 1991. The proposed monitoring well locations are shown on Figure 1.

A summary of the well data for wells proposed to be installed during the Second Operable Unit RI is given below.

Drilling Method	Number of Wells	Estimated Depth Per Well (Feet)
Cased Boring	2	600
Mud Rotary (Modified)	3	600
Mud Rotary (Modified)	3	400
Hollow Stem Auger	3	185
Total	11	4,755

The field hydrogeologist will be present at the operating drill rig and will be responsible for the collection and logging of soil samples, monitoring of drilling decontamination operations, recording of ground-water data, deciding on final drilling depths and screen intervals (in consultation with the USEPA, the Town and LKB), preparing the boring logs and well completion diagrams, and recording the well installation procedures. Additionally, the field hydrogeologist will maintain a log recording daily events, including arrivals and departures at the site by any and all personnel and equipment, and times of work start-up and stoppage. Forms to be used by the hydrogeologist as part of the Quality Assurance/Quality Control (QA/QC) measures of the project are included in Appendix H of this SAP.

Drilling fluids, soil cuttings, purge water, development water, and decontamination water will be disposed on landfill property. A recharge area will be designated on-site over native soil for purge water, development water, and decontamination water. ~~Bemas~~ Berms hay bales, or other means will be used to prevent runoff of the water. This recharge area will be located a sufficient distance from existing monitoring wells to prevent potential impacts to these wells. A separate area on the landfill surface will be designated for drilling fluids and soil cuttings.

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If any problems are encountered or questions arise during the field investigation that are beyond the scope of responsibility of the field hydrogeologist, then field activities will be temporarily halted and the project manager and/or the project director will be consulted. In this way, a technically sound decision can be made by more experienced individuals.

CASED BORING METHOD

The cased boring method (air rotary) will be employed using a Barber rig, which advances the boring by hydraulically rotating the steel casing into the ground while simultaneously drilling with a roller bit. Cuttings are continuously removed from the casing by forcing compressed air through the drilling rods. An oil filter will be installed in the air line that will be placed in the well being drilled. Small volumes of water are used for controlling dust while drilling through the unsaturated zone. The collection of formation samples and ground-water samples for analysis in the field is discussed later in this appendix.

MODIFIED MUD ROTARY METHOD

The modified mud rotary method consists of drilling each boring to within 20 feet of the screen zone by employing the conventional mud rotary method (described below). The screen zones will have been already determined based on the lithological profile at each drilling location and on the vertical water quality profiles established during the drilling of the two exploratory borings (see Appendix B of this SAP). The mud rotary method uses a roller bit that loosens formation material (cuttings) for removal. The cuttings are removed by a drilling fluid composed of potable water mixed with 100 percent bentonite clay that contains no additives. The source of potable water will be from the water district fire hydrants located near the site. The viscosity of the drilling fluid (mud) can be varied in response to formation changes. The mud rotary method is a direct rotary drilling method whereby drilling fluid is pumped down the drill pipe and forced up through the annular space between the drill pipe and borehole wall to the land surface. To prevent the loss of

drilling fluids in the unsaturated zone (upper 100 feet), 10-inch diameter steel casing will be installed by the cased boring method before drilling begins using the mud rotary method. The final 30 feet of each boring will be drilled by the reverse rotary method (described below). The same drilling rig for both drilling methods (or a conventional reverse rotary drilling rig) may be used to complete each boring. After drilling the portion of the boring using the mud rotary method, the swivel on the drilling head will be changed. Because the final 30 feet of each boring will use potable water as the drilling fluid (see below), a residual mud cake will not form on the borehole wall in this interval where the screen will be set. The mud will then be flushed out of the boring by pumping fresh potable water through the drilling rods to force the mud out of the boring annulus. The drilling mud will be pumped into a tanker truck and will be disposed at a designated location at the landfill. As soon as the mud is removed, the reverse rotary method will be employed to complete the boring. As discussed previously, the source of the potable water used for flushing the drilling mud from the well boring and for the reverse rotary drilling method will be from the water district fire hydrants located near the site. Before the reverse rotary drilling method begins, a 1-inch diameter air line will be inserted inside the 3-inch diameter drilling rods. Compressed air pumped through the air line will force the water (drilling fluid) out of the drilling rods and induce water from the well annulus to flow back up into the drilling rods, constantly replacing the water removed through the drilling rods and out of the boring. Although a residual mud cake will remain on the borehole wall in the depth interval drilled using mud, there will still be some fluid or water loss to the aquifer. Thus, it will be necessary to add potable water to the well annulus to supplement the water being recirculated in the boring.

HOLLOW-STEM AUGER METHOD

The shallow monitoring wells will be installed using the hollow-stem auger method, provided that the well depths do not exceed the method's depth limits (about 165 feet $\pm$ 20 feet). This method involves rotating a string of auger flights into the subsurface. Soil cuttings are continuously lifted to land surface along the outside of the flights, while drill

rods and a plug are used inside the flights to keep the soil out. The plug and rods are removed for well installation inside the flights.

Potable water may be used during or after drilling (if necessary) to keep loose sand from heaving inside the auger flights (due to differential hydrostatic pressure) because heaving would prevent installation of well screen and casing. If water is added, the volume used will be carefully documented so that at a minimum, an equivalent volume can be removed during well development.

SAMPLING

GROUND-WATER SAMPLING FOR ANALYSIS IN THE FIELD

During the drilling of the on-site and off-site exploratory borings by the cased boring method, ground-water samples will be collected through the drilling rods with a bailer at 20-foot intervals until the boring is terminated. Samples will be analyzed for leachate indicator parameters. After each 20-foot sampling depth is reached, and before a water sample is collected for in-field analysis, water will be pumped from the boring using the air compressor on the drilling rig. Thus, a representative ground-water sample from each sampling depth will be available for collection. The bailer used to collect the samples will be lowered using a winch-operated cable from the drilling rig. These samples will be analyzed in the field for leachate indicator parameters to determine the vertical water-quality profiles at these two locations (see Appendix B of this SAP).

FORMATION SAMPLING

Formation samples will be collected from the deep boring at each of the five drilling sites. Composite formation samples will be collected from the exploratory borings on a continuous basis from the cyclone discharge, which is a device that reduces the pressure of the cuttings travelling up the boring to a gravity feed and permits sample collection at

ground surface. Split-spoon samplers will be used to collect formation samples at 20-foot intervals from the deep borings at the three remaining drilling sites by the modified mud-rotary method. To collect a sample, the split-spoon sampler will be sent down the inside of the drilling rods by a wire line. Samples are collected by advancing the split spoon ahead of the drill bit into the undisturbed formation at the desired depth. Before and after each use, the split spoons will be decontaminated by scrubbing with Micro solution and rinsing with analyte-free deionized water.

Data collected during formation sampling will be used to supplement the geophysical logs that will be conducted in each of the five deep well borings. The geophysical logs will provide an excellent continuous lithologic profile. The geophysical log in conjunction with the water-quality data from the two exploratory borings will provide an accurate depiction of the vertical subsurface conditions for selecting monitoring well screen intervals.

At a minimum, the following information will be recorded by the field hydrogeologist:

- Soil sample interval.
- Sample color.
- Sample texture.
- Sample moisture content.
- Organic vapor readings.
- Unusual characteristics.
- Depth to water.
- Drill rig behavior and penetration rate.

A sample/core log form is included in Appendix H of this SAP. Representative soil samples from each sampling interval will be placed in glass jars with screw-type lids. Each sample container will be labeled with the site name and the boring and sample number. These jars will be stored in cardboard boxes for future reference.

MONITORING WELL INSTALLATION

The installation of each monitoring well will begin immediately after borehole completion, or as directed by the field hydrogeologist. Once monitoring well installation has begun, breaks will not be taken until the well has been completed and secured against unauthorized access. In cases of unscheduled delays, such as personal injury, equipment breakdowns, or sudden inclement weather, installation will be resumed as soon as practical.

The monitoring wells to be installed in the cased borings, modified mud rotary borings and hollow-stem auger borings will be constructed of the same materials and follow the same protocols as the wells installed during the On-Site Ground-Water Study. The only exception is that stainless-steel screens will be used instead of PVC screens to facilitate well development. The stainless-steel screens will be 10-feet long and will have openings of 0.010 inches (10 slot).

MONITORING WELLS INSTALLED IN CASED BORINGS

The construction details of the monitoring wells to be installed in the cased borings are depicted on Figure 3. Temporary 14-inch diameter surface casing will be installed to the water table (approximately 100 feet below land surface) to reduce the surface friction on the 10-inch diameter casing, which will be installed to a depth where excessive resistance is encountered. At this point, an 8-inch diameter casing will be used to complete the boring. After the 8-inch diameter casing has been advanced to its full depth, the 4-inch diameter stainless steel screen and PVC casing will be set inside the 8-inch casing and the gravel pack and grout seal will be emplaced as the 8-inch diameter casing is pulled back to the water table and left for final well construction. The gravel pack will extend from the bottom of the borehole to at least 3 feet above the top of the screen and will be emplaced by pouring it slowly by hand in the annular space between the borehole wall and the well casing. The gravel pack will consist of J. Morie Co. No. 1 clean silica graded sand. The grout seal (Volclay) will be emplaced using a side port tremie pipe above the gravel pack and will extend to 2 feet below land surface. Volclay grout is a high density, 100 percent polymer-

free bentonite grout that will form a continuous and flexible seal that will not crack. After the grout seal is emplaced, the 10-inch diameter casing will be removed. The annulus between the 8-inch and 14-inch diameter casings will then be grouted to ground surface as the 14-inch diameter casing is being removed. The 14-inch casing will be left in the ground if there is an excessive loss of grout to the formation as the casing is being removed.

If the 8-inch diameter casing becomes too difficult to advance before the termination criteria are met (see Section 2.1.2 of the OU-2 RI work plan [Geraghty & Miller, Inc. 1991), 6-inch diameter casing will be used to complete the boring to the termination depth. The grout will then be pumped through a tremie pipe from the bottom of the borehole to between 5 and 10 feet of the bottom of the selected screen setting for the intermediate depth well. The 6-inch diameter casing will be pulled back during the grouting process and well construction will continue in the prescribed fashion (see discussion above). Because the vertical profile of the water quality and lithology would have already been obtained from the exploratory boring where the intermediate well would be installed, the deep monitoring well will be drilled and installed by the mud rotary method.

MONITORING WELLS INSTALLED IN THE MODIFIED MUD ROTARY BORINGS

The construction details of the monitoring wells to be installed in the modified mud rotary borings are depicted on Figure 4. As previously mentioned, the upper permeable deposits will be cased off (10-inch diameter steel casing) to a depth of approximately 100 feet below land surface to prevent the loss of drilling fluid. The boring will be continued at a diameter of 8 inches until the completion depth has been reached (the final 30 feet of drilling to be accomplished by the reverse rotary method), whereupon the well construction will proceed as described previously.

MONITORING WELLS INSTALLED IN THE HOLLOW-STEM AUGER BORINGS

The shallow monitoring wells will be installed in the approximately 11-inch diameter boreholes according to the same procedure described above. The construction details of the monitoring wells to be installed in the hollow-stem auger borings are depicted on Figure 5.

Well Construction

Monitoring wells will be constructed of new 4-inch diameter schedule 40 PVC casings and 4-inch diameter 10-foot lengths of stainless-steel screen (10 slot). The well screens will be fabricated and have an inside diameter equal to the well casing. Fittings (couplings) will not restrict the inside well diameter as joints will be internally threaded. Glues, solvents, or chemical cleaners will not be used to join the casing and screen lengths. The lengths of casing and screen will be measured and recorded by the field hydrogeologist.

A monitoring well construction diagram will be prepared by the field hydrogeologist for each well and will show the following information:

1. The total drilled depth of the borehole.
2. The borehole and well casing diameters.
3. The casing and screen composition.
4. The screen setting.
5. The gravel pack interval.
6. The grout seal interval.
7. The height of the well casing (without cap/plug) above ground surface.
8. The protective casing.

Each well construction diagram will be included as part of the QA/QC procedures. An example of a well construction diagram is provided in Appendix H of this SAP.

Monitoring well depths and screen zones will be determined based on the water-quality profile established during the drilling of the two exploratory borings and the lithologic profiles determined from formation sampling and geophysical logging (see Appendix G of this SAP).

Gravel Pack

As mentioned previously, gravel pack consisting of clean silica sand (J. Morie Co. No. 1) will be emplaced by pouring it slowly by hand in the annular space between the borehole wall and the well casing. During emplacement of the gravel pack, a weighted steel tape will be placed down the annulus periodically to measure its depth to ensure that the gravel is not bridging and that it extends from the bottom of the borehole to at least 3 feet above the top of the screen. The gravel pack material will be examined to confirm its identification.

Grout Seal

A grout seal consisting of Volclay grout will be emplaced using a side-port tremie pipe above the gravel pack and will extend to 2 feet below land surface. Volclay grout is a high-density, 100 percent polymer-free bentonite grout that sets up like putty and will form a continuous and flexible seal that will not crack.

Well Completion

The wells will be completed with an aboveground, locking steel protective casing or a locking flush-mounted curb box that will be set over each well head to protect against vandalism. The number for each well will be clearly marked on the protective casing or curb box.

The top of the well casing will be surveyed by a professional surveyor, licensed in New York State to the nearest 0.1 foot and will serve as the measuring point for ground-

water level measurements. The measuring point will be clearly indicated on the well casing for easy identification.

MONITORING WELL DEVELOPMENT

Well development will be accomplished using compressed air, a submersible pump, and/or surge block or any combination of these techniques. If compressed air is used to develop a well, then an oil filter will be installed in the air line placed in the well being developed. Well development will not be attempted until at least 24 hours after installation. Development will continue until the well responds to water-level changes in the formation, and the well produces clear, sediment-free water to the extent possible. The wells will be developed with a goal of producing water of a turbidity of 50 nephelometric units (NTUs) or less. However, in some instances, water may have noticeably high-turbidity levels due to a relatively high percentage of finer grained deposits indigenous to the Magothy Formation. A portable nephelometer will be used in the field to measure turbidity levels. Development water will be disposed at a designated location at the landfill.

Dispersing agents, acids, disinfectants, or other additives will not be used during development nor will they be introduced into the well at any other time. During development, water will be removed from the entire column of water standing in the well by periodically lowering and raising the pump intake.

A record of well development will be maintained along with the other pertinent well data as part of the QA/QC procedures. The following data will be recorded as part of development and QA/QC procedures:

1. The static water level measured from the top of the well casing before, during and after, development is completed.

2. The calculated quantity of fluid standing in the well prior to development.

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3. The sounded depth of the well before and after development to determine if silt has accumulated inside the well.

4. The physical character of water removed including changes during development such as water clarity, color, particulates, and odor.

5. The type and size/capacity of pump used.

6. The surging technique(s) used.

7. The quantity of fluid/water removed and the time for removal (both incremental and total values).

DECONTAMINATION

The drilling casings, rods, split-spoon samplers, tools, water tanks, drilling rig, and any piece of equipment that can come in contact (directly or indirectly) with the formation will be steam cleaned prior to being set up for drilling. The same steam-cleaning protocol will be followed between boreholes (at a fixed site location) and before leaving the site at the end of the project. Decontamination water will be disposed at a designated location at the landfill. The steam-cleaning activities will be monitored by the field hydrogeologist. Casings and screens will be purchased from a manufacturer (Johnson or an approved equal) that steam cleans the casings and screens, and individually wraps them (i.e. they are hermetically sealed).

APPENDIX B

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX B

GROUND-WATER SAMPLING PROTOCOLS

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SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX B

GROUND-WATER SAMPLING PROTOCOLS

INTRODUCTION

The collection, handling, and analysis of ground-water samples will be performed according to the protocols set forth in this appendix to ensure quality assurance/quality control (QA/QC). Quality control includes proper sampling procedures for well purging prior to sampling, sample-removal methods that utilize acceptable materials for all equipment and supplies, sample processing (including filtration, preservation, labeling, and bottle filling), and sample shipment. The Geraghty & Miller project manager and field hydrogeologist will work closely with the analytical laboratory to assure that samples reach the laboratory in proper condition. Water-quality data will be scrutinized according to Geraghty & Miller's QA/QC protocols for data validation as discussed in the Quality Assurance Project Plan (QAPP in Section III).

This sampling protocol is based on technically sound standard practices as published in "Manual of Ground-Water Sampling Procedures" (NWWA/USEPA 1981) and "Handbook for Sampling and Sample Preservation of Water and Wastewater" (USEPA 1982) as well as on the firm's experience.

Pertinent information regarding ground-water sampling procedures will be recorded on the Geraghty & Miller daily log forms and water sampling log forms (see Appendix H of this SAP).

PREPARATION FOR SAMPLING

A written sampling fact sheet containing the information needed by the field team will be prepared by the project manager. The fact sheet will be reviewed and verified by a Geraghty & Miller chemist and will include the protocol to be followed. Information on

the types and nature of chemicals that may be encountered during sampling will be provided along with a list of special precautions that may be needed for the collection, handling, storage, and transportation of the samples. Wells will not be sampled until a minimum of 7 to 14 days have elapsed after well development.

PREPARATION OF SAMPLING EQUIPMENT

The sampling equipment (e.g., bailers, submersible pumps, M-scopes, buckets, filtration equipment for metals) will be thoroughly cleaned before each use following the protocols described on Page II-B-13. Any supplies, such as tubing, that cannot be properly cleaned after each use will be discarded in an appropriate manner. Specific conductance and pH meters will be calibrated according to manufacturer's instructions (see Appendix E of this SAP).

SAMPLING EQUIPMENT

The equipment and materials that will be needed for the collection of ground-water samples are listed below:

Stainless-steel/Teflon Submersible Pumps	Prefilters (cellulose-based membrane)
Electric water-level probe (M-Scope)	Filters (0.45-um, and 5-um cellulose-based membrane
Clean rags	Filter funnels (Gelman polyethylene or glass)
Analyte-free deionized water	Vacuum flasks (1-liter polyethylene or glass)
Plastic sheeting	Vacuum pump (electric or hand operated)
Polypropylene rope	Thermometers
Bailers (Teflon)	Sample bottles
Bailer cord leaders (15-foot lengths of Teflon-covered wire)	Indelible marking pens

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Buckets (graduated)	Brushes
Gloves (Latex, Nitrile, or equivalent)	Measuring tape
pH meter and buffers	3/4-inch Polyethylene tubing
Specific conductance meter and standard	Clear tape
Beakers	

PREPARATION OF WELL FOR SAMPLING

OPENING THE WELL

Upon arrival at the well site, sampling personnel will record the well designations, inspect the well head for damage, wipe the top of the well clean, and then remove the cap and wipe the top of the well casing with clean paper towels. Plastic sheeting will be placed around the well so sampling equipment will be protected from potential contamination on the ground surface.

SOUNDING THE WELL

The total depth of each well will be measured (sounded) to an accuracy about 0.1 foot using a weighted steel or plastic tape prior to sampling. This information together with the depth to water allows the sampling team to calculate the volume of water in the well and to determine if formation material has accumulated at the bottom of the well.

MEASURING THE HEIGHT OF THE MEASURING POINT

The height of the measuring point above or below ground surface will be measured to an accuracy of 0.01 feet as an indication of whether the well may have been disturbed since installation.

MEASURING THE WATER LEVEL

A full round of water levels will be collected prior to sampling the first well. The date and time of each measurement will be recorded. Each measurement will be made to an accuracy of 0.01 feet. Care will be taken to avoid cross contamination of wells by thoroughly cleaning the measuring instrument (M-scope or measuring tape) between wells following the protocols described on Page II-B-15.

The procedure for accessing the well and for measuring the water level is summarized below:

1. Record the well number.

2. Wipe the top of the well casing with a clean paper towel.

3. Remove the well cap and/or plug and wipe the inside of the casing with a clean paper towel. The cap will be placed face up on the plastic sheeting to keep it clean. Personnel will wait approximately 10 minutes prior to measuring the water level to allow the water level in the well to stabilize under atmospheric pressure.

4. Analyze the air within the well casing for volatile organic compounds (VOCs) using an OVA.

5. Decontaminate the first 5 feet of the electric probe or measuring tape and then measure the depth to water from the top of the well casing.

6. Calculate the volume of water in the well.

PURGING THE WELL

Well evacuation is performed to ensure that all standing water is removed from a well before sample collection so that the sample will be representative of the ground water in the vicinity of the screen zone. Before evacuating, the volume of standing water in each well will be calculated by subtracting the depth to water from the sounded depth of the well; this number, the height of the water column in the well, will then be multiplied by a coefficient which relates the diameter of the well to gallons per linear foot to obtain the volume of standing water in the well. The monitoring wells will be evacuated either by bailing or by using a submersible pump based on the diameter of the well being sampled. Purge water will be disposed at a designated location at the landfill. A summary of construction details for the existing on-site monitoring wells is provided in Table 2. Five of the existing on-site wells are constructed of 4-inch diameter casing and each has a permanently installed submersible pump for evacuation and sampling. The remaining ten existing on-site wells are constructed of casing that is less than 4-inch diameter and these must be evacuated by bailing or using a small diameter submersible pump. All the new proposed monitoring wells will be constructed of 4-inch diameter casing and will have permanently installed submersible pumps that will be used for evacuation and sampling. Three well volumes will be evacuated (either by pumping or by bailing) from each well prior to sample collection. Temperature, Ph, and specific conductance will be measured prior to purging and twice before sampling. Unless there is an unusual difference in the readings between the final two successful measurements of these parameters, samples will be collected following the removal of three well volumes.

COLLECTION OF GROUND-WATER SAMPLES

The order of sample collection will be based on the water-quality data base from the OU-1 RI and from the exploratory borings. The cleanest wells will be sampled first followed progressively by more contaminated wells. After the well has been evacuated, a ground-

water sample will be collected within 24 hours and will be placed on ice immediately after collection. Samples to be analyzed for VOCs will be collected first using a bailer after purging three volumes from the well using a submersible pump. The remaining parameters will be collected from the pump discharge in order of decreasing volatility (semi-volatiles, PCBs, metals, cyanide, and leachate indicators). New disposable gloves will be worn by sampling personnel for each well sampled. The sample containers will be inspected to ensure that they are the correct type and number for the respective analytical parameters and have the correct preservative, if required. The labels will then be properly filled out and affixed to the containers and protected by clear tape affixed to the containers. Samples will be carefully poured into each container, avoiding agitation or turbulence, which might result in loss of volatile organics and/or excessive oxygenation of the samples. Care will also be exercised to avoid breakage and to eliminate the entry or contact of, any substance with the interior surface of the bottles, vials, or caps, other than the water sample being collected. Caps will not be removed until sampling begins and then they will be replaced as soon as the container has been filled. The sample containers will be kept cool, dust-free, and out of the sun. The procedures that the sampling team will follow to collect water samples are described below in the order in which they will be performed:

1. Complete labels on all containers and protect labels by wrapping them to each container with clear tape. Information that will be provided on labels includes the following: project name, well numbers, sampling date, etc.

2. Acidify the volatile organic samples to a pH of less than 2 with 1:1 HCl. Determine the amount of acid needed by adding acid drop by drop on a separate 40 milliliter (mL) aliquot. This aliquot will be discarded after use and may be collected at the end of the purge and prior to sampling. The pH will be confirmed with pH paper. After the number of drops have been determined, that amount of acid will be added to the sample vials. Fill the 40 mL vials for

volatile organic analysis first in such a manner as to ensure that there are no air bubbles.

3. Analyze for field parameters (see Appendix E of this SAP for Field Instrumentation Operating Procedures).

4. Fill the remaining sample containers in the order of the parameter's volatilization sensitivity. All parameters requiring chemical preservation will be preserved immediately. The pH will be confirmed by pouring a small amount of the sample on pH paper. The pH paper will not be immersed in the sample bottle. Unfiltered and filtered samples will both be collected for metals analysis. All samples will be placed on ice immediately after collection.

5. Replace the well cap and lock the well.

6. Pack the samples on ice in a cooler with the completed Geraghty & Miller chain-of-custody record form. Samples will be delivered or shipped to the laboratory within 24 hours after sample collection and the recipient's signature will be obtained on the chain-of-custody record form.

7. Discard the disposable sampling equipment such as used cord, gloves, and plastic sheeting.

The samples for metals analysis will be filtered through a 0.45-micrometer (um) filter. Prefilters or a 5.0-um filter will only be used if the sample cannot be filtered through the 0.45-um filter due to the presence of excessive suspended particulate matter. After filtration, the sample will be transferred to the pre-acidified container for metals analysis, and pH paper will be used to ensure that the sample has been acidified to less than pH 2. Unfiltered samples will also be collected for metals analysis.

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FIELD TESTING FOR LEACHATE INDICATORS

Primary and secondary landfill leachate-indicator parameters will be analyzed in the field according to the procedures described in this appendix. Ammonia, alkalinity, and hardness are the primary leachate indicators, and chloride, specific conductance, temperature, and pH are the secondary leachate indicators (see Section 2.1.8 of the OU-2 RI work plan [Geraghty & Miller, Inc. 1991]). These analyses will be conducted on groundwater samples collected from 20-foot intervals during the drilling of the two exploratory borings and from the Nassau County monitoring wells and public supply wells located within a 2-mile radius of the landfill. Twenty percent of the samples will be sent to the laboratory for confirmatory analysis of ammonia, alkalinity, chloride, and hardness. The analytical results of the field testing (in conjunction with the lithologic profiles) will be used to establish the action levels for determining the termination depths of the two exploratory borings and also for determining the screen zones for all of the monitoring wells at the five drilling locations.

Control samples will be prepared and analyzed on a daily basis, and field meters will be recalibrated, as necessary. Prior to analysis, water samples from each 20-foot sampling interval will be decanted from a bailer and composited into a clean 10-gallon plastic bucket that will be used to distribute the sample for analyzing the field parameters. Immediately following sample collection a total-immersion type thermometer will be placed into the bucket to record the temperature of the water sample. An aliquot of the sample will then be poured into a plastic beaker for each field parameter and covered in order to avoid excessive agitation or prolonged exposure to air. Because the analytical methods for the field analyses are sensitive to turbidity and because the samples will be collected from an open borehole and are, therefore, expected to be turbid, samples from each 20-foot interval will be prefiltered using Whatman 2V filter membranes. These filters are designed to retain particles of 8 um or larger. The procedure for the analysis of each of the field parameters is described below.

AMMONIA

The ammonia concentration in the water samples will be determined using an ammonia-ion-specific electrode (Corning Model No. 476130) and meter (Corning Model No. 620). A 100-mL sample of water will be placed into an Erlenmeyer flask, set on top of a magnetic stirrer, and a Teflon-coated stirring bar will be placed in the flask. The ISE will be immersed into the water sample. Once the meter reading stabilized, a 1 mL solution of 10 normal sodium hydroxide (NaOH) is added to the sample to convert ammonium ion to ammonia [$NH_4OH \rightarrow NH_3$] by raising the pH to above 11. The ammonia contained in the sample diffuses through the electrode's membrane and changes the pH of its internal solution. The pH change is measured by the electrode and converted to an ammonia concentration by Standard Method 417 (APHA, AWWA, and WPCF 1985).

ALKALINITY

The alkalinity determinations will performed by using a digital titration kit, supplied by the Hach Company (Hach). Sample volumes are selected according to the Hach manual. The sample volume will be placed in a 250-mL Erlenmeyer flask. When the sample concentration is above the instrument calibration range, the sample is diluted with deionized water. The titration end point is monitored with a pH meter, as well as a bromeresolmethyl red indicator. The endpoint of the titration is reached when the sample pH reaches 4.5 and turns a light pink color.

HARDNESS

The hardness determinations will performed by using a digital titration kit, supplied by Hach. Sample volumes will be selected according to the Hach manual. The sample will be placed in a 250-mL Erlenmeyer flask. When the sample concentration is above the instrument calibration range, it is diluted with deionized water. Eriochrome Black T (ManVer Hardness Indicator Powder Pillow) is then added to the sample, turning it red.

The sample is titrated with ethylenediaminetetracetic (EDTA) until a deep blue color is achieved, indicating the end point of the titration.

CHLORIDE

The chloride determinations will be performed by using a digital titration kit, supplied by Hach. Sample volumes are selected according to the Hach instruction manual. The sample will be placed in a 250-mL Erlenmeyer flask. When the sample concentration is above the instrument calibration range, the sample is diluted with deionized water. Diphenyl carbazone powder pillow is then added to the sample, turning it a pale yellow. The sample is titrated with mercuric nitrate [$Hg(NO_3)_2$] until a pink color is achieved indicating the end point of the titration.

TEMPERATURE

Temperature will be measured with a rapidly equilibrating, mercury-filled Celsius thermometer.

SPECIFIC CONDUCTANCE

Specific conductance of the sample will be measured using a Cole-Parmer field conductivity meter (Model No. 9999-99). The conductivity meter will be calibrated daily with known standard concentrations before field measurements are taken. The conductivity probe is rinsed with deionized water before and after each measurement (see Appendix E of this SAP).

pH

The pH of the sample will be measured using a Cole-Parmer pH meter (Model No. 5996-80). The pH meter will be calibrated daily with two standard buffer solutions before any field measurements are taken (see Appendix E of this SAP). The pH meter will be recalibrated frequently during the day to ensure accurate and precise measurements. The pH probe will be is rinsed with deionized water before and after each measurement.

QUALITY CONTROL

QC samples will be used to monitor sampling and laboratory performance. The types of QC samples that will be included in this investigation are replicates and blanks. To ensure unbiased handling and analysis by the laboratory, the identity of replicates will be disguised by means of coding so that the laboratory does not know which samples are used for this purpose. Detailed QC procedures are outlined in the QAPP (Section III).

REPLICATE ANALYSES

Replicate samples are samples collected from the same well and are identical within the limits of normal concentration fluctuations. Collection and analysis of such samples allow a check to be made on laboratory reproducibility. Five percent of all ground-water samples collected at this site will be replicated.

When collecting replicate samples for VOC analysis, each of the two sample vials for the sample and replicate will be alternately filled. For other analytes, the discharge water will be distributed to fill portions of each sample container until the containers are filled. Sampling for replicates is discussed in more detail in the QAPP.

BLANKS

The analysis of trip blanks will be incorporated into this field investigation. Since the trip blank cannot be more than 24 hours older than the associated field samples, trip blanks will be shipped daily to the field from the laboratory. A trip blank is a sample composed of analyte-free water acidified to a pH of less than 2 with 1:1 HCl (prepared by the laboratory), which is filled at the laboratory and travels unopened with the sample bottles back to the laboratory. It is analyzed to determine whether samples may have been contaminated by VOCs as a result of handling in the field, during shipment, or in the laboratory. One trip blank will accompany each day's shipment of water samples to the laboratory for VOC analysis. A field blank for all analytes, except leachate parameters, will also be prepared using analyte-free deionized water provided by the laboratory during each day of sampling to determine whether samples may have been contaminated by air-borne contaminants or the sampling equipment. Field blanks will be collected for dissolved metals by rinsing the clean filtering apparatus. Field blanks will be preserved following the same procedure used for the samples. Blank analyses are discussed in more detail in the QAPP (Section III).

RECORD KEEPING

All Geraghty & Miller personnel involved in sample collection will carefully document the handling history of all ground-water samples and blanks collected. Standard Geraghty & Miller forms (see Appendix H in this SAP) will be completed for this purpose as described below.

DAILY LOG

Daily logs will be used by the field team for QA/QC purposes to record all sampling events and field observations. Entries in the daily log forms will be dated by the person

making the entry, and the logs will be kept in a secure, dry place. The following information will be included on each daily log form:

1. Project name.
2. Date and time of arrival at the site.
3. Client.
4. Location.
5. Weather.
6. Sampling team members.
7. Work progress.
8. QC samples.
9. Departure time.
10. Delays.
11. Unusual situations.
12. Well damage.
13. Departure from established QA/QC field procedures.
14. Instrument problems.
15. Accidents.

WATER SAMPLING LOG

The sampling team will complete a Geraghty & Miller water sampling log form for QA/QC purposes (see Appendix H of this SAP) at the time of sampling to record information about each sample collected. The following information will be included on each Water Sampling Log form:

1. Date and time of sampling.
2. Well evacuation data.
3. Physical appearance of samples (e.g., color and turbidity).

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4. Field observations.
5. Results of field analyses.
6. Sampling method and material.
7. Constituents sampled for.
8. Sample container size, composition, and color.
9. Preservative.
10. Sampling personnel.
11. Weather conditions.

SAMPLE LABELS AND CHAIN-OF-CUSTODY RECORD FORM

Sample labels are necessary for proper sample identification. The labels will be affixed to the sample containers prior to the time of sampling: Labels will not be affixed to container lids or caps. To track QA/QC handling protocols the labels will be filled out by sampling personnel, and the Geraghty & Miller chain-of-custody record form (see Appendix H of this SAP) will be completed in the field before the sampling team leaves the site. Labels will include sample identification, project number, date and time collected, analyses to be performed, and pH adjustment information as required.

The sampling team will be responsible for maintaining custody of the samples until they are delivered to the carrier or the laboratory. The chain-of-custody record form will then be signed and custody formally relinquished. The containers (bearing custody seals) will be in view at all times or will be stored in a secure place restricted to authorized personnel. Chain-of-custody procedures are discussed in more detail in Appendix F of this SAP.

EQUIPMENT DECONTAMINATION

Before sampling begins, between each well sampled, and prior to leaving the site, equipment such as a water-level measuring tape or M-scope, bailers, filtration apparatus (flasks, funnels, and beakers), and buckets will be decontaminated. Decontamination water will be disposed at a designated location at the landfill. Disposable equipment will be discarded in an appropriate manner. Bailers and Teflon bailer cords will be cleaned as follows:

1. Wash with Micro detergent/potable water solution.
2. Rinse with potable water.
3. Rinse with 10% nitric acid solution (acid will be ultra-pure grade).
4. Rinse with potable water.
5. Rinse with analyte-free deionized water.
6. Air dry.
7. Wrap in aluminum foil until ready for use.

Submersible pumps will be flushed inside and out with a Micro detergent/potable water solution, and rinsed inside and out with potable water followed by rinsing with analyte-free deionized water. Pump tubing will be cleaned in the same manner before use and discarded after use. The filtering apparatus will be rinsed with 10 percent nitric acid solution and rinsed with analyte-free deionized water before every use. Water-level measuring tape or M-scope and buckets will be decontaminated by scrubbing with a Micro detergent/potable water solution followed by rinsing with analyte-free deionized water.

APPENDIX C

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX C

GROUND-WATER LEVEL MEASURMENT PROTOCOLS

APPENDIX C

GROUND-WATER LEVEL MEASUREMENT PROTOCOLS

Depth-to ground water in the monitoring wells will be measured as part of the field program. These data will be converted to water-level elevations using surveyed vertical measuring points on individual well casings.

An electronic measuring tape (M-scope) graduated in tenths of a foot will be the primary instrument of measuring the depth to ground water. Prior to insertion, the measuring tape will be cleaned with Micro solution and rinsed with potable water followed by analyte-free deionized water. The tape will be lowered slowly down the center of the casing. After the electronic buzzer sounds and the light illuminates, signifying that water has been encountered in the well, the tape will be held at the pre-marked surveyed point at the top of the well. The measurement (depth to water) will be recorded on a Geraghty & Miller Water-Level/Pumping Record form (Appendix H). The tape will then be removed from the well and cleaned in the same manner as before insertion into the well.

APPENDIX D

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX D

GAS WELL INSTALLATION AND MONITORING PROTOCOLS

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SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX D

GAS WELL INSTALLATION AND MONITORING PROTOCOLS

OFF-SITE SUBSURFACE GAS STUDY

The location of the three proposed off-site shallow gas monitoring wells is depicted on Figure 1. Also shown on Figure 1, are the locations of the proposed on-site gas monitoring cluster wells, which will be installed and monitored during the OU-1 Remedial Design Program. The installation and monitoring protocols for the proposed on-site gas cluster wells are contained in the OU-1 Remedial Design work plan (LKB 1991). The methodology and protocols for installing the three off-site shallow gas monitoring wells are described below:

CONSTRUCTION OF GAS MONITORING WELLS

The gas monitoring wells will be constructed of hand-slotted, 1-inch diameter PVC casing, installed in boreholes drilled to a depth of 3 to 5 feet using a hand-operated bucket auger or a power auger, depending on soil conditions. The screened portion of each gas monitoring well will be approximately 3 feet long. Each borehole will be backfilled with clean sand (No. 1 J. Morie Co.) to above the top of the screen, and then sealed with a bentonite slurry. The construction details of a typical gas monitoring well are shown on Figure 6. The tops of the wells will be capped and fitted with short lengths of polyethylene and silicone tubing to allow attachment of the OVA probe and sampling equipment. Each gas well will be completed at grade level with protective covers (plates) cemented in place to protect against vandalism.

SUBSURFACE GAS MONITORING

The three off-site gas monitoring wells, the on-site gas monitoring wells in the southwestern portion of the site, and on-site gas monitoring Wells G-13 and G-14 will be monitored for methane and total VOCs on three days of low barometric pressure. A Century Systems Model 88 organic vapor analyzer (OVA), calibrated to a methane standard, will be used for these measurements. A charcoal filter probe will be used for measuring methane, and a standard probe will be used to measure total VOCs (including methane).

APPENDIX E

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX E

FIELD INSTRUMENTATION OPERATING PROCEDURES

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX E

FIELD INSTRUMENTATION OPERATING PROCEDURES

INTRODUCTION

Field instruments used during the investigation will be calibrated and operated in accordance with the following standard operating procedures and with the manufacturers' instructions.

GAS MONITORING EQUIPMENT

A Century Systems Model 88 organic vapor analyzer (OVA) flame-ionization detector (FID) will be used during the Off-Site Subsurface Gas Study to monitor the gas monitoring wells for landfill gas (methane and total VOCs). The OVA will be calibrated prior to use on a daily basis using zero air and a mixture of 100 parts per million (ppm) methane and air. The calibration procedure is detailed in the manufacturer's instructions.

AIR MONITORING EQUIPMENT

PHOTOIONIZATION DETECTOR

A photoionization detector (PID) such as a TIP or HNU meter will be used for monitoring VOCs in the breathing zone during the Off-Site Ground-Water Study (see HASP [Section IV]). The PID will be calibrated prior to use on a daily basis. First the PID will be zeroed using background ambient air followed by a mixture of 100 ppm isobutylene and air. The calibration will be checked using a 50 ppb isobutylene in air.

COMBUSTIBLE GAS INDICATOR

A GasTech Model 4320 meter will be used to measure the Lower Explosive Limit (LEL) of explosive gases during the Off-Site Ground-Water Study. This instrument is calibrated by the manufacturers, and therefore, requires no field calibration.

FIELD ANALYSIS INSTRUMENTS

Field analysis of water samples will consist of measurements of pH, temperature, and specific conductance. A description of the equipment that will be used and the calibration procedure for each is provided below.

TEMPERATURE

This field parameter will be measured using a Taylor Pocket Thermometer with a temperature measuring range of -15°C to 105°C.

pH

This field parameter will be measured using an Orion Research (Model 20) pH meter hand-held, portable, digital meter, or equivalent. Two buffer standards (nominal pH values of 4 and 7 or 7 and 11) will be used to calibrate the instrument, prior to analysis of each sample. The probe will be lowered into the sample container and gently stirred on the sample to allow equilibration before the reading is taken.

SPECIFIC CONDUCTANCE

This field parameter will be measured using a battery-powered conductivity meter (Horizon Model 1484-10). Calibration will be made using a 2000 umho standard solution. Calibration of the meter will be adjusted to temperature. The meter will be calibrated daily

when in use. To measure this parameter, the probe will be lowered into the sample container until a stable reading is obtained.

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APPENDIX F

APPENDIX F

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX F

CHAIN-OF-CUSTODY PROCEDURES

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SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX F

CHAIN-OF-CUSTODY PROCEDURES

The chain-of-custody procedure for the collection of samples that will be sent to the laboratory for analysis is given below:

1. The Field Hydrogeologist or sampling team will maintain custody of any samples that are collected until they are delivered to the overnight common carrier or courier for shipment to the laboratory. All samples shipped to a laboratory will be accompanied by the Geraghty & Miller, Inc. Chain-of-Custody Record form (see Appendix H). The Chain-of-Custody Record form (duplicate) will be completed in the field; the original form will accompany the shipment, and the other copy will be retained by the Field Hydrogeologist for the project file. The Chain-of-Custody Record form will list each of the individual sample containers from each well sampled and will be signed by each of the sampling team members who participated in the sampling program.

2. A separate Chain-of-Custody Record form will be filled out for the contents of each shipment container (cooler). The form will be placed in a plastic bag and taped to the underside of the lid of the cooler.

3. To provide a means of detecting any potential tampering during shipment, all shipment containers (coolers) will have a signed Geraghty & Miller seal placed across the outside of the cooler where the lid and cooler join. In addition, a 2-inch wide transparent tape will be wrapped entirely around the cooler securing the lid firmly to the cooler.

4. Receipts from couriers, air bills, and bills of lading will be retained in the field project file.

APPENDIX G

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX G

BOREHOLE GEOPHYSICAL LOGGING

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APPENDIX G

BOREHOLE GEOPHYSICAL LOGGING PROTOCOL

Natural gamma geophysical logs will be obtained from the deep boring at each of the five drilling locations using a Model II Mount Sopris Instrument Company Logging System. Electric logging will also be performed in the three deep mud rotary borings, but will not be possible in the two exploratory borings because of interference from the steel casing. Before and after each use, the logger probe and cable will be cleaned with a solution of Micro laboratory-grade detergent and potable water, and rinsed with distilled water.

Gamma logging involves the measurements of naturally occurring radiation originating from geologic material in the borehole and provides a qualitative guide to correlating stratigraphy and evaluating permeability. Gamma radiation is emitted from certain elements which are unstable and decay spontaneously into other, more stable elements. Although other types of radiation are given off by natural radioactive minerals (alpha and beta emissions), only gamma rays are measured in well logging because only these rays can penetrate materials such as casing and cement grout. Gamma logging has a unique advantage over electric logging because it can be done in either cased wells or open boreholes, whereas electric logging can be conducted only in uncased boreholes filled with fluid.

The minerals commonly found in sedimentary materials such as clay, limestone, and sandstone contain small amounts of radioactive potassium-40 and decay products of uranium and thorium. Potassium is an important constituent of clay, mica, feldspar, and shale, and its radioactive isotope (potassium-40) emits gamma rays. Because these materials tend to be finer grained, elevated gamma responses are often interpreted as corresponding to sediments of relatively low permeability. Coarser grained sand contains no potassium or radioactive potassium-40, and emits gamma rays at relatively low levels. Consequently, the

gamma log shows more radiation (counts per second) at depths corresponding to clay or silt, and lower radiation levels (fewer counts per second) at depths corresponding to sand or sandstone layers, if the sand is mostly quartz.

Geraghty & Miller will conduct the geophysical logging program by using its truck-mounted EG&G Mount Sopris Model II logging system, which consists of a logger and probe. The probe consists of a scintillation-type receiver and counting circuit. The probe, which is attached to a cable, will be lowered and raised the entire length of each well while graphs are produced by the digital logger recorder, located in the truck. Radiation intensity for a given geologic formation is measured by the probe and expressed as the average number of counts per second. Since the logger is fully automated and the probe is factory sealed, no calibration is required.

The following procedure will be followed for the geophysical logging at each location:

1. Activate the power system for the Winch and Cable Depth and Speed Module. A sufficient length of cable will be spooled out to allow the Model II probe to be centered over the well. Tension will be kept on the cable at all times to avoid forming tangles or loop on the cable spool.

2. Set up the tripod, which supports the probe, over the well and center the probe in the well to ensure that the bottom of the probe is even with land surface.

3. Turn the SPEED control fully counterclockwise, the DIRECTION switch in the OFF position, and engage the brake mechanism.

4. Use the DEPTH PRESET switch on the Cable Depth and Speed Module to set the distance from the reference point to the main measuring point for the probe. The LOAD button will be pressed and the LED display will be checked for the proper setting.

300745

5. Activate the system's computer and load the system and data disks into their respective drives. A file will be created by entering (and storing on the data disk) data specific to the well/borehole being logged.

6. Turn the direction switch to DOWN and lower the probe into the borehole. Simultaneously, the RUN key on the computer keyboard will be pressed, thereby initiating the transmission of data from the probe to the data disk. During this time, the output from the Logging Module will be monitored to select the optimal settings to be used while logging in the up direction. If the cable becomes slack, the winch will be stopped immediately.

7. Press the ESC (Escape) key and turn the Direction switch in the UP position upon reaching the bottom of the borehole. Slack will then be taken out of the cable, and it will be stopped on a full depth increment (i.e., fractional part equal to zero).

8. Create a second file similar in fashion to the first; the data transmission will be initiated by simultaneously pressing the RUN key on the keyboard and turning the SPEED knob clockwise, thereby raising the probe to the top of the borehole.

9. Turn the SPEED control fully counterclockwise, set the Direction switch to STOP, and engage the brake mechanism after the top of the borehole is reached. The ESC key on the computer keyboard is pressed, thereby saving the file.

10. Remove the probe from the borehole and decontaminate. The instrument power and winch power switches will then be turned to OFF.

APPENDIX H

SECTION II: SAMPLING AND ANALYSIS PLAN

APPENDIX H

GERAGHTY & MILLER, INC.

QUALITY ASSURANCE/QUALITY CONTROL FORMS

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GERAGHTY & MILLER, INC.
Ground-Water Consultants

SAMPLE/CORE LOG

Boring/Well _____ Project/No. _____ Page _____ of _____

Site Location _____ Drilling Started _____ Drilling Completed _____

Total Depth Drilled _____ (feet)

Hole Diameter _____ (inches) Type of Sample/ Coring Device _____

Length and Diameter of Coring Device _____ Sampling Interval _____ feet

Drilling Fluid Used _____ Drilling Method _____

Drilling Contractor _____ Driller _____ Helper _____

Prepared By _____ Hammer Weight _____ Hammer Drop _____ inches

Sample/Core Depth (feet below land surface) From	To	Core Recovery (feet)	Time/Hydraulic Pressure or Blows per 6 inches	Sample/Core Description
				300749

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WATER SAMPLING LOG

Project/No. _____ Page _____ of _____

Site Location _____

Site/Well No. _____ Coded/ Replicate No. _____ Date _____

Weather _____ Time Sampling Began _____ Time Sampling Completed _____

EVACUATION DATA

Description of Measuring Point (MP) _____

Height of MP Above/Below Land Surface _____ MP Elevation _____

Total Sounded Depth of Well Below MP _____ Water-Level Elevation _____

Held _____ Depth to Water Below MP _____ Diameter of Casing _____

Wet _____ Water Column in Well _____ Gallons Pumped/Bailed Prior to Sampling _____

Gallons per Foot _____

Gallons in Well _____ Sampling Pump Intake Setting (feet below land surface) _____

Evacuation Method _____

SAMPLING DATA/FIELD PARAMETERS

Color _____ Odor _____ Appearance _____ Temperature _____ °F/°C

Other (specific ion; OVA; HNU; etc.) _____

_____

Specific Conductance, umhos/cm _____ pH _____

Sampling Method and Material _____

Constituents Sampled	Container Description From Lab ____ or G&M ____	Preservative

Remarks _____

Sampling Personnel _____

WELL CASING VOLUMES

GAL./FT.							
	1-¼" = 0.06	2" = 0.16	3" = 0.37	4" = 0.65
	1-½" = 0.09	2-½" = 0.26	3-½" = 0.50	6" = 1.47

300750

GERAGHTY & MILLER, INC.
Environmental Services

GAS MONITORING LOG

Page ___ of ___

Project: ______________________________ Checked by: ______________________

Location: ______________________________ OVA Model No: ____________________

Date	Time	Well No.	OVA Reading Total VOCs & Methane (Standard Probe)	OVA Reading Methane (Charcoal Filter Probe)	Total VOCs

GASMONIT.XLS

GERAGHTY & MILLER, INC.

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Envir. ental Services

Laboratory Task Order No. ____

CHAIN-OF-CUSTODY RECORD

Page (of ____

Project Number _____

Project Location _____

Laboratory _____

Sampler(s)/Affiliation _____

_____

SAMPLE IDENTITY	Code	Date/Time Sampled	Lab ID	SAMPLE BOTTLE / CONTAINER DESCRIPTION									TOTAL

Sample Code: L = Liquid; S = Solid; A = Air

Total No. of Bottles/ Containers

Relinquished by: _____ Organization: _____
Received by: _____ Organization: _____ Date ___/___/___ Time _____

Seal Intact? Yes No N/A

Relinquished by: _____ Organization: _____
Received by: _____ Organization: _____ Date ___/___/___ Time _____

Seal Intact? Yes No N/A

Special Instructions/Remarks: _____

Delivery Method: ☐ In Person ☐ Common Carrier _____ SPECIFY ☐ Lab Courier ☐ Other _____ SPECIFY

3075?

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DAILY LOG

Well(s)_____ Project/No. _____ Page_____ of_____

Site Location _____

Prepared By _____

Date/Time	Description of Activities
	300753

GERAGHTY & MILLER, INC.
Ground-Water Consultants

DRILLING & INSTALLATION OF MONITORING WELLS
DAILY CHECKLIST

PROJECT: _____ WELLS/BORINGS: _____

LOCATION: _____ DATE: _____

G&M PERSONNEL ON SITE: _____ TIME: _____

CHECKED BY: _____

ITEMS	OK/NA	COMMENTS
PRIOR TO DRILLING:		
Contractor has checked for underground utilities		
Well locations staked		
Well drilling permits secured		
G&M QA Manual/QA plan available		
Health & Safety plan (HASP) available		
Safety equipment on site		
Contractor equipment adequate		
Drilling equipment steam cleaned		
Drilling water source approved/sampled		
Drilling equipment water sample taken		
Drilling mud & additives approved		
Sand or gravel pack sample approved		
Grout composition approved		
DURING INSTALLATION:		
Formation samples properly taken and bottled or bagged		
Rock color chart used		
OVA monitoring during drilling & sampling		
Cores properly marked and stored		
Hard hat worn		
Proper safety procedures followed		
Hazardous soil, mud or water properly handled*		
Daily Log kept		
Sample/Core Log form filled out		
Well Construction Log prepared		
Materials/Cost Log prepared		
Location Sketch made		
Tremie pipe used in grouting		
Abandoned well/boring grouted & staked		
Protective casing/well cap/lock installed		
Well identification no. attached		
Well development adequate		
Well elevation and location surveyed in		

*See QA Manual, Drilling and Installation of Monitoring Wells, Sect. 2.16.2.

300754

Instructions: Original to Field Project File; copy to Project Manager and to QA Representative.

JUNE 1986

GERAGHTY & MILLER, INC.
Ground-Water Consultants

UTILITIES AND STRUCTURES CHECKLIST

Project: _____ **Prepared by:** _____

Location: _____ **Date:** _____

Instructions. This checklist has to be completed by a G&M staff member as a safety measure to insure that all underground utility lines, other underground structures as well as above-ground power lines are clearly marked out in the area selected for boring or excavation. **DRILLING OR EXCAVATION WORK MAY NOT PROCEED UNTIL LINES ARE MARKED AND THIS CHECKLIST HAS BEEN COMPLETED.** Arrangements for underground utility markouts are best made at the time of the preliminary site visit to allow client and/or utility company sufficient time. Keep completed checklist and maps onsite; send copy to Project Manager.

Assignment of Responsibility. Client is responsible for having underground utilities and structures located and marked. Preferably, the utilities themselves should mark out the lines.

Drilling or Excavation Sites. Attach a map of the property showing the proposed drilling or excavation site (or if sites are widely separated, several maps) clearly indicating the area(s) checked for underground utilities or underground structures and the location of above-ground power lines.

Utilities and Structures

Type	Not Present	Present	How Marked?[1)]
Petroleum products line			
Natural gas line			
Steam line			
Water line			
Sewer line			
Storm drain			
Telephone cable			
Electric power line			
Product tank			
Septic tank/drain field			
Overhead power line			

1) Flags, paint on pavement, wooden stakes, etc.

Name and affiliation of person who marked out underground lines or structures.

_____ NAME _____ ORGANIZATION _____ PHONE

Emergency Procedures

Persons at site or facility to contact in case of emergency

1. _____ Phone _____

2. _____ Phone _____

Fire Dept.: Phone _____ Ambulance: Phone _____

Utility: Phone _____ Utility: Phone _____

Utility: Phone _____ Utility: Phone _____

Directions to nearest hospital (describe or attach map).

300755

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LOCATION SKETCH

Well(s) _____ Project/No. _____ Page _____ of _____

Site Location _____

Observer _____

(Locate all wells, borings, etc. with reference to three permanent reference points; tape all distances; clearly label all wells, roads, and permanent features)

N

300756

0 ft ft

GERAGHTY & MILLER, INC. Environmental Services

WATER LEVEL/PUMPING TEST RECORD

PAGE ____ OF ____

PROJECT ________ WELL ________ SITE ________

SCREEN SETTING ________ MEASURING POINT DESCRIPTION ________ HEIGHT ABOVE GROUND SURFACE ________

STATIC WATER LEVEL ________ MEASURED WITH ________ DATE/TIME ________

DRAWDOWN ☐ START OF TEST ________ PUMPING WELL ________

RECOVERY ☐ END OF TEST ________

DISTANCE FROM WELL MEASURED TO PUMPING WELL (r) ________ DISCHARGE RATE ________ ORIFICE ________

DATE & TIME	WELL OR t (mins)	HELD (ft)	WET (ft)	DEPTH TO WATER (ft)	s (ft)	DEW.[1] CORR. (ft)	ART.[2] s' (ft)		Q (gpm)	MANO-METER (in)	REMARKS[3]
										300757

1) Dewatering Correction 2) Equivalent Artesian Drawdown 3) pH, Spec. Cond., Temp., Weather, Sand, Turbidity, etc.

G&M Form 10 6-86 Southprint 89-1249

RAGHTY & MILLER, INC.
Ground-Water Consultants

MATERIALS COST LOG

Page _____ of _____

Wells: _____ Project/No.: _____
Site Location: _____ Calculated By: _____ Checked By: _____

Well	Footage Drilled (ft)	Split Spoons (ea)	Screen Diam/Length (ft)	Casing Diam/Length (ft)	Sand or Gravel (bag)	Bentonite Pellets (lb)	Bentonite Powder (bag)	Cement (bag)	Developing Time (hrs)	Rig time (hrs)	Other Items*
Totals											
Unit Cost	$	$	$	$	$	$	$	$	$	$	$
Cost Totals	$	$	$	$	$	$	$	$	$	$	$

*Include items such as Shelby tubes, standby time, protective casings, etc.

GERAGHTY & MILLER, INC.
Ground-Water Consultants

SAMPLE CONTAINER INVENTORY

Project _____

Shipped from _____ Shipped to _____

(laboratory) _____ _____

_____ _____

Phone _____ Attn. _____

SHIPMENT CONTENTS

Shipped			Received	
Bottle Size and Composition	Preservative	Quantity	Quantity	Condition/Comments

Packed by _____ Received by _____

Date _____ Date _____

Shipped by _____ Inspected by _____

Date _____ Date _____

Sealing Method _____ Seal Intact? _____

Remarks:

300759

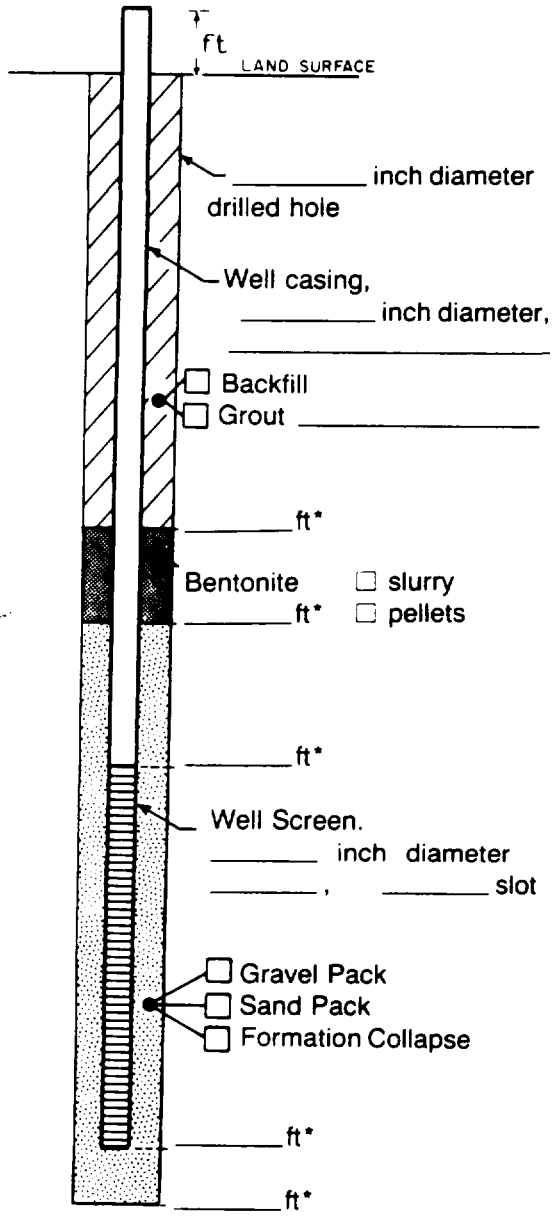
G&M Form 08 1-87

Southprint 87-1783

GERAGHTY & MILLER, INC.
Ground-Water Consultants

WELL CONSTRUCTION LOG

(UNCONSOLIDATED)



Measuring Point is
Top of Well Casing
Unless Otherwise Noted.

*Depth Below Land Surface

Project _____ Well _____

Town/City _____

County _____ State _____

Permit No. _____

Land-Surface Elevation

and Datum _____ feet ☐ Surveyed

_____ ☐ Estimated

Installation Date(s) _____

Drilling Method _____

Drilling Contractor _____

Drilling Fluid _____

_____

Development Technique(s) and Date(s)

_____

_____

_____

Fluid Loss During Drilling _____ gallons

Water Removed During Development _____ gallons

Static Depth to Water _____ feet below M.P.

Pumping Depth to Water _____ feet below M.P.

Pumping Duration _____ hours

Yield _____ gpm Date _____

Specific Capacity _____ gpm/ft

Well Purpose _____

_____

_____

Remarks _____

_____

_____

_____

_____

_____

300760

Prepared by _____

GERAGHTY & MILLER, INC.
Environmental Services

SAMPLING OF MONITORING WELLS
DAILY CHECKLIST

PROJECT: _____ WELL(S): _____

LOCATION: _____ DATE: _____

G&M PERSONNEL ON SITE: _____ TIME: _____

CHECKED BY: _____

ITEMS	OK/NA	COMMENTS
PRIOR TO SAMPLING: Health & safety precautions (HASP) received; equipment ready.		
Sample containers, coolers, received from laboratory; ice or ice packs ready.		
Sampling equipment and supplies inventoried, clean and operational.		
Check in with client at site.		
Integrity of well noted.		
Well area prepared for sampling; plastic placed around well; gasoline-powered pumps placed downwind.		
Well and water-level measurements made and recorded along with other pertinent field information on water sampling log.		
Field instruments calibrated.		
Sample containers labelled; preservatives added, if necessary.		
DURING AND AFTER SAMPLING: Well purged three to five times its volume.		
Sample collected using a bailer or pump as per sampling plan.		
Measurement of field parameters recorded on sampling log.		
Sample containers filled according to collection protocol of analyses.		
Field and trip blanks collected; replicates or split samples collected as per sampling plan.		
Samples stored at 4°C in coolers for transport to lab.		
Water sampling log and chain-of-custody form completed.		
Reusable equipment decontaminated; non-reusable equipment disposed of in appropriate manner.		
Well secured and locked.		
Laboratory contacted to confirm receipt and condition of samples.		

Additional Comments:

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Instructions: Original to Field Project File; copy to Project Manager and to QA Representative.

GERAGHTY
& MILLER, INC.
Environmental Services

TELEPHONE CONVERSATION RECORD

DATE: _____ TIME: _____ PROJECT: _____

FROM: _____ TO: _____

COMPANY: _____ COMPANY: _____

TELE NO: _____ TELE NO: _____

RE: _____

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SECTION 3

SECTION III

QUALITY ASSURANCE PROJECT PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

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SECTION III

QUALITY ASSURANCE PROJECT PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

1.0 INTRODUCTION

This Quality Assurance Project Plan (QAPP) presents the organizational structure, data quality objectives (DQOs) and data management scheme for conducting the OU-2 Remedial Investigation (RI) field program audit and defines the specific quality control (QC) checks and quality assurance (QA) auditing processes. The QAPP is designed to assure that the precision, accuracy, representativeness, comparability, and completeness (the PARCC parameters) of the collected data are known, documented, and adequate to satisfy the DQOs of the study. The format and contents of the QAPP have been prepared in accordance with the following United States Environmental Protection Agency (USEPA) guidance documents: "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans" (USEPA 1983); "Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA" (USEPA 1988); "Data Quality Objectives: Development Guidance for Uncontrolled Hazardous Waste Site Remedial Response Activities" (USEPA 1987); "NEIC Policies and Procedures" (USEPA 1986); and "Region II CERCLA Quality Assurance Manual" (USEPA 1989a).

The QAPP serves as an overall summary of the QA/QC structure of the project. Some parts of the structure are described in this document (e.g., data management); and other parts are described in the SAP and are incorporated into the QAPP by reference (e.g., Standard Operating Protocols and Procedures [SOPs], which provide detailed descriptions of the methodologies that will be followed for the RI field tasks).

The internal laboratory SOPs and quality assurance/quality control (QA/QC) procedures are described in the laboratory QAPP, an independent plan provided by the

analytical laboratory (Industrial & Environmental Analysts, Inc. [IEA] Monroe, Connecticut), and appended to this document (Appendix A). The SOPs provided by IEA are consistent with the USEPA Contract Laboratory Program (CLP) Statements of Work (SOWs) planned for this project.

The RI will be performed to obtain representative data to define the off-site groundwater quality conditions north-northeast of the landfill and the off-site extent of subsurface gas migration southwest of the landfill. These data will be used to evaluate the nature and extent of the contamination for the development of appropriate remedial alternatives for the feasibility study (FS).

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The project organizational structure is discussed in Section 8.0 of the Sampling and Analysis Plan (SAP) (Section II). The responsibilities of the key personnel are detailed below.

- The Project Coordinator is responsible for overseeing the implementation of the Consent Order. To the maximum extent possible, all documents, including reports, approvals and other correspondence concerning the activities performed pursuant to the terms and conditions of the Consent Order will be directed through the project coordinator.

- The Project Director is responsible for the overall QA including technical adequacy of the RI activities and reports, and conformance to the scope of work

- The Project Manager is responsible for the following: sampling QC; overall project coordination; adherence to the project schedules; directing, reviewing, and assessing the adequacy of the performance of the technical staff and subcontractors assigned to the project; implementing corrective action, if warranted; interacting with the project coordinator; preparing the monthly progress and RI reports; and maintaining full and orderly project documentation.

- The Project QA/QC Manager is responsible for performing systems auditing, and for providing independent data quality review of project documents and reports.

- The Project Health and Safety Coordinator is responsible for implementing the site-specific health and safety directives in the HASP (Section IV) and for contingency response.

- The Field Hydrogeologist is responsible for coordination of the activities of field personnel and of the drilling and surveying subcontractors; adherence of the field work to the procedures specified in the QAPP and SAP (Section II); and rigorous documentation of the field work. The field hydrogeologist is also designated as the site safety officer.

- The Data Validator is responsible for review of laboratory data for compliance with the QA objectives for the PARCC parameters; and notifications to the project manager of any QC deficiencies.

- The RI Project Support Team members include the sampling team, support staff (e.g., data processors, secretaries, and in-house experts in hydrogeology and chemistry, etc.) who are responsible for the technical direction and adequacy of the work in their respective areas of specialty which are or may be required to meet the project objectives.

Tasks that will be performed by subcontractors include drilling, surveying, and analytical (laboratory) testing. The internal project organizational structure within the laboratory is described in the laboratory QAPP (Appendix A of this QAPP). Resumes of key project personnel are presented in Appendix A of the SOP Overview (Section I).

3.0 QUALITY ASSURANCE/QUALITY CONTROL

The overall QA objective is to develop and implement procedures for field measurements, sampling, and analytical testing that will provide data of known quality that is consistent with the intended use of the information. This section defines the objectives by (1) describing the use of the data (2) specifying the applicable QC effort (field checks and analytical support levels), and (3) defining the QC objectives (data quality acceptance criteria).

3.1 DATA USAGE AND REQUIREMENTS

The field measurements and laboratory analyses will be used to support one or more steps in the RI process. These field steps include off-site characterization of the potential contamination, evaluation of remedial alternatives, if necessary, and engineering design of remedial alternatives. The intended uses of the data from each of the RI activities are summarized in Table 1. The data to be collected range from qualitative information (based on field observations) to quantitative laboratory analyses.

An important factor in the use of the data will be the ability to evaluate site conditions with respect to the applicable or relevant and appropriate requirements (ARARs). The RI will generate analytical data that will allow comparison with ARARs as listed in Table 3.

During the well installation program, a photoionization detector (PID), such as a TIP or HNU meter will be used to monitor for the presence of VOCs in the breathing zone (see the HASP in Section IV of this document). During the subsurface gas study, the new off-site gas monitoring wells and the existing gas monitoring wells in the southwestern portion of the landfill will be monitored for total volatile organic compounds (TVOCs) and methane. The

operating procedures for the PID, including calibration and QC, are discussed in Appendix E of the SAP (Section II). Leachate indicators (hardness, alkalinity, ammonia, pH, temperature, chloride, and specific conductance) will be analyzed in the field during the drilling of the two exploratory borings to determine the vertical water-quality profiles at these locations. Ground-water samples collected from the Nassau County monitoring wells/and public supply wells will also be analyzed in the field for the leachate indicators. The field analysis procedures for the leachate indicators are discussed in Appendices B and E of the SAP. Twenty percent of these samples will be sent to the laboratory for confirmatory analysis. The methods and quantitation limits to be used to analyze these samples by the laboratory are listed in Appendix B of the QAPP.

For both sampling rounds, ground-water samples will be collected from the existing and new monitoring wells and analyzed for the same parameters employed during the OU-1 (on-site) RI (Geraghty & Miller, Inc. 1989) as listed in Appendix B of this QAPP. To provide for lower quantitation limits, the document, "Contract Laboratory Program Statement of Work for Low Concentration Water for Organics Analysis" (USEPA 1990) will be followed to analyze the ground-water samples collected for VOCs.

The documents, "Contract Laboratory Program Statement of Work for Inorganics Analysis" (USEPA 1990b) and the "Contract Laboratory Program Statement of Work for Organics Analysis" (USEPA 1991) will be followed by the laboratory for the analyses of inorganics, semivolatiles, and PCBs for the ground-water samples collected during the OU-2 RI. SOPs for sample control, calibration, analysis of samples, data analysis, data validation, data reporting, internal QC checks, system performance audits, preventive maintenance, and data assessment were prepared in accordance with the Statements of Work (SOWs) for USEPA CLP analysis. Analytical procedures are described in more detail in Section 7.0 (Analytical Procedures) of this QAPP. The sample handling procedures described in the

laboratory QAPP (Appendix A of this QAPP) are consistent with the SOWs mentioned above.

Quantitation limits for the organic and inorganic parameter analyses are provided in the parameters list (Appendix B of this QAPP); however, dilution or interference effects may make it necessary to raise these limits. The laboratory will make every effort to achieve quantitation limits as low as practicable and will report estimated concentration values at less than the detection limit by flagging the value with J.

3.2 LEVEL OF QUALITY CONTROL EFFORT

The laboratory will follow standard QC measures to provide data of known and defensible quality. The data quality elements that will be checked and documented include the PARCC parameters which are discussed separately below:

3.2.1 Precision

Measurements of data precision are necessary to demonstrate the reproducibility of the analytical data. Precision of the ground-water sample data will be determined from the analyses of matrix spike and matrix spike duplicates (MS/MSDs). MS/MSD samples will be collected at a frequency of 5 percent (one MS/MSD pair per 20 samples), or one per 2-week sampling period. An extra sample volume will be collected for each replicate and MS/MSD sample taken. MS/MSD samples will be labeled on the sample container and appropriate sample log and chain-of-custody forms. Laboratory precision requirements are provided in Appendix A of this QAPP.

3.2.2 Accuracy

Accuracy is the relationship of the reported data to the "true" value. The accuracy of the methods used for the analyses of ground-water samples will be evaluated through the use of calibration standards, MS/MSD analyses, and surrogate spikes. MS/MSD samples will be collected and analyzed at a frequency of 5 percent (one MS and one MSD per 20 samples per matrix), or one MS/MSD pair per 2-week period. An extra sample volume will be collected for each MS/MSD sample taken. Laboratory accuracy requirements are provided in Appendix A of this QAPP.

3.2.3 Representativeness

All data obtained during the RI, including data from replicate samples, should be representative of actual conditions at the sampling location. Field replicates will be collected and analyzed at a frequency of 5 percent (one per 20 samples) or at least one per sample matrix (ground-water only) if less than 20 samples are to be collected. Considerations for evaluating the representativeness of the data include, but are not limited to, the following: the sampling location; the methods used to obtain samples at the site; and the appropriateness of the analytical method to the type of sample obtained. All field sampling activities will be performed according to the protocols and SOPs described in the SAP (Section II). Laboratory representativeness requirements are provided Appendix A of this QAPP.

3.2.4 Comparability

Comparability will be achieved by utilizing standardized sampling and analysis methods and data reporting format. The data will be generated such that it is comparable to the existing data base.

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3.2.5 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement program compared to the total amount collected. The validity of the collected data will be evaluated utilizing the appropriate QA/QC guidelines. Laboratory completeness requirements are provided in Appendix A of this QAPP.

The sampling team will use many different types of QA/QC samples to ensure and document the integrity of the sampling procedures, laboratory sample handling procedures, and the validity of the measurement data.

Field replicate samples will be collected to also demonstrate the reproducibility of the sampling technique. These analyses will be in addition to the replicates that IEA must run and will not be replaced by a laboratory-generated replicate. The replicate sampling locations will be selected for each sampling event. Since the replicate will be "blind" to the laboratory, it will have a coded identity on its label and on the chain-of-custody record form. The actual sampling location will be recorded on a daily log form and on the water sampling log form.

Analyte-free deionized water will be obtained from the laboratory to be used for trip blanks, collecting field blanks, and the final decontamination rinse where required. This water will be prepared and analyzed by the laboratory on a monthly basis and a record of this procedure will be kept on file. Protocols for the handling of trip blanks, collection of field blanks, and decontamination of equipment are provided in the SAP (Section II). To determine if cross-contamination has occurred during ground-water sampling, one field blank per day of sampling will be prepared using analyte-free deionized water provided by the laboratory. Field blanks will be analyzed for all the test parameters, except leachate parameters, listed in Appendix B of this QAPP using CLP methods.

One trip blank, consisting of two 40-mL vials filled by the laboratory with analyte-free deionized water, will be provided by IEA daily for each container used to ship and store volatile organic samples during each sampling event. Trip blanks will not be more than 24 hours older than the associated ground-water samples and will be analyzed for VOCs only.

The USEPA standard series of analytical support levels to denote types of analysis and the associated level of QC effort are listed below.

Level 1. Field screening or analysis using portable instruments.
Level 2. Field analyses using more sophisticated instruments.
Level 3. Standard USEPA-approved laboratory methods.
Level 4. USEPA CLP routine analytical services laboratory methods.
Level 5. USEPA CLP non-standard services laboratory methods.

The analytical support levels which will be used to generate the project data are summarized in Table 1. As shown in this table, the analyses that will be performed during the RI will fall within Levels 1, 3, and 4. Ground-water samples that will be tested in the field for primary and secondary leachate indicators will be performed at Level 1. The measurements for TVOCs and methane in the gas monitoring wells will also be performed at Level 1. The ground-water samples that will be sent to IEA for analysis will be performed using standard USEPA laboratory methods (Level 3) and by the routine analytical services methodology in CLP SOWs for Inorganics and Organics Analyses (Level 4).

3.3 QUALITY CONTROL OBJECTIVE

The QC objective for the OU-2 RI is to provide data of known and defensible quality. Several different types of QC check samples will be analyzed and the results will be

compared to data quality acceptance criteria and/or QC control limits that are specified for each method. The laboratory will routinely run these QC samples in accordance with the protocols and frequencies specified in the CLP SOWs for Organics and Inorganics Analyses and will provide a comparable level of QC effort for the non-CLP analytical parameters. The QC check samples include the following:

- Blank samples
  - Preparation
  - Method
  - Holding
  - Calibration
- Initial and continuing calibrations
- Surrogate spikes
- Matrix spikes/analytical spikes
- Duplicate samples
- Control samples
- Reagent check samples

The QC control limits, or data quality acceptance criteria, for each of the types of QC check samples are also specified in the laboratory QAPP (Appendix A of this QAPP). The specific types and frequencies of QC checks which will be performed in support of each test method, the calibration procedures for each instrument, and the QC control limits and/or data quality acceptance criteria for each of the types of QC check samples, are specified in detail in the laboratory QAPP (Appendix A of this QAPP).

4.0 SAMPLING PROCEDURES

Samples will be collected in accordance with the approved project SOPs. These are provided in the SAP (Section II) and include the following:

- Ground-Water Monitoring Well Specifications and Installation Protocols (Appendix A of the SAP).
- Ground-Water Sampling Protocols (Appendix B of the SAP).
- Ground Water-Level Measurement Protocols (Appendix C of the SAP).
- Gas Monitoring Well Installation and Sampling Protocols (Appendix D of the SAP).
- Field Instrumentation Operating Procedures (Appendix E of the SAP).
- Chain-of-Custody Procedures (Appendix F of the SAP).
- Borehole Geophysical Logging Protocols (Appendix G of the SAP).
- Geraghty & Miller, Inc. QA/QC Forms (Appendix H of the SAP).

The SOPs specify detailed step-by-step protocols for sample collection and address the following as appropriate:

- Use of sampling equipment.
- Decontamination of sampling equipment.
- Pre-sampling requirements (well evacuation volumes).
- Field screening procedures.
- Field analysis procedures (for hardness, alkalinity, ammonia, and chloride).
- Field QC check sample collection procedures (blanks, rinsates, replicates).
- Sample packaging and shipment.
- Sampling documentation and chain-of-custody.

All samples will be delivered to the laboratory 24 hours from time of collection. Preservation, container, and holding time requirements for the parameters to be analyzed are listed in Table 4.

5.0 SAMPLE CUSTODY

A chain-of-custody record will be maintained for each sample collected and will provide an accurate written record that can be used to trace the possession and holding of samples from collection through analysis and reporting. Sample bottles to be used for this project will be selected, prepared, and quality controlled according to OSWER Directive #9240-0-005 "Specifications and Guidance for Obtaining Contaminant-Free Sample Containers" (USEPA 1989b).

The procedures that will be followed to provide the chain-of-custody in the field from sample collection through shipment to the laboratory (including sample preservation) are specified in Appendices B, E, and F of the SAP (Section II). The procedures that will be used to continue the chain-of-custody for each sample from its arrival in the laboratory through analysis and reporting are specified in the laboratory QAPP (Appendix A of this QAPP). The laboratory sample custody procedures conform to the guidelines in the USEPA CLP. The project samples will be retained by the laboratory until the holding times are exceeded, or until permission to discard is received.

6.0 CALIBRATION PROCEDURES

The calibration procedures for field instrumentation are discussed in the SAP (Section II). These procedures are described for the following instruments:

- Water-level recorder (M-scope).
- OVA flame ionization detector.
- PID (TIP or HNU).
- pH meter.
- Specific conductance/temperature meter.
- Ammonia meter.
- Geophysical logger.
- Hach test kits.
- Combustible gas indicator.

The calibration procedures for laboratory instrumentation are discussed in the laboratory QAPP (Appendix A of this QAPP).

7.0 ANALYTICAL PROCEDURES

The methods for analyzing the leachate parameters are listed in Table 1 and the laboratory QAPP (Appendix A of this QAPP). The analytical methods for testing for the volatile, semivolatile, PCB, and inorganic parameters are those specified in the USEPA CLP SOWs. The types and frequencies of QC checks will be those specified in the analytical methods and are discussed in Section 3.3 (Quality Control Objectives) of this QAPP. Full CLP data packages will be requested for the volatile, semivolatile, PCB, and inorganic parameters and comparable data packages for the non-CLP analytical parameters.

8.0 DATA REDUCTION, VALIDATION, AND REPORTING

The laboratory procedures for reducing, validating, and reporting the analytical data are described in the laboratory QAPP (Appendix A of this QAPP). The laboratory data will also be validated consisting of a systematic review of the analytical results and QC documentation, and will be performed in accordance with the guidelines in "CLP Organics Data Review and Preliminary Review" (USEPA 1992a) and "Evaluation of Metals Data for the Contract Laboratory Program (CLP)" (USEPA 1992b).

On the basis of this review, the data validator will make judgments and comments on the quality and limitations of specific data, as well as on the validity of the overall data package. The data validator will prepare documentation of his or her review and conclusions using the standard USEPA Inorganics Regional Data Assessment and Organics Regional Data Assessment forms to summarize any overall deficiencies that require attention. General laboratory performance will also be assessed by the data validator. These forms will be accompanied by appropriate supplementary documentation, clearly identifying specific problems.

The data validator will inform the project manager of data quality and limitations, and assist the project manager in interacting with the laboratory to correct any data omissions and/or deficiencies. The laboratory may be required to rerun or resubmit data depending on the extent of the deficiencies, and their importance in meeting the data quality objectives within the overall context of the project.

The validated laboratory data will be reduced and tabulated electronically. The tabulated format will be suitable for inclusion in the data validation report and the RI report and will be designed to facilitate comparison and evaluation of the data. The data tabulations will be sorted by classes of constituents (e.g., VOCs, semivolatile organic compounds, inorganics). Each individual table will contain the following information:

sample number; analytical parameters; detection limits; concentrations detected; and qualifiers, as appropriate.

The field measurement data will be similarly reduced into a tabulated format suitable for inclusion in the RI report and will be designed to facilitate comparison and evaluation for the data. These tabulations will include but not be limited to the following information:

- Field screen (OVA) results.
- Field analyses (pH, temperature, leachate indicators, and specific conductance).
- Well construction details.
- Water-level measurements and surveyed measuring point elevations.
- Gas monitoring results.

Field logs will be transferred into typed formats or organized in their original form for inclusions as RI report appendices. The following log forms will be used:

- Sample/Core Logs.
- Well Construction Logs.
- Water Level Logs.
- Water Sampling Logs.
- Gas Sampling Logs.

The tables and logs will be compiled whenever feasible by the field hydrogeologist, who will inform the project manager of any problems encountered during data collection, identify apparent inconsistencies, and provide opinions on the data quality and limitations. The tables and logs will be used as the basis for data interpretation and will be checked against the original field documentation by an independent reviewer prior to use.

9.0 INTERNAL QUALITY CONTROL

The field hydrogeologist will make use of the following types of QA/QC samples to ensure and document the integrity of the sampling and sample handling procedures and the validity of the measurement data: field replicates, field blanks, and laboratory-prepared trip blanks. The frequencies for collecting the QA/QC samples are specified in Section 3.2 (Level of Quality Control Effort) of this QAPP. The procedures for collecting the QA/QC samples are specified in the SAP (Section II).

Two types of quality assurance mechanisms are used to ensure the production of analytical data of known and documented quality: analytical method QC, and program QA. The internal quality control procedures for the analytical services on samples to be provided are specified in the laboratory QAPP (Appendix A of this QAPP). These specifications include the types of control samples required (sample spikes, surrogate spikes, reference samples, controls, blanks), the frequency of each control, the compounds to be used for sample spikes and surrogate spikes, and the quality control acceptance criteria. The laboratory will be responsible for documenting that both initial and ongoing instrument and analytical QC criteria are met in each package. This information will be included in the packages generated by IEA and will be evaluated during the validation performed by Geraghty & Miller.

The field QA/QC analytical results will also be compared to acceptance criteria, and documentation will be performed showing that those criteria have been met. Any samples in nonconformance with the QC criteria will be identified and reanalyzed by the laboratory, if possible. The following QC procedures will be employed by the laboratory for analyses of ground-water samples:

- Proper storage of samples.

- Use of qualified and/or certified technicians.
- Use of calibrated equipment traceable to National Bureau of Standards or USEPA standards.
- Formal independent confirmation of all computation and reduction of laboratory data and results.
- Use of standardized test procedures.
- Inclusion of replicate samples at a frequency of one replicate per 20 samples.

10.0 PERFORMANCE AND SYSTEM AUDITS

System audits will be performed on a periodic basis, as appropriate, to assure that the RI field program is implemented in accordance with the approved project SOPs and in an overall satisfactory manner. Examples of systems audits that will be performed by Geraghty & Miller project personnel during the RI are as follows:

- The field hydrogeologist will supervise and check on a daily basis the following tasks: that the ground-water program, subsurface gas program and other field programs are conducted correctly; that monitoring wells are installed and developed correctly; that field measurements are made accurately; that equipment is thoroughly decontaminated; that samples are collected and handled properly; and that all field work is accurately and neatly documented. QA checklists will be filled out daily during the drilling, installation, and development of new monitoring wells, and during sampling programs. The QA daily checklists for these activities are provided in Appendix H of the SAP (Section II).

- On a timely basis, the data validator will review the data package submitted by the laboratory to check the following information: that all requested analyses were performed; that sample holding times were met; that the data were generated through the approved methodology with the appropriate level of QC effort and reporting; and that the analytical results are in conformance with the prescribed acceptance criteria. The data validator will evaluate the data quality and limitations on the basis of these factors.

- The project manager will oversee the field hydrogeologist and data validator, and check that the management of the acquired data proceeds in an organized and expeditious manner.

- System audits of the laboratory are performed on a regular basis by the USEPA, as well as by the New York State Department of Environmental Conservation (NYSDEC). These audits are discussed in the laboratory QAPP (Appendix A of this QAPP).

Performance audits of laboratories participating in the CLP are performed quarterly in accordance with the procedures and frequencies established by USEPA for the CLP. The laboratory performance evaluation audits are discussed in the laboratory QAPP (Appendix A of this QAPP).

11.0 PREVENTIVE MAINTENANCE

Geraghty & Miller has established a program for the maintenance of field equipment to ensure the availability of equipment in good working order when and where it is needed, as indicated in the following examples:

- An inventory of equipment, including model and serial number, quantity, and condition will be maintained. Each item will be tagged and signed out when in use, and its operating condition and cleanliness will be checked upon return. Routine checks will be made on the status of equipment, and spare parts will be stocked. An equipment manual library will also be maintained.

- The field hydrogeologist is responsible for making sure that the equipment is tested, cleaned, charged, and calibrated in accordance with the manufacturer's instructions before being taken to the field.

The laboratory also follows a well-defined program to prevent the failure of laboratory equipment and instrumentation. This preventive maintenance program is described in the laboratory QAPP (Appendix A of this QAPP).

12.0 DATA ASSESSMENT PROCEDURES

The field- and laboratory-generated data will be assessed for the PARCC parameters. Both quantitative and qualitative procedures will be used for these assessments. The criterion for assessment of field measurements will be that the measurements were taken properly using calibrated instruments. Assessment of the sampling data with respect to field performance will be based on the criteria that the samples were properly collected and handled. Field QC check sample results will also be considered in assessing the representativeness and comparability of the samples collected. The project manager will have overall responsibility for data assessment and integration of that assessment into data use and interpretation.

The laboratory will calculate and report the precision, accuracy, and completeness of the analytical data. Precision will be expressed as the relative percent difference (RPD) between values for duplicate samples. Accuracy will be expressed as percent recoveries (PR) for surrogate standards and matrix spike compounds. The precision and accuracy results will be compared to the prescribed QC acceptance criteria. The QC acceptance criteria prescribed for each test method are presented in the laboratory QAPP. For the organic and inorganic parameters listed in Appendix B of this QAPP, the QC acceptance criteria conform to control limits established in the CLP SOWs. Completeness is expressed as the percentage of valid data, based on the total amount of data intended to be collected.

Rigorous QA/QC procedures will be followed for the collection of samples. The SAP sampling protocols will be strictly adhered to in order to maintain consistency in sampling and representativeness and comparability of the samples.

The assessment of data representativeness with respect to laboratory performance will be based on sample handling and analyses with respect to holding times and also on the

method blank results. Data comparability will be assessed based on laboratory performance with respect to USEPA analytical protocols.

Split or duplicate samples may be collected by the USEPA or its authorized representative during the RI field programs. These samples will be collected in accordance with the procedures provided in Appendix B of the SAP (Section II). The USEPA will provide the project manager with the oversight work plan or similar document identifying the number and location of split samples to be taken. The USEPA will be provided at least 10 days advance notice by the project manager for each sampling event. Split or duplicate samples may also be collected by Geraghty & Miller, if any samples are collected independently by the USEPA that relate to the RI program. The same 10-day minimum notification is applicable.

13.0 CORRECTIVE ACTIONS

The QA/QC program contained in this QAPP will enable problems to be identified, controlled, and corrected. Potential problems may involve non-conformance with the SOPs and/or analytical procedures established for the project, or other unforeseen difficulties. Any persons identifying an unacceptable condition will notify the field hydrogeologist, where applicable, and/or the project manager. The project manager, with assistance from the project QA/QC manager, will be responsible for developing and initiating appropriate corrective action and verifying that the corrective action has been effective. For laboratory analysis, both the identified deviations and corrective actions will be documented on the Corrective Action report, which is provided in the laboratory QAPP (Appendix A of this QAPP).

Corrective actions may include repeating measurements, resampling and/or reanalysis of samples, and amending or adjusting project procedures. If warranted by the severity of the problem (e.g., if monitoring wells require resampling or if the project schedule may be affected), the project coordinator and USEPA remedial project manager will be notified. Additional work, which is dependent upon an unacceptable activity, will not be performed until the problem has been eliminated.

The laboratory maintains an internal closed-loop corrective action system and is described in the laboratory QAPP (Appendix A of this QAPP).

14.0 QUALITY ASSURANCE REPORTS

Regular QA reporting throughout the duration of the project, as well as reporting on an as-needed basis will include the following:

- Monthly progress reports will be submitted to the USEPA remedial project manager. At a minimum these reports will include the following: a description of the activities that have taken place during the month; all validated results of sampling, tests, analytical data, and interpretations received; a description of all data anticipated and activities scheduled for the next month; and a description of any problems encountered or anticipated.

- Conference calls and/or meetings will be scheduled if requested by the project coordinator or by the USEPA remedial project manager to discuss any concerns that may arise during the course of the RI field program that might require significant corrective actions, changes to the scope of work, or departures from the approved project SOPs.

- Serious deficiencies in sampling and/or monitoring data will be reported to the USEPA as soon as practicable after such deficiencies have been noted.

The laboratory's internal QA reporting is described in the laboratory QAPP (Appendix A in this QAPP).

15.0 REFERENCES

Geraghty & Miller, Inc. 1989. Interim Remedial Investigation Report, Syosset Landfill, Syosset, New York. August 1989.

U.S. Environmental Protection Agency (USEPA). 1983. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans. QAMS-005/80.

U.S. Environmental Protection Agency (USEPA). 1986. NEIC Policies and Procedures. EPA-330/9-78-001-R.

U.S. Environmental Protection Agency (USEPA). 1987. Data Quality Objectives: Development Guidance for Uncontrolled Hazardous Waste Site Remedial Response Activities. OSWER Directive 9355.0-7B. Washington D.C.: Hazardous Site Control Division.

U.S. Environmental Protection Agency (USEPA). 1989a. Region II CERCLA Quality Assurance Manual, Revision I.

U.S. Environmental Protection Agency (USEPA). 1989b. Specifications and Guidance for Obtaining Contaminant-Free Sample Containers. OSWER Directive 9240.0-005.

U.S. Environmental Protection Agency (USEPA). 1990a. Contract Laboratory Program Statement of Work for Low Concentration Water for Organics Analysis. OLCO1.0

U.S. Environmental Protection Agency (USEPA). 1990b. Contract Laboratory Program Statement of Work for Inorganics Analysis. ILMO1.0.

U.S. Environmental Protection Agency (USEPA). 1991. Contract Laboratory Program Statement of Work for Organics Analysis OLM01.6.

U.S. Environmental Protection Agency (USEPA). 1992a. CLP Organics Data Review and Preliminary Review. SOP No. HW-6, Revision 8.

U.S. Environmental Protection Agency (USEPA). 1992b. Evaluation of Metals Data for the Contract Laboratory Program (CLP), Revision 11.

APPENDIX A

SECTION III: QUALITY ASSURANCE PROJECT PLAN

APPENDIX A

LABORATORY QUALITY ASSURANCE PROJECT PLAN

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CONTENTS

16.0 STANDARD OPERATING PROCEDURE - THE ANALYSIS OF SAMPLE EXTRACTS FOR PESTICIDE/PCBs BY USEPA CLP PROTOCOLS

17.0 STANDARD OPERATING PROCEDURE - PERKIN-ELMER 3030 ATOMIC ABSORPTION SPECTROMETER (GRAPHITE FURNACE)

18.0 STANDARD OPERATING PROCEDURE - PERKIN-ELMER 5100PC ATOMIC ABSORPTION SPECTROMETER (GRAPHITE FURNACE) 5100N

19.0 STANDARD OPERATING PROCEDURE - COLD VAPOR ATOMIC ABSORPTION (CVAA) FOR THE DETERMINATION OF MERCURY IN WATER

20.0 STANDARD OPERATING PROCEDURE - THERMO JARRELL ASH ICAP61

21.0 STANDARD OPERATING PROCEDURE - ICP SET UP PE6500

22.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF SAMPLES FOR TOTAL CYANIDE CLP PROTOCOL

23.0 STANDARD OPERATING PROCEDURE - TRACKING METALS AND CYANIDE SAMPLE ANALYSIS

24.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF TOTAL ALKALINITY IN WATER SAMPLES

25.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF TOTAL DISSOLVED SOLIDS IN WATER

26.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF AMMONIA - NITROGEN

27.0 STANDARD OPERATING PROCEDURE - FOR CHLORIDE

28.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF SULFATE IN WATER

29.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF HARDNESS IN WATER

30.0 STANDARD OPERATING PROCEDURE - ANALYSIS OF NITRATE AND NITRITE FOR WATER SAMPLES

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1991 QUALITY ASSURANCE PROGRAM PLAN (QAPmP)

FOR

IEA, INC.-CONNECTICUT

MONROE, CONNECTICUT 06468

300799

1991 QUALITY ASSURANCE PROGRAM PLAN (QAPmP)

FOR

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MONROE, CONNECTICUT 06468

1.0 INTRODUCTION

This Quality Assurance Program Plan (QAPmP) covers laboratory operation at Industrial and Environmental Analysts, Inc.-Connecticut (IEA, Inc.-CT) located at 200 Monroe Turnpike, Monroe, Connecticut. The purpose of this general QAPmP is to provide information on laboratory operations as required for specific Quality Assurance Project Plans (QAPjPs), and to provide the basis for the Quality Assurance Program at IEA, Inc.-CT.

This QAPmP is based upon USEPA guidelines as are specified in the following EPA documents:

1) NPO and ORD QAPP Guidance, Quality Assurance Management Staff (QAMS), USEPA, September, 1987.

2) QAMS-004/80, Guidelines and Specifications for Preparing Quality Assurance Program Plans. Quality Assurance Management Staff (QAMS), USEPA, 1979.

The second document is also available as EPA publication, EPA-600/8-83-024. Regulatory guidance for preparing QAPmPs is given in CFR part 30, #30.503, paragraph (e).

2.0 QUALITY ASSURANCE (QA) POLICY STATEMENT

It is the policy of IEA, Inc.-CT that the Quality Assurance (QA) Program will be appropriate to assure that all data collected and reported will be of known and documented value.

The objective of the QA Program (QAPmP) is to ensure, assess and document that all data collected, stored and reported are scientifically valid, defensible and of the precision and accuracy required to meet the objectives of our clients.

It is the goal of IEA, Inc.-CT to provide the best laboratory services to our clients. To accomplish this, the product which we produce, analytical measurement data, must be of defined quality and at the same time conform to government regulations and requirements.

All laboratory activities, from sample receipt to analysis to final report generation, must adhere to the laboratory Standard Operating Procedures (SOPs) which have been developed to provide quality environmental data with adequate documentation to be legally defensible and hence of maximum use by our customers.

Laboratory SOPs form the framework of the Quality Assurance Program Plan (QAPmP) at IEA, Inc.-CT. The purposes of the QAPmP are basically to ensure that our SOPs fulfill all government and/or private client requirements, that proper documentation of all quality control (QC) activities is accomplished, and that the SOPs are followed by the laboratory staff.

It is the policy of the laboratory, therefore, to build the quality control requirements into our SOPs. By adhering to the SOPs, laboratory management is thereby assured that the quality of the data produced will provide the basis for long term relationships with our clients through customer satisfaction.

3.0 MAJOR MISSION ELEMENTS REQUIRING QUALITY ASSURANCE

The primary focus of the laboratory is measurement of specific analytes in environmental samples (air, soil and water). All elements of laboratory measurements are subject to the requirements of the laboratory's QAPmP. The major types of laboratory operations affected are as follows:

- Characterization of waste samples per the Resource Conservation and Recovery Act (RCRA) for either disposal or delisting purposes.
- Analysis of drinking water samples in support of the Safe Drinking Water Act.
- Analysis of environmental samples in accordance with contracts with the USEPA CLP program and various state agencies.
- Analysis of environmental samples for contaminants such as those compounds found on the EPA priority pollutant list, target compound list, etc. for site assessment purposes.

4.0 PLANNING FOR ENVIRONMENTAL DATA COLLECTION ACTIVITIES (EDCAs)

According to EPA's QAMS[1], IEA, Inc.-CT is defined as an "Analytical Support Group". EPA's QAMS defines an "Analytical Support Group" as "any group -- section, branch, or entire laboratory -- that provides physical, chemical or biological sampling or analysis on a service basis". According to QAMS, such analytical support groups do not manage the EDCA projects but only provide services. IEA, Inc.-CT, as an analytical support group, does not determine or write Data Quality Objectives (DQOs) or Quality Assurance Project Plans (QAPjPs).

4.1 Adherence to Data Quality Objectives (DQOs)

Data Quality Objectives (DQOs) have been specified by EPA as the process of defining that type of data will be required by the client for a site or project. The following two steps of defining DQOs must be taken before a project begins:

1) "...the user (of data) must first specify the quality of data he needs, then..."

2) "...the degree of quality control necessary to assure that the resultant data satisfy his specifications must be determined."

The laboratory is in the business of measuring environmental samples in order to generate data which is to be used by our customers to make sound decisions in relation to site contamination (or lack of), water/air/soil quality, public health concerns and the environment in general. Some of the sites have formal QAPjPs with specified DQOs, however, most sites do not have a formal QAPjP.

Each sample analyzed from a given site can present a set of unique analytical problems which potentially fall outside the realm of established methodologies and protocols. It is the goal of the laboratory to produce data which is 100 percent usable to the client but in the real world this is not always possible. For some projects or sites, achieving 50 percent usable data may be considered an excellent achievement by the laboratory while at another site, achieving 90 percent usable data may be considered barely adequate.

It is the policy of IEA, Inc.-CT to meet the requirements of DQOs in QAPjPs when the laboratory has contracted to provide measurement data in support of an on-going client contract. These DQOs must be clearly defined by the client to the laboratory.

4.2 Guidance for the Preparation of Standard Operating Procedures (SOPs)

Routine analytical work will be performed according to standard operating procedures (SOP's must satisfy the requirements of the regulations for which they were developed). All measurements will be made using methods developed in the laboratory or methods mandated by regulatory or legal/contractual requirements. No method will be used to obtain data until it is known to be applicable and competence has been gained in its use. If it appears that available techniques are not sufficient to obtain reliable measurements, the user will be informed and advised of any research required and the estimated cost. The attainment of statistical control is a prerequisite for reporting any data. Any deviations from SOPs must be supportable and documented in the case narrative in the final report to the client.

Existing SOPs must be reviewed and updated as required but at a minimum of once per year.

The QAO will maintain an index of all SOPs in use.

RCRA has proposed in SW846, 3rd Edition, 1st Update, a standardized 10 element format for all revised RCRA methods. In light of RCRA's new requirements, the following SOP format should be employed when new measurement SOPs are written or when major rewrites are made to SOPs.

All SOPs should provide complete documentation as to how each sample was measured for each parameter. This formal documentation in the form of sample preparation logs, standard preparation logs, instrument logs, corrective action reports/case narrative reports (CAR/CNRs), Quality Control Approval Reports (QCARs), and similar logs must provide an immediate, complete and long-term description of how samples were measured and what problems were encountered. It is the laboratory policy to retain laboratory measurement records for a minimum period of five years. It is very

important that all measurement records be well organized and that they provide a complete description of how each sample was measured.

Of special importance in writing good SOPs is that each SOP meet the regulatory requirements of both the method and of any governing contract(s). It is important that IEA, Inc.-CT have SOPs prepared and ready to use for each of the environmental regulations which we support. This means that some groups may have four or five SOPs to cover one type of measurement (all five SOPs could be printed separately or compiled into one SOP).

New SOPs are developed by line supervisors and/or their designated staff when any sampling, or analytical procedure is recognized to be repetitive and routine. The Quality Assurance (QA) Officer will be available to assist in the development of SOPs.

Newly developed SOPs shall be reviewed by the analytical group, the group leader and by laboratory management and contain a sign off page.

The following outline lists some of the necessary elements that should be covered in most measurement SOPs. SOPs should cover each element. If a listed element in the outline does not apply, then the element should still be listed with the additional descriptor, "Not Applicable".

1) Introduction

<u>IEA, Inc. Policy on Business Ethics and Conduct</u>

State in the beginning of each SOP that "It is the intent of this procedure to comply with the IEA business ethics policy". Each member of the group/section should read and understand this ethics policy statement. It is the responsibility of all division managers to communicate and train employees about the requirements of the IEA, Inc. Policy on Business Ethics and Conduct to ensure compliance with this policy. A copy of the IEA Ethics Policy is in Appendix A of this QAPmP.

2) Scope and Application - Generally describe the SOP and its general application.

   a) Other related SOPs and analytical methods
   b) Water samples
   c) Soil/sediment samples

3) Summary of Method - Summarize the method in one paragraph.

4) Interferences - Describe any interferences to the method.

5) Apparatus and Materials - List in detail.

6) Reagents

   a) Inventory of Reagents, Standards and Solvents

   It is laboratory policy that each measurement group set up and maintain a system to inventory all standards, reagents and solvents used

in measurements. The inventory system should record the date the item was received and the lot/batch number. A unique IEA inventory number should be given to each item in the inventory. A set of index cards or a computer spread sheet (with hard copy and magnetic backup) may be used to maintain inventory records and updates. Each inventoried item should be marked with the date of receipt and an inventory record number. Self-sticking sample seals may be used as labels. The inventory system should also provide for documentation of the date the material was opened and used up. It is the section/group leader's responsibility to maintain an inventory system of reagents, standards and solvents.

b) Quality of Solvents, Reagents and Standards

Traceability of Calibration Standards - It is laboratory policy to purchase standards for organics and inorganics measurements which have been previously traced to reference standards.

If traceable standards are not available, then the measurement group traces the non-certified standard to EPA or NBS reference standards or other certified standards.

Assay of Purity - The chemical purity of the reagents and chemicals should be specified in the SOP. Any assays of materials should be kept in a central file.

Recordkeeping Requirements - Strict control of reagents used in laboratory operations shall be maintained to minimize contamination or degradation. Dilutions and solutions prepared from them will be initialed, dated and clearly labeled as to content. All reagents will be appropriate to the operation performed. A record will be maintained of all reagents used. Laboratory records should be kept organized and accessible for five years (minimum).

7) Sample Collection, Preservation and Handling

a) Preservation Requirements and Procedures - Each measurement SOP should clearly specify the preservation and holding time requirements of the regulatory method. Specify a system in the SOP to ensure that preservation and holding time requirements are met and documented. Corrective action reports should be used to document all preservation blank problems, contamination, etc.

b) Regulatory Holding Times - Clearly list all holding times for environmental samples and extracts.

For samples:

- water samples
- soil/sediment samples
- other matrixes

For extracts and concentrates

8) Procedure

a) Safety - General safety rules as outlined in the IEA Health and Safety Manual shall be followed. Use of gloves, eye protection and lab coats is required for work with dangerous chemicals. Hazardous materials with high vapor pressures should only be opened under hoods.

The responsibility of following good safety practices is everyone's. No employees should be using hazardous chemicals without proper training and safety equipment.

b) Sample and Data Chain of Custody Procedures - Upon receipt of the samples in the laboratory the sample custodian and the sample control group are responsible for obtaining all necessary shipping documentation and verification of all data entered in the laboratory sample custody records.

The specific procedures and requirements for receiving samples are specified in the SOP for sample control. In general the group signs and dates all shipping records, verifies the samples are in good condition and properly preserved, verifies the field chain of custody documents (if present) are in order, and assigns laboratory sample numbers to the incoming samples and stores the samples according to the requirements of the analytical protocols (refrigeration).

Each measurement SOP should specify procedures for maintaining sample and data chain of custody records. It is the responsibility of the group leader to ensure that chain of custody records are signed and maintained.

Any and all deviations from established laboratory chain of custody procedures must be noted on the CAR/CNRs and also documented in the case narrative for the job.

c) Sample Preparation - Each and every sample received by the laboratory will be handled in a conscientious manner. All aspects of sample preparation will be performed according to an approved SOP. The integrity of each sample shall be maintained through appropriate chain of custody procedures. Any limitations on analytical results due to the sample will be specified in the case narrative. All preparation methods shall include a QCAR which states the minimum quality control requirements of the SOP.

d) Sample Screening

e) Standards Preparation Log - It is the laboratory policy to provide full and complete documentation on the use and composition of all standards used for preservation or measurement or spiking of all environmental samples. This includes lot numbers of solvents, reagents, and standards used and the date and initials of the analyst who prepared the standard.

Specify instructions in the SOP (or a separate SOP) on the maintenance of a standards preparation log notebook. Lot numbers, weights, volumes and the initials of the analyst should be recorded. Assign an IEA preparation code number to each and every standard prepared including dilutions of standards. Use the following labeling convention: group:date:page number (of standards prep book).

f) Calibration - Each analytical method has its own set of calibration procedures and verification. Each measurement SOP must specify calibration requirements of the method. For specific procedures, each SOP must be consulted.

If the measurement requires the use of RCRA procedures, then the following general guidelines apply:

- Volatile Organics (VOA) GC/MS - Instrument tuned to BFB. 5-point initial calibration verified every 12 hours per SW846/CLP protocols.

- Semi-Volatile Organics (GC/MS): Instrument tuned to DFTPP. 5-point initial calibration verified every 12 hours per SW846/CLP protocols.

- Organochlorine Pesticides/PCB's (GC/ECD): Calibration per SW846/CLP protocols. Verification every 10 samples. Dual column confirmation.

- Metals (ICP, GFAAS and FAAS): Calibration per SW846/CLP protocols. Verification every 10 samples.

- Miscellaneous: Calibration and verification per method.

g) Measurement of Samples - It is laboratory policy that all measurements be made using appropriate SOPs. All SOPs shall be in writing and available for review. Any significant changes to or deviations from a SOP shall be documented in case narratives and include the reasons for any changes.

Laboratory measurement SOPs are based upon the following regulatory methods:

- Test Methods for Evaluating Solid Wastes, RCRA SW846 Methods (both 2nd and 3rd editions).

- Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020. Revised March, 1983.

- USEPA "Contract Laboratory Program Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration," SOW 7/88, Revisions 2/89 and 6/89.

- USEPA "Contract Laboratory Program Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration," SOW 2/88.

- Methods for the Determination of Organic Compound in Finished Drinking Water and Raw Source Water, EPA EMSL/CI. September, 1986.

h) Quantitation of Results - The SOP should clearly state the requirements of the method concerning the calculation of results. Provide examples as to how results are determined.

i) Qualitative Decisions (if any) - SOPs that require the identification of Tentatively Identified Compounds (TICs) must specify the EPA CLP procedures that must be followed in order to properly identify non-target compounds. CLP SOW guidelines for the identification of TICs are currently being evaluated by EPA. SOPs must require that the current CLP guidelines be followed. The QAO will audit TIC data for conformance to these written guidelines.

9) Quality Control

a) Quality Control Approval Reports (QCARs) - The laboratory group and section leaders are responsible for the data their groups generate. As such, all data must be reviewed and approved prior to release. This is documented in Quality Control Approval Reports (QCARs) which must accompany each data package as it is submitted to the data management group.

The laboratory Quality Assurance Officers will periodically examine data packages at random to ensure that all QCARs are present and to ascertain that the data package meets the requirements as stated in the SOP. These findings are transmitted to laboratory management via Monthly QA Progress Reports (MQAPRs).

b) Required Method QC Limits - Each SOP explains the type and frequency of quality control checks. This includes such items as analysis of EPA reference standards, matrix spikes, laboratory duplicates, blanks, the use of internal standards and surrogate spikes, etc. In general all calibration are checked before an analysis can begin. If the analytical system does not pass the initial QC limits, then the system is determined to be out of control, and cause of the problem is determined before measurements can continue. Once the problem is corrected, QC measurements are repeated to verify the calibration. If the system is still out of control, the instrument is re-examined until the problem is corrected. The following are examples of typical method QC items.

- Tuning and/or Calibration of Instrument
- Other Types of Calibration

- Laboratory Reagent Blank (LRB) - The laboratory measures a reagent blank sample at the frequency required by the method. These LRBs must be within required QC limits before sample analysis can begin.
- Internal Standard Precision
- Surrogate Spike Analysis - Surrogate spikes are added to all organics samples prior to extractions or analysis. Results of these are checked to verify the recoveries meet the requirements of the SOP. If the recoveries are out, the sample is reanalyzed to prove that the system is in control.
- Laboratory Fortified Blank (LFB)
- Laboratory Fortified Matrix Sample (LFM) - In general the laboratory performs a matrix spike/matrix spike duplicate for organic QC and a duplicate/matrix spike for inorganic QC. The results of these analyses are used to generate control charts to monitor the precision and accuracy of each parameter analyzed.
- Quality Control Sample (QCS)
- Laboratory Duplicates (LD1 and LD2)

c) Preventive Maintenance of Equipment - The policy of the laboratory is to have service contracts for all of its major instruments. These contracts provide routine preventive maintenance according to the manufacturer's requirements. Additionally the laboratory maintains an inventory of expendable parts and supplies to minimize downtime and to allow laboratory personnel to make minor repairs if necessary.

Each measurement SOP must list the preventive maintenance schedule for each instrument which is to be followed by in-house and extramural repair contractors. In addition, each measurement group must maintain a log of all in-house and extramural preventive maintenance activities.

10) Method Performance

a) Regulatory Requirements - Each regulatory method requires different approaches in determining method performance. SOPs must state the basis for the measurement and documentation of method performance for each method of analysis.

The laboratory is constantly assessing the changing Method Performance requirements of EPA regulations. For example, proposed drinking water organics methods may require a new approach to mandatory quality control. These new method performance requirements may require new software and computer hardware to meet the mandatory QC requirements of the regulatory methods.

b) Method Detection Limits (MDLs) - It is laboratory policy to determine MDLs for all methods prior to implementation of SOPs. Requirements for the determination of MDLs are specified in each regulatory method.

c) Accuracy of the Method - In order to routinely assess the precision and accuracy of the data generated, the laboratory performs monthly statistical analysis of the spike and spike duplicate data as part of our QA program. These results are used to generate control charts based upon the EPA Handbook of Analytical Quality Control for Environmental Laboratories. These charts allow for trend analysis to identify potential problems prior to their occurrence.

d) Precision of the Method

11) Data Reduction and Data Handling

a) Recordkeeping Procedures - It is the responsibility of all members of the laboratory to maintain complete records of all operations performed. All records shall be neat and organized. All laboratory records are the property of the laboratory and shall not be removed from the premises without permission from supervisors. All records are considered confidential and shall be safeguarded. Unauthorized changes, loss or destruction of records can be grounds for dismissal from the laboratory. Consult the IEA, Inc. Ethics Policy regarding integrity of data and employee conduct.

All measurement data will be recorded in logbooks or on preprinted log sheets in permanent ink. Transcriptions will be avoided whenever possible. The record will reflect the measurement performed and all appropriate details for conclusions related to the measurement. The record shall be signed and dated by the individual performing the measurement of the day the measurement is performed. Corrections shall be made by drawing a single line through the error, and initialing and dating the error.

As described above, all SOPs should provide complete documentation as to how each sample was measured for each parameter. SOPs should not permit measurement data or QC results to be routinely recorded in personal lab notebooks. Measurement records must be recorded in preprinted record logs or preprinted measurement logs. This policy will facilitate the organization and archiving of all laboratory data for future reference.

All injection forms, instrumentation forms, sample prep forms and QC forms which are used to process samples and measurement results should be described and attached to the SOP. The SOP should specify where these records and forms are cataloged and stored.

b) Data Validation - At a minimum, all data will be subject to supervisory review. Sensitive data requires higher level review and release. All releases must be in writing. Oral preliminary releases are prohibited unless prior permission of the

appropriate supervisor(s) is granted and provided that they are subsequently followed by written confirmation of review results.

c) Delivery Times for Reporting Results

d) Preparation of Final Report - Each analytical group in the laboratory is responsible for generating the data for all analyses the group performs. In general the data must first meet all the specific QA/QC associated with the SOP that was used for the analysis prior to any release of the data. The analytical group leader (supervisor) is responsible for the final verification of the data from the analysis.

The laboratory employs a system of QA sign-off sheets called Quality Control Approval Reports (QCARs), where each analyst must sign off that their respective part of the analysis is complete and meets the QA/QC requirements of the governing SOP. QCARs are signed and placed in each job folder along with any CAR/CNRs which detail any problems which were encountered in the measurement of samples. Any deviations in SOPs are noted on CAR/CNRs and explained in the case narrative which is incorporated into the final report. The group leader has final sign-off responsibility on the QCAR and is responsible for assuring the overall quality of the data.

12) Special Procedures

13) Use of Other Required SOPs

14) Documentation of Problems

a) Corrective Action Reports - Include and attach to each measurement SOP a copy of the most recent revision of the IEA, Inc./CT Corrective Actions Report/Case Narrative Report (CAR/CNR) attached). Provide instructions as to when a CAR/CNR should be completed. The CAR/CNR provides a routine written communication vehicle to describe most types of problems which may occur throughout the laboratory. In many cases, CAR/CNRs should be used instead of memos or notes. Most problems described in case narratives should be supported by a CAR/CNR. Each group/section may also have additional unique CARs which are specific for the group/section.

The CAR/CNR system requires that the provider of the corrective action (solution to the problem) sign and date the CAR/CNR and send secondary copies of the solution to the problem back to the originator of the CAR/CNR and to the QAO.

The system requires that the originator of the CAR/CNR be the person who is responsible to see that a solution is found to the documented problem. Each originator of CAR/CNRs will report to the QAO if corrective actions have not been taken by the designated provider of solutions.

Under the CAR/CNR system, the QAO will monitor and log the progress of CAR/CNRs and will report in his/her Monthly QA Progress Report the status of major corrective actions taken in the past month. It is the QAO's responsibility to see that laboratory problems are documented and solved in a timely manner.

b) Case Narratives - It is laboratory policy that any and all problems related to client samples and the measurement of client samples be documented in the case narrative of the final laboratory report which goes to the client. The mechanism for documenting problems which should be included in the case narrative are described above in Section a (above). It is the responsibility of the data management group to see that information on CAR/CNRs be included in the final case narrative.

15) References for SOP

5.0 THE AUDIT/REVIEW PROGRAM

5.1 Categories of Audits and Reviews - EPA has classified audits of a QAPmP into four categories. According to NPO and ORD QAPP Guidance, Quality Assurance Management Staff, USEPA, September 1987, audits are classified into the following areas:

- Management System Reviews (MSRs)
- Audits of Data Quality (ADQs)
- Technical Systems Audits (TSAs)
- Performance Evaluations (PEs)

5.2 Management System Reviews (MSRs) - Management System Reviews (MSRs) "assess the effectiveness of the implementation of the approved QAPmP(s). MSRs evaluate a specific group's QA program associated with environmental data collection activities to either affirm or to identify areas where additional attention would bring significant benefits". MSRs have been historically a review of one's organizations QA management system. This type of review of IEA, Inc.-CT's QA management system by IEA, Inc.-CT will continue, however, the laboratory will may have MSRs conducted by outside organizations and contractors.

5.3 Audits of Data Quality (ADQs) - ADQs are normally conducted by the end user of the data to determine if specific data of QAPjP meets the requirements of the data quality objectives (DQOs).

At IEA, Inc.-CT, ADQs are conducted by both external auditors and internal auditors. The results from external ADQs is usually a report submitted to the laboratory with a request for corrective actions. The QAO reviews the external ADQs and issue requests for corrective actions of deficiencies. The QAO will look for both technical and managerial causes of QA deficiencies and will make recommendations to the Laboratory Operations Manager (LOM) for corrective actions. It is the responsibility of the LOM to implement these corrective actions.

Internal ADQs are normally initiated by a QAO. The results of the audit are reviewed by the QAO and corrective actions similar to those described above are submitted by the QAO to the LOM for implementation.

5.4 Technical Systems Audits (TSAs) - According to EPA QAMS[2] "Technical Systems Audits (TSAs) focus on the actual quality control and environmental measurement data collection systems. A TSA entails an examination of calibration records, sampling and measurement procedures, general laboratory cleanliness, support systems, equipment and facilities, maintenance and repair records, control charts, etc. TSA auditors must be competent scientists who are familiar with the particular data collection technology and quality control procedures".

At IEA, Inc.-CT, TSAs are performed by both external and internal auditors. Externally auditors are from EPA, state and private client organizations.

The QAO may conduct a TSA on any project or group in the laboratory at any time. Internal TSAs by the QAO are essential to the QA program of the laboratory. The results of any deficiencies noted in all TSAs must be addressed by the LOM and his staff.

5.5 Performance Evaluation Audits (PEs) - According to EPA QAMS[2], "A Performance Evaluation (PE) is the means of evaluating the performance of laboratory technicians and the instrumentation or analytical systems on which they work. A PE is accomplished by providing PE samples containing specific pollutants (in appropriate matrices) unknown to the technician in their concentration and/or identity. PEs are implemented by the QAOs, project officers (POs) or laboratory management".[1]

At IEA, Inc.-CT, PEs are conducted routinely by a number of external organizations. The QAO may submit internal PEs to any group in the laboratory at any time. The QAO routinely submits double-blind PE samples to each measurement group during each quarter of the year. Internal PEs by the QAO are essential to the QA program of the laboratory. The results of any deficiencies noted in all PEs must be addressed by the LOM and his staff. QAOs routinely submit additional PE samples to groups who show deficiencies in measurement accuracy or quality of data.

5.6 Management System Reviews (MSRs)

External Audit Program - On an annual basis, IEA, Inc. will conduct audits of all groups at IEA, Inc.-CT. The results of this comprehensive QA audit will be reported as an External Management Systems Audit Report (EMSAR) to corporate and laboratory management.

The above audit will review the entire, current QAPmP at IEA, Inc.-CT and make recommendations to corporate and laboratory management in the following areas:

. The quality of the existing QAPmP.

- Current procedures for developing, revising and approving SOPs.
- Overall data quality and technical capability.
- Procedures and criteria for designing and conducting audits.
- Recommendations for changes to the IEA, Inc.-CT QAPmP.

5.7 Audits Program - The results of all external audits will be reported by the QAO in their monthly QAPR. These would include all state, federal and private client on-site inspections.

5.8 Internal Audits of Data Quality - On a regular basis the QAO will review data to check for compliance to SOPs. Additionally the QAO will review SOPs to ensure they meet the requirements of the methodologies and applicable regulations.

5.9 Internal Technical System Audits - On a periodic basis the QAO will perform unannounced TSAs. The purpose of this will be to determine if the laboratory staff is following the SOPs, if the SOPs need revision, proper documentation through corrective action reports, case narratives, etc. and conformance to identified critical control points.

5.10 Internal Performance Evaluation Audits - On a quarterly basis the QAO will submit double blind QC samples to the laboratory. The purpose of this will be to check the accuracy of results, assess data quality, assess documentation and completeness of data reporting.

5.11 Audit Reports

Monthly Quality Assurance Progress Report - By the 15th of each month the QAO will issue a report to corporate and laboratory management summarizing all QA/QC activities from the previous month.

6.0 QUALITY ASSURANCE AUTHORITIES AND RESPONSIBILITIES

6.1 Organization Chart - The IEA, Inc.-CT Functional Organizational Chart is also attached. This chart indicates the chain of command for each of the managers and groups at IEA, Inc.-CT.

6.2 Facilities and Equipment - A list of facilities and equipment is attached in Appendix A.

7.0 TRAINING

7.1 Technical Training - All laboratory personnel must have adequate education, training, and experience to carry out their responsibilities at IEA, Inc.-CT. To ensure that each laboratory member has acquired both sufficient and applicable knowledge to carry out their tasks, the QAOs and the Laboratory Management will periodically review the training needs of the staff and make recommendations for any additional training needs.

8.0 REFERENCES

1) NPO and ORD QAPP Guidance, Quality Assurance Management Staff (QAMS), USEPA, September, 1987.

2) QAMS-004/80, Guidelines and Specifications for Preparing Quality Assurance Program Plans. Quality Assurance Management Staff (QAMS), USEPA, 1979.

ATTACHMENTS

APPENDIX A

IEA, Inc. Policy Statement on Business Ethics and Conduct

IEA, Inc.-CT Organization Chart

IEA, Inc.-CT Floor Plan

IEA, Inc.-CT Capital Equipment Inventory

IEA, Inc.-CT Corrective Action Report/Case Narrative Report (CAR/CNR)

APPENDIX B

Laboratory Detection Limits

APPENDIX A

IEA, Inc.-Connecticut

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INDUSTRIAL & ENVIRONMENTAL ANALYST'S, INC.

ETHICS POLICY

The management of Industrial & Environmental Analyst's, Inc. (IEA) recognizes our responsibility to clients and fellow employees to ensure that fair and ethical business practices are followed at all facilities.

Management is fully aware that any ethical violations severly damage the reputation of the entire organization as well as individual employees at all levels. Therefore, it is imperative that high ethical standards be maintained at all times by all employees.

The management and staff are committed to maintaining a carefully controlled analytical environment which assures the consistent generation of accurate data which meets the data quality objectives of our clientele.

The following represents the IEA ethics policy which has been adopted to clearly identify the corporate position on ethical practices. Failure to comply with this policy cannot and will not be tolerated.

The Company and All its Employees will:

- Fully comply with all applicable federal, state, and local laws and regulations.
- Produce analytical products that are accurate, defensible and which represent sound professional judgement at all times.
- Provide employees with guidance and an understanding of the ethical and quality standards required in the environmental industry. In this regard, all employees should feel free to identify any ethical misconduct without fear of retribution. Any employee involved in any form of ethical misconduct will be subject to immediate disciplinary action including potential termination of employment.
- Present services to clients in a confidential, honest and forth-right manner and strive to deliver quality products at a fair price.

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INDUSTRIAL & ENVIRONMENTAL ANALYST'S, INC.

ETHICS POLICY -Continued...

- Treat employees equitably by compensating them fairly, acknowledging their scientific contributions, and providing them opportunities for professional growth and development.

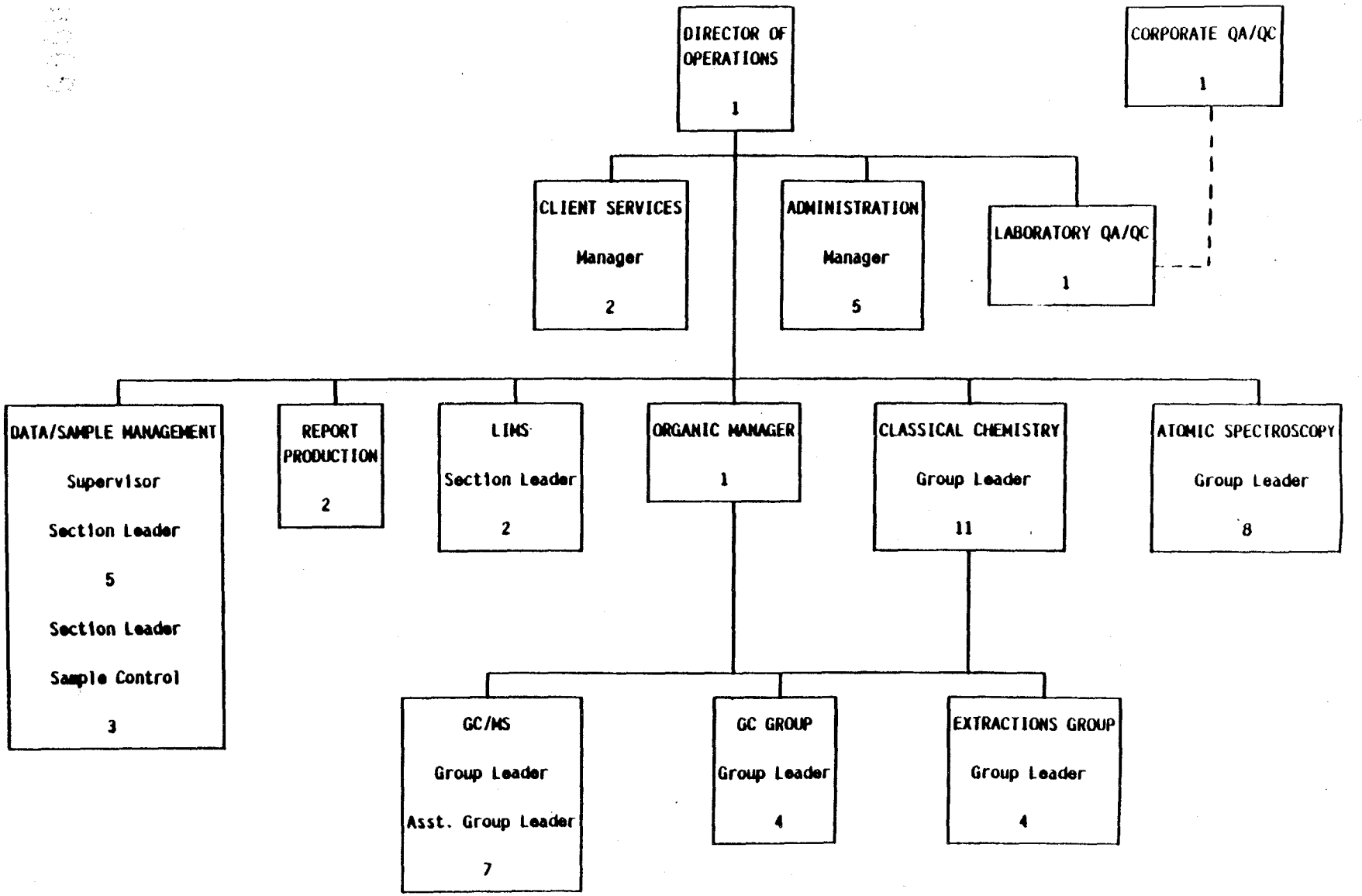
- Offer employment opportunities to qualified candidates regardless of their race, creed, color, sex or age.

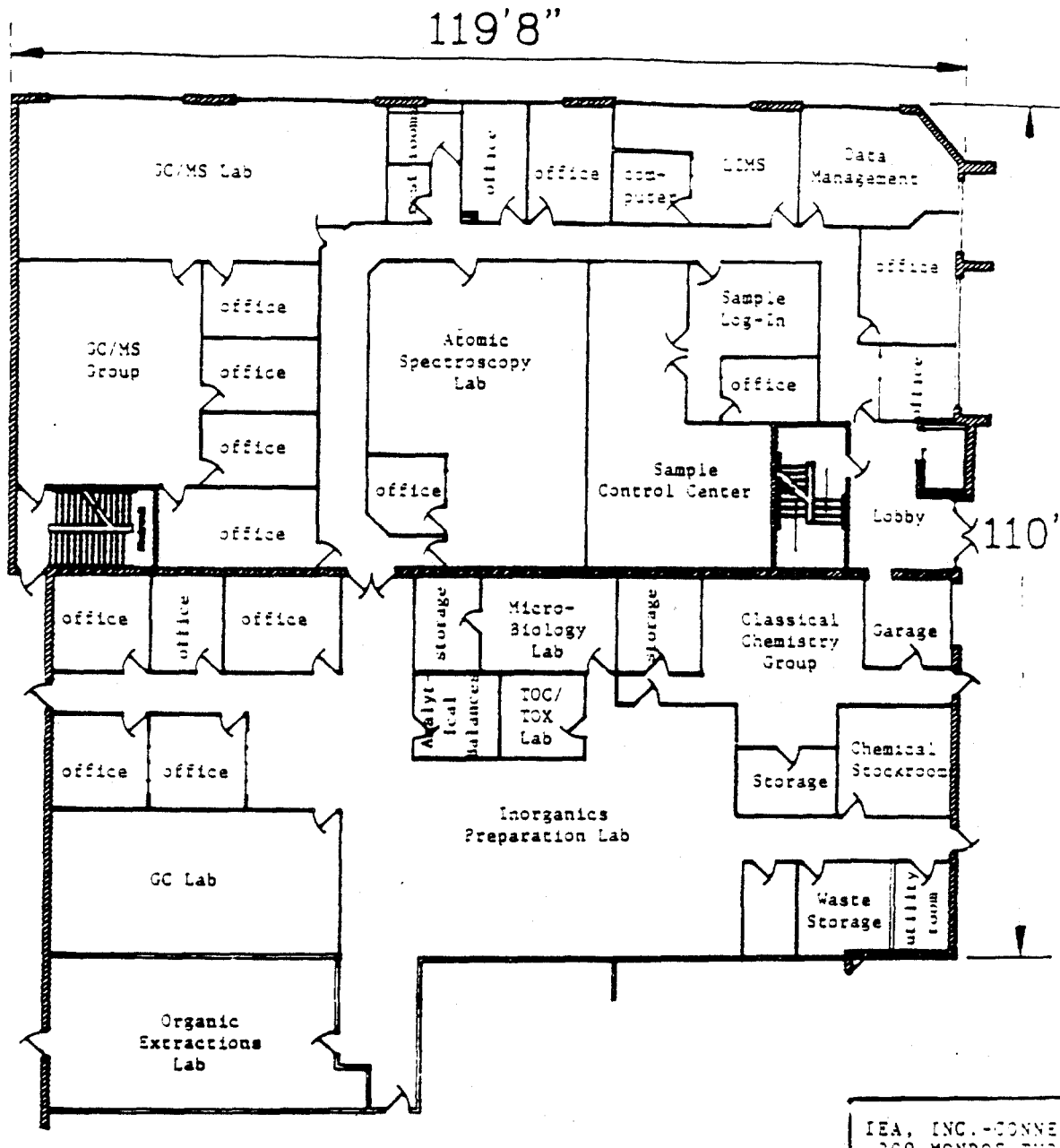
- Be a responsible corporate citizen of the community by operating in an environmentally sound manner at all times.

- Maintain all facilities in a safe amd professional manner through maintenance of a safety awareness program and providing the necessary safety equipment and training to protect all employees from preventable injury and chemical exposure.

IEA/CT, INC.

ORGANIZATION (BY FUNCTION) 1/91





IEA, INC.-CONNECTI
200 MONROE TURNPI
MONROE, CT
FLOOR PLAN

300821

IEA, INC. - CONNECTICUT

EQUIPMENT LIST

Equipment Name	Manufacturer	Model Number	Serial Number
Spectrophotometer, UV-VIS	Perkin-Elmer	Hitachi 200	522-5
IR-Spectrophotometer	Perkin-Elmer	1310	134423
Turbidimeter	Hach Company	2100A	851017142
TOC Analyzer	Xertex-Dohrmann	DC-80	HF2029
TOX Analyzer	Xertex-Dohrmann	MC3 A,B	MF 2106
Fluorometer	Sequoia-Turner Corp.	112-003	D 01491
pH/ISE Meter	Orion	SA 720	SR45A
Conductivity Meter	Cole-Parmer Instrument	1484-20	1421
Flash Point Apparatus	Precision Scientific	Pensky-Martin	10 Au-12
Oven	Fisher Scientific	55G	291
Incubator	Blue M Electric	100 A	IN1-1362
Bio Refrigerator	Frost Queen	R20/L	00029
Centrifuge	Garver Manufacturing	549	10883
Water Bath	Blue M Electric	MW-1220	MX-2520
D.O. Meter	YSI	51A	0241
Autoclave	Market Forge	STM-E	034200
Gas Chromatograph	Perkin-Elmer	8320	83N546502
Gel Permeation Chromatograph	ABC	1002B	7323
Refrigerator	WW	4EF	F3976
Oven	ASP	D 1142	144011
Oven	ASP	D 1162	149010
Sonicator	Sonics & Materials	SM500	6892
Sonicator	Tekmer	TM500	7264
Mercury Analyzer	Spectro-Products	HG4	4708
ICP-Sequential	Perkin-Elmer	6500	128238
ICP-Semiultaneous	Jarrell-Ash	JA61	67782
Flame AA	Perkin-Elmer	2380	126443
Furnace AA	Perkin-Elmer	Z3030	3131
Furnace AA	Perkin-Elmer	Z5100	130911
Furnace AA	Perkin-Elmer	Z5100 PC	135141
Computer	NEC	Power Mate2	77000690B
Gas Chromatograph	Hewlett Packard	5890	7518A05422
Gas Chromatograph	Hewlett Packard	5890	2728A14615
Auto Sampler	Hewlett Packard	7673A	2607A02433
Auto Sampler	Hewlett Packard	76732A	2441A03468
Mass Selective Detector	Hewlett Packard	5970	2513A00923
Mass Selective Detector	Hewlett Packard	5970	2716A10638
Computer Terminal	Hewlett Packard	150 II	2528A05525
Computer Terminal	Hewlett Packard	2623A	---
Computer Terminal	Hewlett Packard	150 II	2720Y05798
Computer Terminal	Hewlett Packard	150 II	2720Y03266

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Equipment Name	Manufacturer	Model Number	Serial Number
Scanning Interface	Hewlett Packard	59824A	---
Scanning Interface	Hewlett Packard	59824A	---
Tape Drive	Hewlett Packard	9144	---
Disc Drive	Hewlett Packard	7958	---
9 Track Magnetic Tape	Hewlett Packard	7970E	---
9 Track Magnetic Tape	Hewlett Packard	7970E	---
Computer	Hewlett Packard	HP1000A	---
Computer	Hewlett Packard	HP1000	---
Disc Drive	Hewlett Packard	7914	---
Disc Drive	Hewlett Packard	7914	---
P&T	Tekmar	LSC-2	227
P&T	Tekmar	4000	192
P&T	Tekmar	4000	398
P&T	Tekmar	14-2000-000	88068001
P&T	Tekmar	LSC-2	1324
P&T	Tekmar	ALS	679
P&T	Tekmar	14-2962-200	88061015
P&T	Tekmar	ALS	494
P&T	Tekmar	ALS	1068
GC/MS	Hewlett Packard	5995B	2217A00358
GC/MS	Hewlett Packard	5995C	2413A00659
GC/MS	Hewlett Packard	5995C	2413A00430
Terminal	Hewlett Packard	45849A	2530A13541
Terminal	Hewlett Packard	35751	2643A07666
CRT	Hewlett Packard	35731A	8633K26810
Printers (partial list)	Hewlett Packard	2934A	2635A32940
Printers	Hewlett Packard	2934A	2715A43948
Printers	Hewlett Packard	2225A	2512S30379
Printers	Hewlett Packard	2225A	2510S32359
Terminal	Hewlett Packard	35751	2630A06622
CRT	Hewlett Packard	35731A	8610K20516
Magnetic Tape Unit	Hewlett Packard	7970E	N/A
Scanning Interface	Hewlett Packard	59824A	N/A
Scanning Interface	Hewlett Packard	59824A	N/A
Cart. Tape Unit	Hewlett Packard	7914	N/A
5010 Auto Desorber	Tekmar	14-2150-000	133-GT
Cart. Tape Unit	Hewlett Packard	7914	N/A
GC Analyzer	Perkin-Elmer	Sigma 1	0815150019
GC data System	Perkin-Elmer	Sigma 1-data system	0815150010
Auto Sampler	Perkin-Elmer	AS100	94975
Satellite Integrator	Perkin-Elmer	SI316	N4397071
Satellite Integrator	Perkin-Elmer	SI316	316N671510
Integrator/Plotter	Perkin-Elmer	LCI-100	N431931C
Integrator/Plotter	Perkin-Elmer	LCI-100	P5490100

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Equipment Name	Manufacturer	Model Number	Serial Number
GC	Perkin-Elmer	Sigma 3	09331700213
AutoSampler	Perkin-Elmer	AS100 B	95234
Data Station	Perkin-Elmer	7500	901732
Printer	Perkin-Elmer	P-132	052597
Plotter	Perkin-Elmer	GP100	062005
GC	Hewlett-Packard	5890	2541A06301
GC	Hewlett-Packard	5890	2750A14340
GC	Hewlett-Packard	5730A	1609A00459
Autosampler	Hewlett-Packard	7673A-Dual Tower	2546A00709
Autosampler	Hewlett-Packard	7673A	2718A06503A
Integrator	Hewlett-Packard	3396A	2804A01106
Integrator	Hewlett-Packard	3393A	2332A00280
Integrator	Hewlett-Packard	3392A	2736A11930
Auto Analyzer	LaChat	Quikchem Ion Analyzer	---
GC	Varian Hall/PID	3300	---

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IEA, INC.-CONNECTICUT

Reported By: _____
Group
Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE REPORT

Client: _____ Job Number: _____ Sample Number(s): _____

Problem Reported By: _____

Category of Problem: _____
Poor Communication With ___. Sample Matrix Instruc.

Description of Problem: _____

_____
Use back of CAR/CNR or attached sheet if more space is required

_____

_____

_____

_____

_____

_____

Recommended Corrective Action(s): _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline For CAR: _____ Reply to Originator Required? [ ]

Actual Corrective Action(s) Taken: _____
Document Actual Corrective Actions On Thi.

_____
Form and Send a Copy of Completed CAR/CNR to QA Officer and Job Folder

_____

_____

_____

_____

_____
Signature and Date

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Initial Distribution: Lab manager, Marketing Manager, Branch Manager, QA Officer, Client Services, Job Folder, Other: _____

SECONDARY DISTRIBUTION: ORIGINATOR, QA OFFICER, OTHER: _____

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Document Problem and Corrective Action(s) In Case Narrative: [ ]

Attachments: Telephone Log, Services Change Request, Memo, Other:

APPENDIX B

IEA, Inc.-Connecticut

TABLE 1.0

VOLATILE TARGET COMPOUND LIST

Compound Name	CAS#	Precision	Accuracy	Low Level Water Limits (ppb) CRQL	MDL*
Chloromethane	74873	±37	99	10	3
Bromomethane	74839	±27	99	10	4
Vinyl Chloride	75014	±28	107	10	3
Chloroethane	75003	±23	97	10	3
Methylene Chloride	75092	±16	105	5	3
Acetone	67641	±64	106	10	3
Carbon Disulfide	75150	±108	138	5	1
1,1-Dichloroethene	75354	±16	109	5	2
1,1-Dichloroethane	75343	±16	109	5	1
1,2-Dichloroethene (total)	540590	±16	106	5	1
Chloroform	67663	±92	170	5	1
1,2-Dichloroethane	107062	±17	112	5	1
2-Butanone	78933	±29	102	10	3
1,1,1-Trichloroethane	71556	±17	86	5	3
Carbon Tetrachloride	56235	±17	103	5	1
Vinyl Acetate	108054	±4	90	10	1
Bromodichloromethane	75274	±18	97	5	1
1,2-Dichloropropane	78875	±18	97	5	1
cis-1,3-Dichloropropene	10061015	±39	122	5	1
Trichloroethene	79016	±15	104	5	1
Dibromochloromethane	1244812	±22	109	5	1
1,1,2-Trichloroethane	79005	±19	99	5	1
Benzene	71432	±15	98	5	1
trans-1,3-Dichloropropene	10061026	±26	128	5	1
Bromoform	75252	±17	102	5	1
4-Methyl-2-Pentanone	108101	±20	114	10	2
2-Hexanone	591786	±25	112	10	3
Tetrachloroethene	127184	±13	106	5	2
1,1,2,2-Tetrachloroethane	79345	±18	101	5	1
Toluene	108883	±10	102	5	1
Chlorobenzene	108907	±15	100	5	1
Ethylbenzene	100414	±13	98	5	1
Styrene	100425	±18	90	5	1
Xylene (total)	1330207	±24	97	5	2

Low Level Soil CRQLs and MDLs are the same as above.
Medium Level Soil CRQLs and MDSs are 125 times higher.

* The reference to the MDL calculation is Federal Register 40 CFR Part 136 Appendix B, October 26, 1984.

TABLE 2.0

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)*

	Semi-Volatiles	Case Number	Precision	Accuracy	Quantitation Limits (water ug/L a.g ** CRQL
1.	Phenol	108-95-2	±6	37	10
2.	bis(2-Chloroethyl)ether	11-44-4	±14	82	10
3.	2-Chlorophenol	95-57-8	±11	77	10
4.	1,3-Dichlorobenzene	541-73-1	±13	67	10
5.	1,4-Dichlorobenzene	106-46-7	±13	68	10
6.	Benzyl alcohol	100-51-6	±21	77	10
7.	1,2-Dichlorobenzene	95-50-1	±13	71	10
8.	2-Methylphenol	95-48-7	±11	72	10
9.	bis(2-Chloroisopropyl)ether	108-60-1	±14	81	10
10.	4-Methylphenol	106-44-5	±10	66	10
11.	N-Nitroso-di-n-dipropylamine	621-64-7	±14	81	10
12.	Hexachloroethane	67-72-1	±14	60	10
13.	Nitrobenzene	98-95-3	±12	81	10
14.	Isophorone	78-59-1	±14	87	10
15.	2-Nitrophenol	88-75-5	±11	83	10
16.	2,4-Dimethylphenol	105-67-9	±15	69	10
17.	Benzoic acid	65-85-0	±20	27	50
18.	bis(2-Chloroethoxy)methane	111-91-8	±13	87	10
19.	2,4-Dichlorophenol	120-83-2	±12	84	10
20.	1,2,4-Trichlorobenzene	120-82-1	±14	75	10
21.	Naphthalene	91-20-3	±12	77	10
22.	4-Chloroaniline	106-47-8	±36	65	10
23.	Hexachlorobutadiene	87-68-3	±15	67	10
24.	4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	±15	82	10
25.	2-Methylnaphthalene	91-57-6	±11	72	10
26.	Hexachlorocyclopentadiene	77-47-4	±11	28	10
27.	2,4,6-Trichlorophenol	88-06-2	±12	88	10
28.	2,4,5-Trichlorophenol	95-95-4	±14	90	50
29.	2-Chloronaphthalene	91-58-7	±10	83	10
30.	2-Nitroaniline	88-74-4	±16	91	50
31.	Dimethylphthalate	131-11-3	±23	49	10
32.	Acenaphthylene	208-96-8	±9	85	10
33.	2,6-Dinitrotoluene	606-20-2	±13	97	10
34.	3-Nitroaniline	99-09-2	±19	109	50
35.	Acenaphthene	83-32-9	±9	86	10
36.	2,4-Dinitrophenol	51-28-5	±30	74	50
37.	4-Nitrophenol	100-02-7	±20	22	50
38.	Dibenzofuran	132-64-9	±10	91	10
39.	2,4-Dinitrotoluene	121-14-2	±19	104	10
40.	Diethylphthalate	84-66-2	±21	80	10
41.	4-Chlorophenyl-phenyl ether	7005-72-3	±10	94	10
42.	Fluorene	86-73-7	±12	93	10
43.	4-Nitroaniline	100-01-6	±42	90	50
44.	4,6-Dinitro-2-methylphenol	534-52-1	±18	70	50

TABLE 2.1

TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)*(Continued)

	Semi-Volatiles	Case Number	Precision	Accuracy	Quantitation L (water ug/L a,b CRQL
45.	N-nitrosodiphenylamine	86-30-6	±15	99	10
46.	4-Bromophenyl-phenylether	101-55-3	±9	91	10
47.	Hexachlorobenzene	118-74-1	±10	93	10
48.	Pentachlorophenol	87-86-5	±19	68	50
49.	Phenanthrene	85-01-8	±10	90	10
50.	Anthracene	120-12-7	±9	87	10
51.	Di-n-butylphthalate	84-74-2	±12	88	10
52.	Fluoranthene	206-44-0	±13	88	10
53.	Pyrene	129-00-0	±13	91	10
54.	Butylbenzylphthalate	85-68-7	±15	93	10
55.	3,3'-Dichlorobenzidine	91-94-1	±6	68	20
56.	Benzo(a)anthracene	56-55-3	±11	91	10
57.	Chrysene	218-01-9	±11	95	10
58.	bis(2-Ethylhexyl)phthalate	117-81-7	±13	96	10
59.	Di-n-octylphthalate	117-84-0	±16	102	10
60.	Benzo(b)fluoranthene	205-99-2	±14	88	10
61.	Benzo(k)fluoranthene	207-08-9	±30	86	10
62.	Benzo(a)pyrene	50-32-8	±11	87	10
63.	Indeno(1,2,3-cd)pyrene	193-39-5	±13	98	10
64.	Dibenz(a,h)anthracene	53-70-3	±12	94	10
65.	Benzo(g,h,i)perylene	191-24-2	±13	94	10

a. Low soil/sediment contract required quantitation limits (CRQL and MDL's) for semi-v tile TCL compounds are 33 times the individual water CRQL and MDL.

b. Medium soil/sediment contract required quantitation limits (CRQL and MDL) for semi-v tile TCL compounds are 60 times the individual low soil/sediment CRQL and MDL.

* Specific quantitation limits are highly matrix dependent. The quantitation li listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitat limits calculated by the laboratory for soil/sediment, calculated on dry weight basis required by the contract, will be higher.

+ The reference to the MDL calculation is Federal Register 40 CFR, Part 136 Appendix October 26, 1984.

The reference to the Precision and Accuracy is SW846, 3rd Edition, Chapter One, Sect 1.1.8.

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Table 3.0

Instrument ID: GC4A/GC4B

Laboratory: TEA-CT - MDL

EPA PRIORITY POLLUTANT AND TCL PESTICIDES/PCB'S LIST AND MDL

All values are ug/L.

Compound	Replicate Analyses R_1	R_2	R_3	R_4	R_5	R_6	R_7	SD	MDL[1,2]
alpha-BHC	0.093	0.101	0.099	0.093	0.096	0.097	0.100	0.0032	0.010
beta-BHC	0.090	0.104	0.097	0.090	0.091	0.095	0.103	0.0059	0.019
gamma-BHC	0.074	0.083	0.080	0.074	0.080	0.079	0.082	0.0036	0.011
delta-BHC	0.089	0.100	0.095	0.090	0.090	0.093	0.097	0.0041	0.013
Heptachlor	0.088	0.095	0.092	0.088	0.090	0.092	0.093	0.0026	0.008
Aldrin	0.088	0.104	0.092	0.084	0.084	0.091	0.098	0.0073	0.023
4,4'DDE	0.105	0.118	0.118	0.102	0.101	0.106	0.110	0.007	0.022
Dieldrin	0.219	0.127	0.120	0.113	0.108	0.111	0.114	0.0396	0.124
4,4'DDD	0.437	0.445	0.445	0.449	0.455	0.455	0.455	0.0069	0.022
Endrin Aldehyde	0.082	0.091	0.092	0.094	0.098	0.096	0.091	0.0051	0.016
4,4'DDT	0.500	0.517	0.503	0.503	0.531	0.507	0.498	0.0117	0.037
alpha-Chlordane	0.498	0.485	0.515	0.503	0.495	0.482	0.495	0.011	0.035
gamma-Chlordane	0.511	0.500	0.533	0.507	0.504	0.487	0.509	0.014	0.044
Endosulfan I	0.102	0.138	0.156	0.115	0.117	0.120	0.149	0.0198	0.062
Endosulfan II	0.493	0.502	0.497	0.503	0.518	0.509	0.497	0.0085	0.027
Endosulfan Sulfate	0.590	0.616	0.630	0.597	0.610	0.605	0.590	0.015	0.046
Endrin	0.397	0.465	0.471	0.473	0.474	0.484	0.483	0.0302	0.095
Heptachlor Epoxide	0.103	0.143	0.141	0.111	0.107	0.117	0.127	0.0161	0.051
Methoxychlor	0.421	0.510	0.509	0.493	0.515	0.532	0.500	0.0357	0.112
Endrin Ketone	0.479	0.470	0.491	0.485	0.478	0.469	0.0490	0.0089	0.028
Toxaphene	0.784	0.8327	0.7776	0.8637	0.8236	0.7826	0.8226	0.0321	0.101
PCB-1016	1.558	1.383	1.492	1.463	1.520	1.697	1.452	0.0995	0.313
PCB-1221	1.655	1.307	1.342	1.618	1.411	1.311	1.326	0.1494	0.470
PCB-1232	1.451	1.553	1.548	1.437	1.406	1.384	1.325	0.0837	0.263
PCB-1242	1.483	1.514	1.440	1.467	1.552	1.520	1.440	0.0426	0.134
PCB-1248	1.442	1.465	1.619	1.508	1.656	1.544	1.514	0.0780	0.245
PCB-1254	1.287	1.054	1.244	1.368	1.332	1.665	1.260	0.1837	0.577
PCB-1260	1.280	1.411	1.694	1.358	1.628	1.413	1.256	0.1670	0.525

Table 3.1

[1] The reference to the MDL calculation is Federal Register 40 CFR Part 136 Appendix B, October 26, 1984.

The reference to the Precision and Accuracy is from SW 846 - 3rd Edition Chapter One Section 1.1.8

[2] Low and Medium soil/sediment MDL for Pesticides/PCB TCL compounds are 150 and 2400 times the individual low water MDL respectively.

Instrument ID: GC4A/GC4B **Laboratory: York/CT - MDL**

EPA PRIORITY POLLUTANT AND TCL PESTICIDES/PCB'S

Compound	Precision	Accuracy
alpha-BHC	±10	107
beta-BHC	±17	109
gamma-BHC	±15	100
delta-BHC	±15	101
Heptachlor	±16	84
Aldrin	±38	70
4,4'DDE	±35	91
Dieldrin	±21	105
4,4'DDD	±41	104
Endrin Aldehyde	±5.5	92
4,4'DDT	±36	109
alpha-Chlordane	±14	108
gamma-Chlordane	±11	115
Endosulfan I	±28	103
Endosulfan II	±26	92
Endosulfan Sulfate	±16	115
Endrin	±15	103
Heptachlor Epoxide	±11	115
Methoxychlor	±23	94
Endrin Ketone	±1.7	96
Toxaphene	±4	81
PCB-1016	±6.6	151
PCB-1221	±11	142
PCB-1232	±5.8	144
PCB-1242	±22	117
PCB-1248	±5.1	154
PCB-1254	±14	132
PCB-1260	±18	114

Table 4.0

ICAP INSTRUMENT DETECTION LIMITS (IDL'S)

ELEMENT	IDL(ug/l)	ROUNDED IDL (ug/l)	CRDL(ug/l)
Ag	3.4	4.0	10.0
Al	104	104	200.0
Ba	1.3	2.0	200.0
Be	0.2	1.0	5.0
Ca	14.0	14.0	(1000.0) 5000.0
Cd	0.9	1.0	5.0
Co	1.6	2.0	50.0
Cr	2.2	3.0	10.0
Cu	3.2	4.0	25.0
Fe	57.3	58.0	100.0
K	449.0	449.0	(1000.0) 5000.0
Mg	17.6	18.0	(1000.0) 5000.0
Mn	0.8	1.0	15.0
Na	73.5	74.0	(1000.0) 5000.0
Ni	4.1	5.0	40.0
Pb	14.4	15.0	(100.0)
Sb	17.8	18.0	60.0
Se	35.6	36.0	(500.0)
V	1.9	2.0	50.0
Zn	2.8	3.0	20.0
Ti	1.8	2.0	(20.0)
Mo	3.0	3.0	(20.0)
Sn	7.6	8.0	(100.0)
As	18.0	18.0	(300.0)

CRDL's in parenthesis are either normally run by graphite furnace, or are not typically reported by the metals department and represent the detection limits employed in reporting these parameters.

Table 4.1

FURNACE INSTRUMENT DETECTION LIMITS

	PARAMETERS in ug/l			
INSTRUMENT	As	Se	Tl	Pb
Perkin Elmer 3030	---	---	(1.6)2.0	(1.0)1.0
Perkin Elmer 5100	(0.4)1.0	(1.1)2.0	(0.6)1.0	(0.9)1.0
Perkin Elmer 5100N	(0.4)1.0	(0.8)1.0	(0.7)1.0	(0.7)1.0

Values in parentheses are the unrounded values. These are used whenever NYSDEC '87 protocols are requested. However whenever CLP 7/88 protocols are requested, the rounded values are employed.

Table 4.2
IEA INC.-CT

ELEMENT	PRECISION*	ACCURACY**
ALUMINUM	7.7	101.2
ANTIMONY	13.3	94.9
ARSENIC	19.1	90.4
BARIUM	6.1	98.7
BERYLLIUM	8.2	98.9
CADMIUM	7.9	99.5
CALCIUM	8.6	93.3
CHROMIUM	7.1	96.8
COBALT	7.3	94.4
COPPER	8.5	95.8
IRON	7.8	107.9
LEAD	13.6	98.0
MAGNESIUM	7.5	91.3
MANGANESE	10.3	97.1
MERCURY	14.1	101.9
NICKEL	6.2	96.8
POTASSIUM	2.4	98.3
SELENIUM	9.7	96.8
SILVER	6.5	98.9
SODIUM	1.7	97.7
VANADIUM	6.2	95.2
ZINC	6.1	99.5

* PRECISION = %RELATIVE STANDARD DEVIATION
%RSD = (RSD/AVERAGE % RECOVERY)*100

** ACCURACY = AVERAGE SPIKE RECOVERY

TABLE 5.0

COMMON CLASSICAL CHEMISTRY DETECTION LIMITS

All concentrations are mg/L.

Parameter	Method	RDL	MDL	Precision[1]	Accuracy[2]
Ammonia-Nitrogen	350.1	0.04	0.01	±8.2	91.4
Chloride	325.1	3.0	0.43	±7.6	92.6
Chemical Oxygen Demand	410.1	10.0	4.7	±13.3	94.1
Cyanide	CLP	0.010	0.008	±5.1	97.8
Cyanide	335.2	0.005	0.001	±10.1	98.3
Fluoride	340.2	0.10	0.02	±14.0	94.5
Nitrate-Nitrogen	353.2	0.10	0.02	±9.8	97.7
Phosphorus, total	365.2	0.15	0.06	±13.1	95.8
Sulfate	375.3	10.0	6.70	±3.9	103.2
Total Organic Carbon	415.2	0.50	0.28	±1.7	105
Total Organic Halides	450.1	0.010	0.008	±9.2	105
Phenols	420.2	0.005	0.005	±4.6	96.1
Total Kjeldahl Nitrogen	351.2	0.100	0.058	±3.8	97.4

[1]Percent RSD
[2]Average spike recovery (percent)

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Monroe, Connecticut 06468
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STANDARD OPERATING PROCEDURE

GLASSWARE/PLASTICWARE CLEANING

AS:071587:2

300836

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

STANDARD OPERATING PROCEDURE
GLASSWARE/PLASTICWARE CLEANING

Abstract

In order to assure that all samples are prepared free from contamination, all beakers, volumnetrics and plasticware are cleaned according to a prescribed procedure.

INTRODUCTION

This procedure outlines the steps necessary in order to wash all associates glassware and plasticware for sample preparation in the Atomic spectroscopy Group to insure that it is free from contamination.

It is the intent of this procedure to comply with the IEA business ethics policy.

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to all glassware necessary in the standard and sample preparation of both water and soil samples for metals analysis.

2.0 SUMMARY OF METHOD

2.1 The glassware is washed with a phosphate-free detergent and then acid washed.

3.0 INTERFERENCES-N/A

4.0 APPARATUS AND MATERIALS

4.1 Pertinent glassware for the specific procedure.

5.0 REAGENTS

5.1 Pertinent glassware for the specific procedure.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE-N/A

7.0 PROCEDURE

7.1 All graduated cylinders, beakers and volumetric flasks are initially washed thoroughtly with Liquinox and then rinsed with tap water to remove any soap residue. Detergent washed glassware is then stored until ready for use.

7.2 All graduated cylinders, beakers and volumnetric flasks must be acid washed immediately before use. To acid wash, rinse twice with 1:1 HNO_3, discarding into the acid waste drum, followed by two rinses with nanopure water, which is also discarded into the acid waste drum. Exception: Volumetrics may be acid washed and set aside for later use provided that they are kept capped until use.

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7.3 Before using any of the Class A pipettes, rinse thoroughly with 1:1 HNO_3 and nanopure water, then rinse pipette with a small volume of the liquid to be pipetted and discard into the acid waste drum. After use, rinse the pipette thoroughly with 1:1 HNO3 and nanopure water and return to the drawer.

7.4 Watch glasses are kept in a large beaker of 1:1 HNO_3 under the hood and must be rinsed with nanopure water before use. They should be detergent washed and rinsed with tap water before being returned to the beaker.

7.5 All plasticware currently used is disposable and received in "clean" condition, therefore no detergent wash is required. All plasticware should, however, be rinsed with nanopure water before use. This rinse water may be discarded down the sink.

7.6 BOD bottles, used for Cold Vapor Analysis by AA (CVAA), for the determination of mercury, are cleaned differently. After a mercury run, the BOD bottles should be emptied into the acid waste drum and rinsed thoroughly with tap water. Pour approximately 20 ml of hot 1:1 HNO_3 into each botte, cap it, and carefully shake. After allowing the acid to cool, discard into the acid waste drum and thoroughly rinse the bottles with nanopure water.

8.0 QUALITY CONTROL

8.1 Using an aliquot of nanopure water treated in the same manner as the samples, a preparation blank during each digestion provides a means of checking for contamination of the sample.

8.2 If the level of any analyte of interest exceeds the CRDL for that analyte in the preparation blank it is assumed that the samples themselves were contaminated, possibly due to inadequate cleaning of the glassware used in the preparation.

9.0 METHOD PERFORMANCE-N/A

10.0 DATA PERFORMANCE-N/A

11.0 SPECIAL PROCEDURES-N/A

12.0 USE OF OTHER REQUIRED SOP'S

12.1 Applicable SOP's include those dealing with sample or standard preparation, the specific instrument operation as well as any necessary corrective action required.

13.0 DOCUMENTATION OF PROBLEMS

13.1 If a preparation blank exceeds the CRDL during analysis, and contamination is suspected, a corrective action must be filed by the analyst who discovered the problem. Refer to the Corrective Action SOP for instructions on how to proceed.

14.0 REFERENCES

14.1 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020.

AS:071587:2

STANDARD OPERATING PROCEDURE
GLASSWARE/PLASTICWARE CLEANING

ABSTRACT

In order to assure that all samples are prepared free from contamination, all beakers, volumetrics and plasticware are cleaned according to a prescribed procedure.

INTRODUCTION

This procedure outlines the steps necessary in order to wash all associated glassware and plasticeware for sample preparation in the Atomic Spectroscopy Group to insure that it is free from contamination.

It is the intent of this procedure to comply with the ~~DHC~~ IEA *business ethics policy.*

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to all glassware necessary in the standard and sample preparation of both water and soil samples for metals analysis.

2.0 SUMMARY OF METHOD

2.1 The glassware is washed with a phosphate-free detergent and then acid washed.

3.0 INTERFERENCES-N/A

4.0 APPARATUS AND MATERIALS

4.1 Pertinent glassware for the specific procedure.

5.0 REAGENTS

5.1 Liquinox
5.2 Nitric acid, mixed 1:1 with nanopure water (1:1 HNO3).

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE-N/A

7.0 PROCEDURE

7.1 All graduated cylinders, beakers and volumetric flasks are initially washed thoroughly with Liquinox and then rinsed with tap water to remove any soap residue. Detergent washed glassware is then stored until ready for use.

7.2 All graduated cylinders, beakers and volumetric flasks must be acid washed immediately before use. To acid wash, rinse twice with 1:1 HNO3, discarding into the acid waste drum, followed by two rinses with nanopure water, which is also discarded into the acid waste drum. Exception: Volumetrics may be acid washed and set aside for later use provided that they are kept capped until use.

7.3 Before using any of the Class A pipettes, rinse thoroughly with 1:1 HNO3 and nanopure water, then rinse pipette with a small volume of the liquid to be

pipetted and discard into the acid waste drum. After use, rinse the pipette thoroughly with 1:1 HNO3 and nanopure water and return to the drawer.

7.4 Watch glasses are kept in a large beaker of 1:1 HNO3 under the hood and must be rinsed with nanopure water before use. They should be detergent washed and rinsed with tap water before being returned to the beaker.

7.5 All plasticware currently used is disposable and received in "clean" condition, therefore no detergent wash is required. All plasticware should, however, be rinsed with nanopure water before use. This rinse water may be discarded down the sink.

7.6 BOD bottles, used for Cold Vapor Analysis by AA (CVAA), for the determination of mercury, are cleaned differently. After a mercury run, the BOD bottles should be emptied into the acid waste drum and rinsed thoroughly with tap water. Pour approximately 20 ml of hot 1:1 HNO3 into each bottle, cap it, and carefully shake. After allowing the acid to cool, discard into the acid waste drum and thoroughly rinse the bottles with nanopure water.

8.0 QUALITY CONTROL

8.1 Using an aliquot of nanopure water treated in the same manner as the samples, a preparation blank during each digestion provides a means of checking for contamination of the samples.

8.2 If the level of any analyte of interest exceeds the CRDL for that analyte in the preparation blank it is assumed that the samples themselves were contaminated, possibly due to inadequate cleaning of the glassware used in the preparation.

9.0 METHOD PERFORMANCE-N/A

10.0 DATA REDUCTION AND DATA HANDLING-N/A

11.0 SPECIAL PROCEDURES-N/A

12.0 USE OF OTHER REQUIRED SOP'S

12.1 Applicable SOP's include those dealing with sample or standard preparation, the specific instrument operation as well as any necessary corrective action required.

13.0 DOCUMENTATION OF PROBLEMS

13.1 If a preparation blank exceeds the CRDL during analysis, and contamination is suspected, a corrective action must be filed by the analyst who discovered the problem. Refer to the Corrective Action SOP for instructions on how to proceed.

14.0 REFERENCES

14.1 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020.

STANDARD OPERATING PROCEDURE
GLASSWARE/PLASTICWARE CLEANING

ABSTRACT

In order to assure that all samples are prepared free from contamination, all beakers, volumetrics and plasticware are cleaned according to a prescribed procedure.

INTRODUCTION

This procedure outlines the steps necessary in order to wash all associated glassware and plasticeware for sample preparation in the Atomic Spectroscopy Group to insure that it is free from contamination.

It is the intent of this procedure to comply with the IEA business ethics policy.

1.0 SCOPE AND APPLICATION

1.1 This procedure is applicable to all glassware necessary in the standard and sample preparation of both water and soil samples for metals analysis.

2.0 SUMMARY OF METHOD

2.1 The glassware is washed with a phosphate-free detergent and then acid washed.

3.0 INTERFERENCES-N/A

4.0 APPARATUS AND MATERIALS

4.1 Pertinent glassware for the specific procedure.

5.0 REAGENTS

5.1 Liquinox
5.2 Nitric acid, mixed 1:1 with nanopure water (1:1 HNO3).

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE-N/A

7.0 PROCEDURE

7.1 All graduated cylinders, beakers and volumetric flasks are initially washed thoroughly with Liquinox and then rinsed with tap water to remove any soap residue. Detergent washed glassware is then stored until ready for use.

7.2 All graduated cylinders, beakers and volumetric flasks must be acid washed immediately before use. To acid wash, rinse twice with 1:1 HNO3, discarding into the acid waste drum, followed by two rinses with nanopure water, which is also discarded into the acid waste drum. Exception: Volumetrics may be acid washed and set aside for later use provided that they are kept capped until use.

7.3 Before using any of the Class A pipettes, rinse thoroughly with 1:1 HNO3 and nanopure water, then rinse pipette with a small volume of the liquid to be

pipetted and discard into the acid waste drum. After use, rinse the pipette thoroughly with 1:1 HNO3 and nanopure water and return to the drawer.

7.4 Watch glasses are kept in a large beaker of 1:1 HNO3 under the hood and must be rinsed with nanopure water before use. They should be detergent washed and rinsed with tap water before being returned to the beaker.

7.5 All plasticware currently used is disposable and received in "clean" condition, therefore no detergent wash is required. All plasticware should, however, be rinsed with nanopure water before use. This rinse water may be discarded down the sink.

7.6 BOD bottles, used for Cold Vapor Analysis by AA (CVAA), for the determination of mercury, are cleaned differently. After a mercury run, the BOD bottles should be emptied into the acid waste drum and rinsed thoroughly with tap water. Pour approximately 20 ml of hot 1:1 HNO3 into each bottle, cap it, and carefully shake. After allowing the acid to cool, discard into the acid waste drum and thoroughly rinse the bottles with nanopure water.

8.0 QUALITY CONTROL

8.1 Using an aliquot of nanopure water treated in the same manner as the samples, a preparation blank during each digestion provides a means of checking for contamination of the samples.

8.2 If the level of any analyte of interest exceeds the CRDL for that analyte in the preparation blank it is assumed that the samples themselves were contaminated, possibly due to inadequate cleaning of the glassware used in the preparation.

9.0 METHOD PERFORMANCE-N/A

10.0 DATA REDUCTION AND DATA HANDLING-N/A

11.0 SPECIAL PROCEDURES-N/A

12.0 USE OF OTHER REQUIRED SOP'S

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14.0 REFERENCES

14.1 "Methods for Chemical Analysis of Water and Wastes," EPA-600/4-79-020.

SOP/FORM MODIFICATION
APPROVAL SHEET

SOP/Form Modification Requested By: D. TRINQUE, Date: 5 FEB 90

Deadline for Modification of SOP/Form: 1 MARCH 90

Deadline for Implementation of Modified SOP/Form: 15 MARCH 90

Date SOP/Form Modified: 15 FEB 90

Date Typing of Modification Sent to Typist: FEB 15, 1990

Date Typing of Modification Completed: _____

Code of SOP/Form: AS:021590:2

Pages Changed: ALL

Approval of Modification to SOP/Form

Group/Section Leader: Dm Trinque, Date: 2/15/90

Lab Manager: _____, Date: _____

QA Director: _____, Date: _____

Lab Director: _____, Date: _____

Date Modification to be Implemented: 2/15/90

Copies of Final Approved Modification Sent to:

Section/Group Members, Lab Manager, Lab Directors (all labs), Marketing Manager, QA Directors (all labs)

300845

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STANDARD OPERATING PROCEDURE

SAMPLE CONTAINER PREPARATION

SC:042590:2

300846

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

Abstract

Sample control procedures at IEA, Inc.-CT are comprised of five key interrelated elements:

(1) Sample Container Preparation
(2) Sample Receipt
(3) Sample Log-In
(4) Sample Storage
(5) Sample Disposal

The SOP for sample control therefore addresses in three separate SOP's, items (1); (2) and (3); and (4) and (5).

The intention of this SOP is to delineate sample control procedures from preparation of sample bottles to their ultimate disposal.

These procedures are designed to ensure that sample integrity is maintained from receipt to analysis to final disposition.

This SOP is intended for all outgoing sample containers.

STANDARD OPERATING PROCEDURE
SAMPLE CONTAINER PREPARATION

Introduction

It is the responsibility of the sample control group and especially the sample custodian to ensure that all sample containers sent and received by IEA are in good condition upon receipt, the samples are labeled properly, the proper documentation is filled out, and the samples are stored in a manner to ensure proper chain-of-custody. The purpose of this document is to delineate the procedures followed by the sample control group in achieving proper custody procedures and maintenance of sample integrity from preparation through receipt and analysis to final disposal. A copy of this section of the laboratory SOP is to be kept in the sample control area and known to all personnel in the group.

1.0 SAMPLE CONTROL PERSONNEL RESPONSIBILITIES IN BOTTLE PREPARATION

The responsibilities and duties of the bottle preparation staff should include, but not be limited to:

1) Prepare and label appropriate sample bottles.

2) Carefully package sample bottles, field blank water and trip blanks to prevent bottle breakage.

3) Initiate client chain of custody.

4) Ship or deliver sample coolers, meet shipping requirements of the USDOT or common carrier.

2.0 BOTTLE PREPARATION

2.1 Bottle orders will be issued from project management upon final scheduling of a sampling event. I-Chem Series 300 or equivalent precleaned sample containers are to be used for NJDEP and NYSDEC bottle orders.

All employees responsible for bottle preparation will be trained and familiarized with the following procedures:

1) Take bottles from stock and label for appropriate parameter(s).

2) Organize bottles in sample sets, i.e. TCL organics for 1 sample: 3/40 Ml VOA vials, 2/1L amber BNA bottles, 2/1L amber pesticide/PCB bottles.

All bottles are preserved according to the specifications outlined in Attachment 1.

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3) Wrap each sample container with bubble wrap.

4) Open a cleaned cooler, line with bubble wrap. For NJDEP samples record cooler ID number on NJDEP cooler tracking log (Figure 1.1).

5) Include a trip blank if volatile sample containers are included in the sample sets. Reference section 5.0.

6) Count and record sample sets and individual bottles packaged into cooler. Allow space for sufficient ice packs to maintain 4°C temperature within the cooler.

2.2 Chain-of-custody documentation is initiated at the time of bottle preparation. Each cooler must have enough field chain-of-custody forms (see Figure 1.0) for the bottles present.

1.) Determine the number of bottle sets in the cooler. Count out sufficient field chain-of-custody forms.

2) Put in the client name and project identification (if known). Put the bottle set number in the box on the far left of the form. In the box labeled "bottle type and preservative" put in the bottle type (i.e. VOA-vial) and preservative (i.e. HCl, H_2SO_4, etc.). In the same row in the box labeled "tests" put in the test to be done on the sample in the particular bottle (i.e. VOA, BNA, metals).

3) Fill out the rest of the form in the same way. Note that total and dissolved metals will require two separate boxes (see Figure 1.2).

4) At the bottom of the form print your name, the date and time (military). Sign the form.

5) In the box at the lower right fill in the shipper (i.e. Federal Express) and the airbill number.

6) Get two numbered cooler seals (see Figure 1.3) and a plastic bag. Put one of the seals in the box. Print your name and date on the other. In the same box as in step 5 put the seal number on the line labeled "outgoing seal ID#" make a copy of the chain-of-custody form.

7) Put the original chain-of-custody form(s) in the plastic bag along with the unlabeled cooler seal. Keep the signed and dated cooler seal aside. Put the bag in the cooler (make sure ice packs are already in the cooler). Close the cooler and secure with tape.

8) Put the cooler seal on the cooler over the seam where the cooler opens (on the front). Put a piece of clear tape over the seal.

9) Give the copy(s) of the form to your supervisor.

10) If the bottle order required I-Chem 300 series glassware or equivalent (pre-cleaned and documented) give the documentation to your supervisor also.

Special Notes for NJDEP

For NJDEP the DEP Form 095 is required. Only one sample is allowed per form. Calculate the number of sample bottles sets in the cooler and obtain the proper number of forms (see Figure 2.0). Fill out the top box of the form (see Figure 2.1) for each sample, put in plastic bag and proceed with step 7. Do not put an IEA field custody form with NJDEP samples.

3.0 SHIPMENT, DELIVERY, PICKUP OF SAMPLE COOLERS

Secured coolers can be shipped by overnight carrier, personally delivered by an IEA carrier or picked up at the laboratory.

1) Coolers may be sent by common carrier, appropriate airbill must be completed and copied to project management for tracking purposes. Shipping is to be billed directly to IEA, and invoiced through project management.

2) Coolers may be delivered directly to the client. Directions to the delivery site will be supplied. The delivery trip should be recorded on the IEA delivery log.

3) Coolers may be picked-up from IEA by client personnel during normal hours of lab operations (Monday through Friday, 8:30 a.m. to 9:00 p.m. - sample control hours are 8:30 a.m. to 5:00 p.m., lab hours are 8:30 a.m. to 9:00 p.m.).

4.0 BOTTLE TYPES - I-Chem Series 300 Pre-Cleaned Containers or Equivalent

AQUEOUS SAMPLES[1]

Parameter	Bottle Type	#/Sample[2]	Preservative
Volatiles*	40 mL glass teflon-lined septum	3	Cool, 4°C, dark or HCl, 4°C (Note 1)
Semi-volatiles	1L amber glass teflon-lined cap	2	Cool, 4°C, dark
Pesticides/PCB's	1L amber glass teflon-lined cap	2	Cool, 4°C, dark
Volatiles* (drinking water)	40 mL glass teflon lined septum	4	Cool, 4°C, dark

NON-AQUEOUS SAMPLES

Parameter	Bottle Type	#/Sample[2]	Preservative
Volatiles	40 mL glass wide-mouth, teflon-lined cap	1	Cool, 4°C, dark
Semi-volatiles and/or pesticides/PCB's	250 mL glass (short form) teflon-lined cap	1	Cool, 4°C, dark

Note 1: Client will determine if HCl is to be added to VOA vials at the time the order is placed with client services.

* Project manager will ascertain the presence of chlorine in a sample. If chlorine is found or chlorine content cannot be determined, a chlorine test kit will be provided along with ascorbic acid to neutralize chlorine.

[1] Including field blanks for aqueous and non-aqueous matrices.

[2] When matrix spike and matrix spike duplicate volumes are required, triplicate (3x) number of sample containers will be supplied.

5.0 FIELD BLANK WATER AND TRIP BLANKS

5.1 Water for field blanks will be obtained and trip blanks prepared immediately before sample coolers are packaged.

5.2 NJDEP requires that ASTM Type I water must be transferred from "same bottle type" to "sample bottle type" to generate an appropriate field blank.

1) Ample water will be provided for field blank preparation for all parameters.

Volatiles
Field blank: 3/40 mL septum vials
Water supplied: 1/250 mL glass septum bottle filled with organic-free reagent water

Semi-volatiles
Field blank: 2/1L amber glass bottles
Water supplied: 3/1L glass bottles filled with organic-free reagent water

Pesticides/PCB's
Field blank: 2/1L amber glass bottles
Water supplied: 3/1L glass bottles filled with organic-free reagent water

Note: If both semi-volatiles and pesticides/PCB's are required from the same site, only 5L of organic-free reagent water will be supplied.

5.3 Trip blank will be provided in each cooler when volatile analyses are required per approval of the client. A trip blank set consists of 2/40 mL septum vials "over filled" with organic-free reagent water (reverse meniscus). The vials should be capped tightly, inverted and tapped. If the sample vials contain any air bubbles, they are considered invalid and must be emptied and reprepped.

For NJDEP:

Field and trip blank water for all organic compounds must be traceable to an analyzed laboratory blank. Upon filling the containers, fill an extra set of bottles. Contact the Project Manager or Laboratory Manager who will assign a job number and fill out the analytical request forms.

FIELD CHAIN OF CUSTODY RECORD

IEA JOB # :

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468

Client :

Project ID :

IEA Project Mgr :

Rush: Y N TAT: Due Date:

Page ___ of

Protocols :

Deliverables :

Bottle Set #	Client Sample ID	Date/Time Sampled	*Matrix	Lab ID	Bottle Type & Preservative	TESTS										QC Required Y/N	Comments
					Field Filt?	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N		
		/															
		/															
		/															
		/															
		/															
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		/															
		/															
		/															
		/															
		/															
AS CC EX GC BNA VO				Ref. #													

* Matrix Codes

A - Air S - Soil
AQ - Aqueous SL - Sludge
C - Complex
D - Drum Was.

Bottles Prepped By :
Date/Time : /
Signature :

Samples Collected By :
Date/Time : /

Bottles Rec'd By :
Date/Time : /
Signature :

In Lab By :
Time : /

Shipper :
Air Bill # :
Outgoing Seal ID#:
Intact On Field Receipt?
Incoming Seal ID#
Intact On Lab Receipt?

FIGURE 1.1

IEA-CT/ NJDEP Cooler Tracking Log

DATE	NAME	Cooler ID	# Sample Sets	# Bottles	Seal Number

COMMENTS:

300854

[redacted]
an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468

FIELD CHAIN OF CUSTODY RECORD

IEA JOB # : 55800E

Page 1 of 1

Client : ABC Company

Project ID : Quarterly Monitoring

IEA Project Mgr :

Rush: Y N TAT: Due Date:

Protocols :

Deliverables :

Bottle Set #	Client Sample ID	Date/Time Sampled	*Matrix	Lab ID	TESTS →	Volatiles	BNA's Pest/PCB	Total metals	Dissolved metals	BOD, TDS, Chloride, Sulfate						QC Required Y/N	Comments
					Bottle Type & Preservative	VOA vial + HCl	1-liter amber glass UNP	1-liter plastic HNO_3	1-liter plastic HNO_3	1-liter plastic UNP							
					Field Filt?	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N		
001		/				3	3	1	1	2							
002		/				3	3	1	1	2							
003		/				3	3	1	1	2							
004		/				3	3	1	1	2							
005		/				2	0	0	0	0							Trip Blank
		/															
		/															
		/															
		/															
		/															

AS CC EX GC BNA VO Ref. #

* Matrix Codes

A - Air S - Soil
AQ - Aqueous SL - Sludge
C - Complex OIL - Oil
D - Drum Was... OTHER - Other

Bottles Prepped By : John Smith
Date/Time : 02/29/90 / 1130 HRS
Signature : John Smith

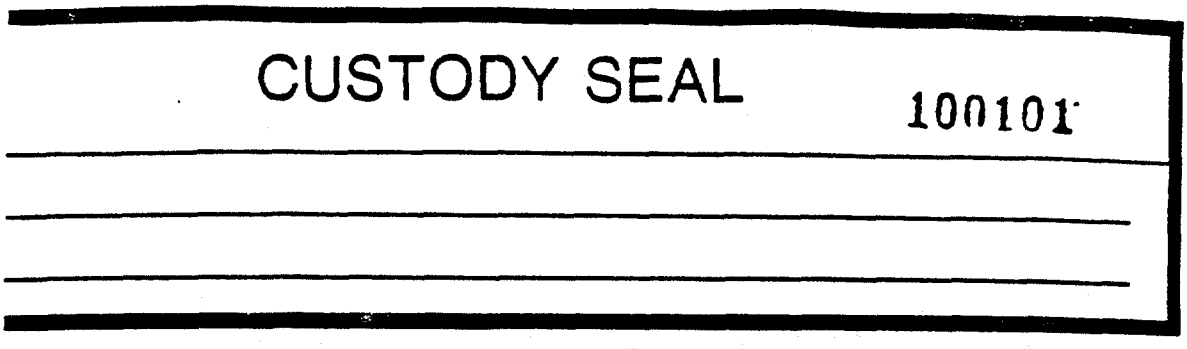
Samples Collected By :
Date/Time : /

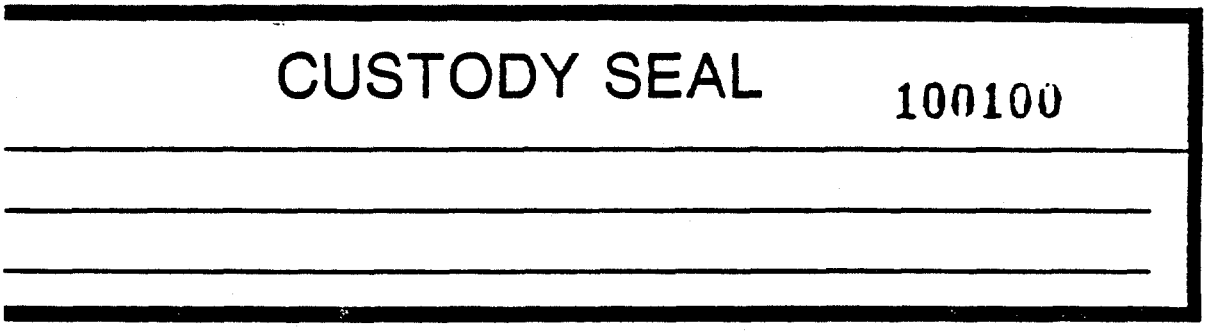
Bottles Rec'd By :
Date/Time : / /
Signature :

In Lab By :
Time : /

Shipper : Fed Exp.
Air Bill # : 98160471371
Outgoing Seal ID#: 10010
Intact On Field Receipt? N
Incoming Seal ID#
Intact On Lab Receipt N

Figure 1.3





300856

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2/91

Figure 2.0

New Jersey Department of Environmental Protection

Page ___ of ___

EXTERNAL CHAIN OF CUSTODY AND SAMPLE ANALYSIS REQUEST FORM
(with Shipping Container)

INSTRUCTIONS: *Use 1 form per sample.*
Enter all dates and times on this form in the following format: (24 hr. clock, day/month/year)

Name of Laboratory: _____

Name/Title of Individual Preparing Sample Bottle(s) and Shipping Container: *(Print)*

Name _____ Title _____

Time/Date Sample Shipping Container Sealed: _____ / _____ Laboratory Affixed Seal No. _____

SAMPLING INFORMATION

Division _____ Bureau _____ Phone (____) _____

Time/Date Sample Shipping Container Opened: _____ / _____

Time/Date Sampling Initiated: _____ / _____ Time/Date Sampling Terminated: _____ / _____

Sample Location:

Company: _____

Address: _____

Physical Location of Sample: _____

DEP Field Sample Number: _____

REQUESTED ANALYSIS(ES)

Parameter	Method	Container: Preserv.	Container: Volume	Container: Qty	Description of Sample Matrix

Preservative Added: *(Check one)* ☐ Laboratory ☐ Field ☐ N/A

Contract No.: _____ Task No.: _____ Report Format: _____

Samples are: *(Check one)* ☐ Emergency ☐ Non-emergency

INTERNAL CHAIN OF CUSTODY

Relinquished	Received	Time/Date	Reason for Change of External Custody
XXXXXXXXXXXXXXXXXXXX			Break Seal/Sample

Name/Title of Individual Resealing Shipping Container: *(Print/Sign)*

Name: _____ Title: _____

Time/Date Sample Shipping Container Resealed: _____ / _____ DEP Affixed Seal No. _____

Name/Title of Individual Breaking Shipping Container Seal and Accepting Responsibility at the Laboratory for the Sample: *(Print/Sign)* Name: _____ Title: _____

Time/Date Sample Shipping Container Opened: _____ / _____

Time/Date Internal Chain of Custody Initiated on DEP Form 077 (Internal Chain of Custody) _____ / _____

Distribution: *White - Original (sent with report)* *Green - Contractor Spare, retain with report file* *Yellow - Sample Custodian* *Pink - DEP Field Sampling Personnel* *Gold - Sample Preparation/Shipment*

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3/91

Figure 2.1

New Jersey Department of Environmental Protection

Page 1 of 1

EXTERNAL CHAIN OF CUSTODY AND SAMPLE ANALYSIS REQUEST FORM
(with Shipping Container)

INSTRUCTIONS: *Use 1 form per sample.*
Enter all dates and times on this form in the following format: (24 hr. clock, day/month/year)

Name of Laboratory: IEA - CT
200 Monroe Turnpike
Monroe, CT 06468

Name/Title of Individual Preparing Sample Bottle(s) and Shipping Container: *(Print)*
Name John Smith Title Sample Control Technician

Time/Date Sample Shipping Container Sealed: 02/29/90 1130HRS Laboratory Affixed Seal No. 100101

SAMPLING INFORMATION

Division _____ Bureau _____ Phone (___) _____
Time/Date Sample Shipping Container Opened: _____ / _____
Time/Date Sampling Initiated: _____ / _____ Time/Date Sampling Terminated: _____ / _____

Sample Location:
Company: _____
Address: _____
Physical Location of Sample: _____
DEP Field Sample Number: _____

REQUESTED ANALYSIS(ES)

Parameter	Method	Container: Preserv.	Container: Volume	Container: Qty	Description of Sample Matrix

Preservative Added: *(Check one)* ☐ Laboratory ☐ Field ☐ N/A
Contract No.: _____ Task No.: _____ Report Format: _____
Samples are: *(Check one)* ☐ Emergency ☐ Non-emergency

EXTERNAL CHAIN OF CUSTODY

Relinquished	Received	Time/Date	Reason for Change of External Custody
XXXXXXXXXXXXXXXXXX			Break Seal/Sample

Name/Title of Individual Resealing Shipping Container: *(Print/Sign)*
Name: _____ Title: _____
Time/Date Sample Shipping Container Resealed: _____ / _____ DEP Affixed Seal No. _____
Name/Title of Individual Breaking Shipping Container Seal and Accepting Responsibility at the Laboratory for the Sample:
(Print/Sign) Name: _____ Title: _____
Time/Date Sample Shipping Container Opened: _____ / _____
Time/Date Internal Chain of Custody Initiated on DEP Form 077 (Internal Chain of Custody) _____ / _____

Distribution: *White - Original (sent with report)* *Green - Contractor Spare, retain with report file* *Yellow - Sample Custodian* *Pink - DEP Field Sampling Personnel* *Gold - Sample Preparation/Shipment*

300858

ATTACHMENT 1

Parameter	Preservation/Amount	Container
VOL	HCL - 4 drops or Unpreserved	40 mL glass teflon-lined septum screw cap
BNA	Unpreserved	2/1L glass amber teflon-lined cap
Pest/PCB	Unpreserved	1/1L glass amber teflon-lined cap
Metals	HNO_3 1 mL to pH under 2	1/1L plastic
Cyanide	NaOH & ascorbic 1 mL to pH over 12.0	1/1L plastic

300859

NJDEP X-408 SAMPLE CONTAINER ORDER CHECKLIST

The following items must be verified for NJDEP Sample Container Shipment

Item	Yes/No
Is a NJDEP Form-095 present?	_____
Is the NJDEP Form-095 filled out correctly?	_____
Are custody seals present and numbered?	_____
Is there adequate field blank/trip blank water present?	_____
Are blue ice or ice packs present?	_____
Is the cooler sealed with a custody seal?	_____
Is the shipping address correct?	_____

300860

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

STANDARD OPERATING PROCEDURE

SAMPLE RECEIPT AND LOG-IN

SC:120588:4

300861

TABLE OF CONTENTS

STANDARD OPERATING PROCEDURE
SAMPLE RECEIPT AND LOG-IN

1.0 SAMPLE RECEIPT

Upon receipt of samples at IEA, Inc.-CT, the sample control group is notified by the receptionist. The samples are brought to the sample control center of the laboratory and placed by the sample control hood. Sign and date any airbills, packing slips, etc. Sample control department operates Monday through Friday from 8:30 a.m. to 5:00 p.m. Sample receipt after normal hours is handled by the second shift supervisor who signs the chain of custody, checks for short holding time samples, then ices the samples with blue ice overnight and locks the samples in the sample control office.

The personnel involved in opening sample kits or coolers wear appropriate safety clothing including eye protection, gloves, and lab coats. The sample shipping carton (cooler, box, etc.) is placed in the hood (which is on). The carton is opened and the sample containers are checked for leaks, breakage, potential hazards, etc. If no problems are observed the samples are transferred to the area for the log-in process. If a problem is noticed, STOP and immediately notify the Sample Custodian and/or the Laboratory Manager. They will decide what actions to take.

Unpack the samples from the shipping container and group them by client ID and wipe off the outside of the bottles with paper towels. Obtain the client chain of custody form. For NJDEP samples, check the shipping container for a NJDEP Form 095 or 096-Sample Analysis Request. (If no Form 095 or 096 is present, contact the Project Manager or the Laboratory Manager immediately. They will advise on whether to proceed or not, see Figure 1.1.) Sign all forms in the appropriate space and check to see there are no discrepancies with sample ID's, bottle types and number of bottles for each sample, etc. Note discrepancies on the custody sheet - sign and date. Sign and date the custody sheet (no initials).

Next obtain an IEA Log-In Record (See Figure 1.0). Do the following:

If a shipping document is present (airbill, etc.) it should be signed and dated. Then do the following:

1) Determine whether or not the project is a continuation of a previously submitted job. This can be accomplished by checking with the appropriate sales person or project manager. If the job is a continuation of a project, assign the appropriate job number. Fill in the job numbers in the bookings record and circle in red.

2) If the project is a new job, assign it the next number in the jobs booking record, and fill in the job number in the bookings record (do not circle).

3) Fill in the appropriate information at the top of the field chain of custody or Sample Log-In Record, including job number, date received, and your name. Fill in client name, client project identification, and total number of samples.

4) Assign each sample a laboratory sample number starting with the lowest number (or letter) as sample number one, unless the job is a continuation of a previous job. If this is the case, determine what the next laboratory sample number is and start with that number on the custody sheet. The convention for sample numbering is that the sample number will be a seven digit number comprised of the last four digits of the IEA job number, followed by a hyphen and a separate number (starting with one) for each sample. The convention is comprised of three pieces of information. The following example illustrates this.

    30xx-YYYY
    where: "3" = the lab division
    where: "xx" = fiscal year
    where: "YYYY" = the sample number

    Suppose four samples were received from a client with the following identifications:

    SW-1
    MW-2A
    MW-2B
    TB

    The project was assigned job number 3089-0015. The samples should be lined up in the following order:

    MW-2A, MW-2B, SW-1, TB

    Sample MW-2A will receive laboratory sample number 0015-001. Sample MW-2B will receive laboratory sample number 0015-002. Sample SW-1 will receive laboratory sample number 0015-003 and sample TB will receive laboratory sample number 0015-004.

5) The client sample identification is filled in on the appropriate line next to the laboratory number (up to 12 characters).

6) Labels are attached to each sample container with the client name and laboratory sample number. These labels are attached to the sample container in such a fashion that they do not obscure any client identification (if possible).

7) Fill in the bottle type(s) present in the column headers for the sample bottle descriptions and the test required.

8) For each sample received record the number of each bottle type present. If no bottles are present put in a zero. Add up the total and fill in the appropriate space with the value.

9) Under matrix fill in the sample type (soil, water, etc.) and any other appropriate information.

10) Sign and date the form in the appropriate box. Fill out the refrigerator locations and page information at the bottom of the form.

11) Obtain a manilla folder. Put the IEA project number, client name and client project ID on the folder. Put the custody sheet in the folder.

For USEPA (See Figure 2.0):

1) Locate the traffic reports. Sign and date the traffic reports in box six.

2) The sample condition and tag number must be noted. Use column F to document this information. If the tags do not match note this on the traffic report. If bottles are broken note this also.

3) Put forms in job folder.

For NJDEP:

1) New Jersey DEP samples require a NJDEP Form 077, Internal Chain of Custody Form (see Figure 2.1). Obtain the form and fill out the required information on the top of the form.

2) Place all forms in the job folder.

For NYSDEC:

1) Find the NYSDEC Contract Lab Information Sheet (see Figure 1.2). Insure the client sample identifications match all bottles.

2) If the caution box is checked, contact the Project Manager or Laboratory Manager immediately. They will advise on how to proceed.

3) Put all forms in the job folder.

Sample Control Record In-House Custody Sheet (See Figure 3.0)

1) Fill out the information required in the upper right-hand corner.

2) Circle the appropriate information in the upper left-hand corner of the form.

3) Put the sheet in the appropriate chain of custody notebook by job number.

Next obtain a Sample Preservative Record (See Figure 4.0). Fill in the header information. For each sample bottle which contains a preservative the preservative must be verified (except for volatile vials and TOX bottles).

First, fill in the sample number in the appropriate column. Record in preservative type in the next column. Each preservative type is checked as follows:

1) Pour a small amount of the sample into a disposable beaker.

2) Nitric acid (for metals) pH must be less than 2. Check with pH paper.

3) Sulfuric acid (for phenols, oil & grease, ammonia, chemical oxygen demand, total kjeldahl nitrogen, total nitrate-nitrite, total organic carbon, total phosphorus) pH must be less than 2. Check with pH paper.

4) Sodium hydroxide/ascorbic acid (for cyanide) check initially with KI paper. No blue color should be present. Next check with pH paper to verify pH greater than 12.

5) Sodium hydroxide/zinc acetate (for sulfide) pH greater than 9. Check with pH paper.

Put the appropriate test paper into the liquid in the plastic beaker for approximately 5 seconds and remove. Wait approximately 5 additional seconds and then compare the color to the chart on the container.

Record the pH value in the first "pH" space on the sample preservation record. If the pH is incorrect do the following:

1) For EPA, NJDEP and NYSDEC samples contact the project manager. They will call the agency for instructions. Do not add any material to the samples without permission from the project manager.

2) For all other clients add the appropriate preservative (use gloves and eye protection) to the container, mix and retest. Record the final pH in the adjustment column.

   NOTE: If a government agency (USEPA, NJDEP, NYSDEC, etc.) requests preservative be added to a sample this must be noted as follows:

   - For EPA, note on the traffic report
   - For NJDEP, note on the Internal Chain of Custody (Form 077)
   - For NYSDEC, note on the Contract Laboratory Information Sheet

If you are unsure of what action to take, check with your supervisor or the Laboratory Manager.

Record the result of the KI-Starch paper in the KI column (positive or negative). If the test is positive add ascorbic acid, mix and retest. Continue until the test is negative.

Sign and date in the appropriate column. Staple sheet(s) to back inside cover of the job folder.

Check over all the forms to ensure there are no discrepancies or errors. Any crossouts must be signed and dated.

Circle each group which has to work to do on the samples. Issue a copy to each group which has responsibilities for the samples (make an appropriate number of copies). Put the original in the job folder. Put a copy for each group in the Group Leader's mailbox.

Send the job folder to the Project Manager/Client Services.

2.0 SAMPLE STORAGE

Samples are segregated by preservative and stored in refrigerators. Volatiles are stored in separate refrigerators. NJDEP samples are stored in separate refrigerators and are ALWAYS locked. The sample custodian (Group Leader) and the Laboratory Manager have keys. See them for access to those refrigerators. EPA samples also have their own refrigerators.

Sample refrigerator temperatures must be maintained between 4-6°C. Freezer temperatures are kept below -12°C.

A temperature log should be taken on a daily basis. The temperature log should be kept in the sample control center. The sample control center is unlocked at 8:00 a.m. by the sample custodian and is locked after normal business hours.

Each refrigerator has its temperature checked on a daily basis. These temperatures are recorded in the temperature logbook. All entries are signed and dated. If a refrigerator is out of service, it should be noted in the logbook. Also, if temperature excursions occur, the corrective action taken should be noted in the logbook.

LOGIN RECORD

IEA JOB # : _____

iea an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468

Client : _____

Project ID : _____

IEA Project Mgr : _____

Rush: Y N TAT: __ Due Date: _____

Page ___ of

Protocols : _____

Deliverables : _____

Bottle Set #	Client Sample ID	Date/Time Sampled	# Matrix	Lab ID	TESTS: Bottle Type & Preservative / Field Filt?										QC Required Y/N	Comments
						Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
AS CC EX GC BNA VO				Ref. #												

* Matrix Codes
A - Air W - Water S - Soil W - Wipe FB - Field Blank
O - Other TB - Trip Blank

c'd In Lab By : _____
e/Time : ___ /
nature : _____

Incoming Seal ID#:
Intact On Lab Recei Y N

Figure 1.1

DEP-095
3/91

New Jersey Department of Environmental Protection

Page ___ of ___

EXTERNAL CHAIN OF CUSTODY AND SAMPLE ANALYSIS REQUEST FORM (with Shipping Container)

INSTRUCTIONS: *Use 1 form per sample.*
Enter all dates and times on this form in the following format: (24 hr. clock, day/month/year)

Name of Laboratory: _____

_____

_____

Name/Title of Individual Preparing Sample Bottle(s) and Shipping Container: *(Print)*

Name _____ Title _____

Time/Date Sample Shipping Container Sealed: _____ / _____ Laboratory Affixed Seal No. _____

SAMPLING INFORMATION

Division _____ Bureau _____ Phone (___) _____

Time/Date Sample Shipping Container Opened: _____ / _____

Time/Date Sampling Initiated: _____ / _____ Time/Date Sampling Terminated: _____ / _____

Sample Location:

Company: _____

Address: _____

_____

Physical Location of Sample: _____

DEP Field Sample Number: _____

REQUESTED ANALYSIS(ES)

Parameter	Method	Container: Preserv.	Container: Volume	Container: Qty	Description of Sample Matrix

Preservative Added: *(Check one)* ☐ Laboratory ☐ Field ☐ N/A

Contract No.: _____ Task No.: _____ Report Format: _____

Samples are: *(Check one)* ☐ Emergency ☐ Non-emergency

EXTERNAL CHAIN OF CUSTODY

Relinquished	Received	Time/Date	Reason for Change of External Custody
XXXXXXXXXXXXXXXXXX			Break Seal/Sample

Name/Title of Individual Resealing Shipping Container: *(Print/Sign)*

Name: _____ Title: _____

Time/Date Sample Shipping Container Resealed: _____ / _____ DEP Affixed Seal No. _____

Name/Title of Individual Breaking Shipping Container Seal and Accepting Responsibility at the Laboratory for the Sample: *(Print/Sign)* Name: _____ Title: _____

Time/Date Sample Shipping Container Opened: _____ / _____

Time/Date Internal Chain of Custody Initiated on DEP Form 077 (Internal Chain of Custody) _____ / _____

Distribution: *White - Original (sent with report)* *Green - Contractor Spare, retain with report file* *Yellow - Sample Custodian* *Pink - DEP Field Sampling Personnel* *Gold - Sample Preparation/Shipment*

300869

CONTRACT LAB SAMPLE INFORMATION SHEET

Print legibly

Figure 1.2

COLLECTED BY	TELEPHONE NUMBER:	REGION NO.	
CONTRACT LAB:	COUNTY:	SAMPLING DATE.	MILITARY TIME
SAMPLING POINT:	OUTFALL NUMBER	CHECK IF SAMPLING IS PART OF INSPECTION	
	SPDES NUMBER	FLOW	MGD
CASE NUMBER	SDG NUMBER	SAMPLE NUMBER	CHECK FOR MS/MD ☐ This Sample
TYPE OF SAMPLE: ☐ Grab ☐ Composite ☐ Term ____ hrs			

SAMPLE MATRIX:
☐ Air ☐ Soil/Sediment ☐ Groundwater ☐ Surface Water ☐ Wastewater ☐ Other (Specify) _____

CHECK THE BOX PRECEDING THE REQUESTED ANALYSIS

PRIORITY POLLUTANTS (Water Part 136)—SPDES

☐ 1. All (SPDES)—Includes 2-6	☐ 2. 13 PP Metals	☐ 3. Volatiles—USEPA 624 (GC/MS)
☐ 4. Acids Base/Neutrals (USEPA 625-GC/MS)	☐ 5. Cyanide	☐ 6. Pesticides/PCB's (USEPA 608-GC)
☐ 7. Halogenated Volatiles (USEPA 601-GC)	☐ 8. Aromatic Volatiles (USEPA 602-GC)	☐ 9. BOD
☐ 10. pH	☐ 11. COD	☐ 12. TSS
☐ 13. Settleable Solids	☐ 14. TKN	☐ 15. Ammonia
☐ 16. Nitrate/Nitrite	☐ 17. Total Phosphrous	☐ 18. Reactive Phosphorus
☐ 19. Oil/Grease	☐ 20. TOC	☐ 21. Total Phenols
☐ 22. Other _____	☐ 59. PCB's at 0.065 ug/L	☐ 60. PCB's congener method

CONTRACT LABORATORY PROTOCOLS

☐ 23. (ALL)—Water—Includes 24-28	☐ 29. (ALL)— Soil/Sediments—Includes 30-34
☐ 24. Base/Neutral/Acid (B/N/A)—Water—GC-MS	☐ 30. B/N/A—Soils/Sediment—GC-MS
☐ 25. Volatile Organic Analysis VOA—Water—GC-MS	☐ 31. VOA—Soils/Sediments—GC-MS
☐ 26. Pesticides/PCB's—Water—GC	☐ 32. Pesticides/PCB's—Soils/Sediment—GC
☐ 27. Metals—Water	☐ 33. Metals—Soil/Sediment
☐ 28. Cyanide—Water	☐ 34. Cyanide—Soils/Sediment
	☐ 35. Other _____

HAZARDOUS WASTES/RCRA ANALYSIS SW-846

☐ 36. EP Toxicity	☐ 37. EP Toxicity (Metals Only)	☐ 38. Ignitability
☐ 39. Corrosivity	☐ 40. VOA—(USEPA 8240)	☐ 41. BNA—(USEPA 8270)
☐ 42. Pesticides/PCB's (USEPA 8080)	☐ 43. TCLP	☐ 44. TCLP (Metals Only)
☐ 45. Reactivity	☐ 46. Dioxin (USEPA 8280)	☐ 47. Appendix IX
☐ 48. Other _____		

MUNICIPAL SLUDGE

☐ 49. RSGB-01 ☐ 50. RSSR-01 ☐ 51. RSGR-01 ☐ 52. RSRB-01 ☐ 53. RSRI-01 (EP Toxicity-Metals only + RSRR-01)
☐ 54. RSRO-01 ☐ 55. RSSB-01 ☐ 56. RSRR-01 ☐ 57. RSRR-02 ☐ 58. Other _____

CAUTION (check if applicable)

☐ **Lab Personnel are expected to use caution when handling DEC samples, however, please use special precautions when handling this sample since it is believed to contain significant concentrations of hazardous and/or toxic material(s).**

Place QA Label Here

300870

P.O. BOX 818 ALEXANDRIA, VA 22313
703/557-2490 FTS 557-2490

CASE NO:

FIGURE 2.0

ORGANIC TRAFFIC REPORT

(OR CLP USE ONLY)

TYPE OF ACTIVITY (CIRCLE ONE) ①
SUPERFUND—PA (SI) ESI RIFS RD RA ER
NPLD O&M OTHER _____
NON-SUPERFUND—_____ PROGRAM

SAMPLE DESCRIPTION
(ENTER IN BOX A)
1. SURFACE WATER
2. GROUND WATER
3. LEACHATE
4. SOIL
5. SEDIMENT
6. OIL (SAS)
7. WASTE (SAS)

REGION NO: SAMPLING COMPANY ②

SAMPLER: (NAME)

SHIP TO: ③
IEA Inc-CT
200 Monroe Turnpike
Monroe, CT 06468
203 261 4458
ATTN: Dana Gervino

SAMPLING DATE: 10/31/24 ④
BEGIN: 0830 END: 1330

DATE SHIPPED: _____ CARRIER: _____ ⑤
AIRBILL NO: Sent Courier

DATE REC'D:

REC'D BY:

LABORATORY CONTRACT NO.

TRANSFER TO: DATE

REC'D BY: CONTR

CLP SAMPLE NUMBER (FROM LABELS)	⑥ SAMPLE DESCRIPTION (FROM BOX 1) 1 2 3 4 5 6 7	⑦ CONCENTRATION L = LOW/MED M = HIGH (SAS)	⑧ RAS ANALYSIS: VOLATILE	BASE/NEUT ACID	PESTICIDE /PCB's	⑨ SPECIAL HANDLING	⑩ STATION LOCATION	⑪ SAMPLE CONDITION ON RECEIPT
								TAG #
AA 1111		L	X	X	X		DD-TB-01	Good: #4-1234
AB 2222			X	X	X		DE-06-11	Good: #4-1234
AC 3333			X	X	X		DF-07-22	1 BNA BROKEN - #4-1234

EPA Form 2075-7 (8-87)

* Tag does not match

300871

WHITE — SMO COPY PINK — CLIENT COPY WHITE — LAB COPY FOR RETURN TO SMO YELLOW

DEP-077
2/89

FIGURE 3.1

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTECTION
TRENTON, NEW JERSEY 08625

INTERNAL CHAIN OF CUSTODY

Laboratory Person Breaking Field Seal on Sample Shuttle & Accepting Responsibility for Sample	Name	Title
Field Sample Seal No.	Date Broken ____/____/____	Military Time Seal Broken
Case No.	Analytical Parameter/Fraction	

SAMPLE NO.	ALIQUOT/EXTRACT NO.	SAMPLE NO.	ALIQUOT/EXTRACT NO.

Date	Time	RELINQUISHED BY	RECEIVED BY	PURPOSE OF CHANGE OF CUSTODY
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	
		PRINTED NAME SIGNATURE	PRINTED NAME SIGNATURE	

IEA, INC.
IN-HOUSE CUSTODY SHEET

Client:

Custody Seal	present/absent	DATE:
	intact/not intact	SAMPLE CUSTODIAN SIGNATURE:
Chain of Custody	present/absent	JOB NUMBER:
Sample Tags	present/absent	CASE NUMBER:
	listed/not listed	AIRBILL NUMBER:
SMO Forms	present/absent	SAMPLE NUMBERS:

LABORATORY SAMPLE NUMBER	REMOVED BY	DATE AND TIME REMOVED	REASON	DATE AND TIME RETURNED	RETURNED TO REF. #
					REF#

300873

FIGURE 3.0

300874

IEA, INC. SAMPLE PRESERVATIVE RECORD

JOB NUMBER:
CLIENT:
CLIENT PROJECT:

LAB NUMBER	PRESERVATIVE	PH	KI	ADJUSTMENT	INITIALS	DATE

SAMPLE CONTROL
QUALITY CONTRTOL APPROVAL REPORT

Job #:_____ Client:_____

New/Cont.:_____ Sample #:_____

I. Initial Inspection

A) Sample condition - all samples intact? _____
If not, group leader notified? _____

B) All airbills and client paperwork signed and dated? _____

C) Client sample ID's match custody sheets? _____
If not, inform group leader.

D) Coolers emptied and lab sample numers match client paperwork and bottles? _____

E) YWC custody sheet correctly filled out, signed and dated? _____

II. Documentation

A) Job folders contain: client chain of custody, traffic reports/airbills/all client paperwork, archive/disposal form, result sheet? _____

B) Preservation requirements have been check and recorded on sample preservation record which is stapled to job folder? _____

C) Preliminary sample notifications have been issued and present in job folder? _____

III. Final Inspection

A) YWC labels on correct bottles? _____

B) Refrigerator locations noted? _____

C) All problems documented? _____

D) All custody/airbills, etc. signed? _____

E) Folder to group leader? _____

The above samples have been received according to the current SOP. All problems documented and approved by the Group Leader.

_____ _____
Sample Custodian Date

300875

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

STANDARD OPERATING PROCEDURE

SOIL/SEDIMENT SAMPLE PREPARATION

AS:041791:0

300876

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Wheaton, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

SOIL /SEDIMENT SAMPLE PREPARATION

INTRODUCTION

It is the policy of IEA Laboratories and of the Atomic Spectroscopy group to ensue that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the IEA, Inc. Policy Statement on Business Ethics and Conduct.

1.0 SCOPE AND APPLICATION

1.1 This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for analysis by furnace atomic absorption spectroscopy (AAS) or by inductively coupled plasma spectroscopy (ICP) for the following metals:

Aluminum	Chromium	Potassium
Antimony	Cobalt	Selenium
Arsenic	Copper	Silicon
Barium	Iron	Silver
Beryllium	Lead	Sodium
Boron	Magnesium	Thallium
Cadmium	Manganese	Tin
Calcium	Molybdenum	Titanium
	Nickel	Vanadium
		Zinc

1.2 This method was derived from 1990 EPA contract and complies with New York State, New Jersey, and SW846 contracts.

2.0 SUMMARY OF METHOD

2.1 A representative 1g (wet weight) sample is digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with either nitric acid or hydrochloric acid. Hydrochloric acid is used as the final reflux acid for the ICP analysis of Al, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Mg, Mn, Ni, K, Ag, Na, V and Zn. Nitric acid is employed as the final reflux acid for the Furnace AA analysis of As, Pb, Se, Tl. A separate sample is dried for a percent solids determination.

3.0 INTERFERENCES N/A

4.0 APPARATUS AND MATERIALS

4.1 250ml pyrex beaker
4.2 watch glasses
4.3 filter paper
4.4 200ml volumetric flasks
4.5 disposable plastic funnels
4.6 500ml pastic bottles
4.7 Top loading scale
4.8 tins
4.9 class "A" pipets

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193)
5.2 Concentrated nitric acid (sp. gr. 1.41)
5.3 Concentrated hydrochloric acid (sp. gr. 1.19)
5.4 Hydrogen peroxide (30%)
5.5 1:1 nitric acid
5.6 1:1 hydrochloric acid

6.0 SAMPLE PRESERVATION AND HANDLING

6.1 Soil/sediment samples must be refrigerated at 4 C from receipt until analysis.
6.2 All glassware must be detergent , acid, and nano washed.
6.3 All disposable plastic ware must be nano rinsed.
6.4 All samples must be signed out of sample control log book and signed in when returned. Refer to Documentation SOP.
6.5 Samples must be returned to their designated refrigerator at the end of each working day.

7.0 PROCEDURE

7.1 Safety goggles, lab coat, and gloves must be worn at all times in the laboratory.
7.2 Generate a LIMS printout of samples to be prepared.
7.2.1 Type in "S MTL,134,METALS" <return>
7.2.2 Type in "MASTER/G" <return>
7.2.3 Type in "5", (user report management) <return>
7.2.4 Type in "4", (generate a predefined report) <return>
7.2.5 For "criteria filename" type in "PREPS" <return>
7.2.6 "Change output devise", type "N" for no, to have the list printed at the wet chem lab printer.
7.2.7 "Enter range/list-test", type in "ICAP\ICAPZ", to obtain a list of samples which need preparing for the ICP. "FURN\FURNZ" will give a list of samples which need to be done for the furnace. <return>
7.2.8 Hit the "ESC" key to exit the program.
7.2.9 Type in "OFF" <return> and shut down the computer.
7.3 Determine samples to prep.
7.3.1 Under column titled "analysis", the first four digits are the job number and the last three are the sample identification number. The abbreviations "Dup" for duplicate and "SPK" for spike may appear after certain samples and indicates that the specific sample was chosen by the client for Quality Control purposes. Refer to section 8.0 before prepping.
7.3.2 Prep seperately ICAP and Furnace. Determine according to due date, up to 20 samples / QC (See 8.0) to prep. Consider keeping samples from the same job together.
7.3.3 Choose matrix 2 or 3 samples. Matrix "2" = soil, matrix "3" = other.
7.3.4 Under the column titled "protocol". SOP may be prepped with any other protocol. NYS89 must be prepped seperately from all other protocols, the exception being SOP (this rule applies to furnace preps only).
7.3.5 Note the refrigerator number under the "location" column on printout.
7.3.6 Check job folder for special instructions. Some CLP jobs comprise many samples and need to be broken down into SDG's for ease of review. SDG's are complete two weeks after the receipt of the first sample. Wait for an SDG to be complete before prepping. If no QC has been indicated and more than one job is being prepped together, choose a sample that has the most elements being indicated for analysis. "TAL" refers to all elements.

7.4 Obtain a pagenated soil prep sheet
7.4.1 Fill in the case # or SAS # and SDG if doing an EPA case.
7.4.2 Circle ICP or Furnace whichever is applicable.
7.4.3 Fill in the sample ID# given, under the column "IEA ID", and if an EPA case or SAS is being prepped, be sure to fill in the EPA sample ID, under the column "IFB ID".
7.5 Samples are to be found in sample control.
7.5.1 Sign out samples in log book after removing from refrigerator.
7.6 Sample preparation can begin.
7.6.1 Assign (write in black marker) each 250ml beaker and 125ml plastic container with a sample ID, which corresponds with each sample to be prepped. Include initials, date, job number, and sample ID's on the prep blank and LCS containers.
7.6.2 Acid wash all glassware and plasticware to be used according to Glassware SOP.
7.6.3 Calibrate the top loading balance with the 1g and 0.5g Class "S" wt. and record on soil prep log on the line for "Calibration".
7.6.4 Mix the sample thoroughly to acheive homogeneity. For each sample, weigh (to the nearest 0.01g) out 1.0 to 1.5 g in a tared beaker, and record its weight.
7.6.5 Describe sample color (column 1), using these abbreviations: R=red, B=blue, Y=yellow, G=green, O=orange, V=violet, W=white, C=colorless, BN=brown, GY=gray, BL=black.
7.6.6 Describe sample texture in column 2: "F" = fine, "M" = medium, "C" = coarse.
7.6.7 Describe artifacts if any in column 4, ex. roots.
7.6.8 Add 10ml of 1:1 HNO3, mix the slurry , and cover with a watch glass.
7.6.9 Heat the sample to 95 C and reflux for 10 minutes. It is critical that the sample reflux, this operation constantly mixes the sample with the acid matrix allowing dissolution and leaching of the analytes of interest.
7.6.10 Cool and add 5ml concentrated HNO3, and reflux for 30 minutes (maintain a covering of solution over the bottom of the beaker).
7.6.11 Cool and rinse sides of beaker and bottom of watch glass with nanopure water, then add 3ml of 30% hydrogen peroxide.
7.6.12 Warm beaker on hot plate to start peroxide reaction, heat until effervescence subsides. Be careful not to lose sample due to excessively vigorous effervescence. Samples with high organic content will require more hydrogen peroxide and more time than sandy samples low in organic content.
7.6.13 Continue to add peroxide in 1 ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than 10ml.
7.6.14 For ICP analysis, add 5ml of 1:1 HCL and 10ml of nanopure water, heat for an additional 10 minutes. Do not boil, Antimony is lost in hydrocholoric medium.
7.6.15 For Furnace analysis, continue heating the acid-peroxide digestate until it is reduced in volume to 2ml. Add 10ml nanopure water and warm.
7.6.16 Allow sample to cool.
7.6.17 Filter sample (using pre-rinsed filter paper) into a 200ml volumetric flask. Rinse down beaker walls and filter paper into volumetric to recover as much of digestate as possible.
7.6.18 Readjust acidity by adding 5ml nitric acid and 5ml hydrochloric acid to the ICP prep and 2ml nitric acid to the furnace prep.
7.6.19 Bring to volume with nanopure water.
7.6.20 Transfer digestate to pre-rinsed and pre-labeled 500ml plastic container.
7.6.21 Wash glassware in soapy water. Return samples to Sample Control and sign log.
7.7 Percent Solids Determination (should be done on the same day as the sample prep)

7.7.1 Calibrate top loading balance with the 5.0g, 1.0g, and 0.5g Class "S" weight and record on Percent Solid form.
7.7.2 Write sample ID's on each weighing dish and fill out % solids log.
7.7.3 Mix sample to be weighed to acheive homogeneity.
7.7.4 Weigh weighing dish to nearest 0.01g, record, tare balance.
7.7.5 Alliquot approximately 5.00g of sample into the pre-weighed, tared dish. Record weight of sample.
7.7.6 The sample chosen for duplicate analysis must also have a duplicate percent solid determination performed.
7.7.7 Place samples in drying oven, note time and temp on % solids log. Oven must be maintained between 103-105 C.
7.7.8 Dry until stable weight is attained, at least 12 hours and no longer than 24 hours.
7.7.9 Remove dry sample from oven, note time and date, and allow to cool in the dessicator.
7.7.10 Weigh and record the sample weight to the nearest 0.01g.
7.7.11 Calculation

$$\% \text{ solids} = \frac{\text{Sample dry weight (g)}}{\text{Sample wet weight (g)}} \times 100$$

7.7.12 A computer program is available for percent solids calculation on the NEC.
7.7.12.1 Turn computer on and type in "solids" at the "C:\>" que.
7.7.12.2 Type in sample ID's and weights
7.7.12.3 Be sure printer is on, hit the F10 key to have the solids calculated and printed.
7.7.13 The LCS need not have a percent solids determination performed, and is equal to 100%.
7.8 Place prep and prep log on appropriate instrument work shelf.
7.9 Remove samples prepped from computer.
7.9.1 Sign on to LIMS. Type "S MTL,134, METALS" <return>
7.9.2 Type "COMPLETE/G", <return>
7.9.3 Enter the full seven digit sample ID. A backslash "\" denotes all inclusive. For example: 0123001\0123010 will erase samples 1-10 of job 0123. Use a comma to seperate individual samples. <return>
7.9.4 Enter the test to be completed. Five tests may be entered. For example: Type in "ICAP-PREP" <return> defaults to 1, enter a numeral 2 if total and dissolved samples were prepped.<return>, "FURN-PREP" <return> 2X.
7.9.5 The sample ID's listed on the screen have been deleted from the file.
7.9.6 If more samples are to be deleted hit <return> and continue entering. Type "N" for no when all sample ID's have been entered and <return>.
7.9.7 Type "OFF" <return>, and turn off computer.
7.10 Filing
7.10.1 After the ICP or Furnace operator signs the soil prep log and % solids log, it is then signed by the group leader, and returned to the individual who did the prep. Make one copy for each job.
7.10.2 File in job folder
7.10.3 Fill out a chain of custody form, include job number and sample ID's completed, sign and date. Date it for the day that the samples were prepped.
7.10.4 The data within the job folder must follow a certain order to facilitate the review process. Chain of Custody, ICP data, Furnace data, Mercury data, Prep log, Traveler.
7.10.5 File the original Preparation Log in the black notebook marked "Soils", and the % solids log in the appropriate notebook.

8.0 QUALITY CONTROL (for each 20 samples prepped)

8.1 Blank sample (ie. nanopure water)
8.1.1 Prep along with other samples exactly as above instructions indicate
8.1.2 The blank is useful in determining if samples are being contaminated during the prep.
8.2 Duplicate sample.
8.2.1 Choose a sample to duplicate from the 20 being prepped, if one is not designated.
8.2.2 Take the duplicate sample along the entire sample preparation proceedure.
8.2.3 The duplicate sample is used to determine precision.
8.3 Spiked sample.
8.3.1 The sample chosen for duplicate should also be the sample chosen for spiking.
8.3.2 Before the acid addition :
8.3.3 Add 5ml / 200ml (using Class "A' pipets) of the ICP spike solution (designated by the iea-w-#) to the soil sample designated as the ICP spike.
8.3.4 Add 4ml / 200ml (using class "A" pipets) of the furnace spike solution (designated by the iea-w-#) to the soil sample designated as the furnace spike.
8.3.5 Take the spiked sample along the entire sample preparation proceedure.
8.3.6 The spike sample provides information about the effect of the sample matrix on the digestion and measurement methodology.
8.4 Laboratory control sample.
8.4.1 A solid LCS must be prepped along with the other samples.
8.4.2 Weigh out as close to 1.00g as possible
8.4.3 The LCS provides information on how adequately the digestion process was done.
8.4.4 Record LCS ID on prep log in appropriate space.

9.0 METHOD PERFORMANCE N/A

10.0 DATA HANDLING

10.1 All soil prep sheets are pagenated and completed originals filed.
10.2 A copy of the prep sheet is filed in the appropriate job folder, and a chain of custody signed and dated.
10.3 All corrections on forms are to be crossed out with a single line, initialed and dated.

11.0 SPECIAL PROCEDURES

11.1 Oysters
11.1.2 Shuck oysters
11.1.3 Acid wash blender
11.1.4 Blend oysters thoroughly
11.1.5 Percent solids determination is unnecessary
11.1.6 Proceed with soil prep
11.2 Animal tissue
11.2.1 Grind with tissue miser
11.2.2 Percent solids determination is unnecessary
11.2.3 Proceed with soil prep

12.0 USE OF OTHER REQUIRED SOP'S

12.1 Documentation and Paperwork
12.2 Glassware

13.0 DOCUMENTATION OF PROBLEMS

13.1 Corrective Action Reports.
13.1.1 Preps must be reprepped if the analysis of the prep blank indicated contamination problems, or the LCS has not been adequately digested and falls outside the specified percent recovery limits set by the EPA.
13.1.2 Notify sample control if a problem occurs with the sample while in your possession, i.e. sample container breaking, mislabled samples.

14.0 REFERENCES

12.1 SW846
12.2 1990 EPA contract
12.3 1989 N.Y. State Dept. of Environmental Conservation

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

SAMPLE PREPARATION OF WATERS

AS:041791:0

300883

Miramar, Florida 305-888-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-879-5 38

SAMPLE PREPARATION OF WATERS

INTRODUCTION: It is the policy of IEA Laboratories and of the Atomic Spectroscopy Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the IEA, Inc. Policy Statement on Business Ethics and Conduct.

1.0 Scope and Application

This method is an acid digestion procedure used to prepare aqueous samples for analysis by furnace atomic absorption spectroscopy or by inductively coupled plasma spectroscopy. Samples prepared by this method may be analyzed by AAS or ICP for the following metals:

Aluminum	Cobalt	Selenium
Antimony	Copper	Silicon
Arsenic	Iron	Silver
Barium	Lead	Sodium
Beryllium	Magnesium	Thallium
Boron	Manbanese	Tin
Cadmium	Molybdenum	Titanium
Calcium	Nickel	Vanadium
Chromium	Potassium	Zinc

2.0 SUMMARY OF METHOD

2.1 Samples alloted for ICP analysis are heated with nitric and hydrochloric acids and substantially reduced in volume. The digestate is filtered and diluted to volume, and is then ready for analysis.

2.2 Samples alloted for furnace analysis are heated with nitric acid and hydrogen peroxide and substantially reduced in volume. The digestate is filtered and diluted to volume, and is then ready for analysis.

3.0 INTERFERENCES N/A

4.0 APPARATUS AND MATERIALS

250ml pyrex beaker
watch glasses
filter paper
disposable funnels
100 ml volumetric flasks
100ml graduated cylinder
hot plates
squeeze bottles
class "A" glassware pipets

5.0 REAGENTS

5.1 ASTM Type II water (ASTM D1193)

5.2 concentrated nitric acid (sp.gr.1.41)
5.3 concentrated hydrochloric acid (sp.gr. 1.19)
5.4 30% hydrogen peroxide
5.5 1:1 nitric acid
5.6 1:1 hydrochloric acid

6.0 SAMPLE PRESERVATION AND HANDLING

6.1 All aqueous samples must be nitric preserved (pH<2), and refrigerated.
6.2 All glassware and plastic containers must be prewashed with detergents, acid and Type II water. Refer to SOP on glassware acid washing.
6.3 All samples removed from sample control must be signed out, and signed in when returning.
6.4 Samples must be returned to their designated refrigerator at the end of each working day.

7.0 PROCEDURE

7.1 Safety goggles, lab coat, and gloves must be worn at all times in the laboratory.
7.2 Generate a LIMS printout of samples to prepare.
7.2.1 Type in "S MTL,134,METALS" <return>
7.2.2 Type in "MASTER/G" <return>
7.2.3 Type in "5", (user report management) <return>
7.2.4 Type in "4", (generate a predefined report) <return>
7.2.5 For "criteria filename" type in "PREPS" <return>
7.2.6 "Change output devise", type "N" for no, to have the list printed at the wet chem lab printer.
7.2.7 "Enter range/list-test", type in "ICAP\ICAPZ", to obtain a list of samples which need preparing for the ICP. "FURN\FURNZ" will give a list of samples which need to be done for the furnace. <return>
7.2.8 Hit the "ESC" key to exit the program.
7.2.9 Type in "OFF" <return> and shut down the computer.
7.3 Determine samples to prep
7.3.1 Under column titled "analysis", the first four digits are the job number and the last three are the sample identification number.
7.3.2 Prep seperately ICAP and Furnace. Determine according to due date, up to 20 samples / QC (See 8.0) to prep. Consider keeping samples from the same job together.
7.3.3 Choose matrix 1 samples. Matrix "1" = water
7.3.4 Under the column titled "protocol". SOP may be prepped with any other protocol. NYS89 must be prepped seperately from all other protocols, the exception being SOP (this rule applies to furnace preps only).
7.3.5 Note the refrigerator number under the "location" column on printout. Most HNO3 preserved water samples can be found in refrigerators 26,27,28,29, and 36.
7.3.6 Check job folder for special instructions. Some CLP jobs comprise many samples and need to be broken down into SDG's for ease of review. SDG's are complete two weeks after the receipt of the first sample. Wait for an SDG to be complete before prepping.
7.4 Obtain a pagenated water prep sheet.
7.4.1 Fill in the case # or SAS # if doing an EPA case.
7.4.2 Circle ICP or Furnace, whichever is applicable.
7.4.3 Fill in the sample ID # given under the column "IEA ID", and if an EPA case or SAS is being prepped, be sure to fill in EPA sample ID given under the column "IFB ID".

7.5 Samples are to be found in sample control.
7.5.1 Sign out samples in log book after removing from refrigerator.
7.6 Sample preparation can begin.
7.6.1 Assign (write in black marker) each 250ml beaker and 125ml plastic container with a sample ID, which corresponds with each sample to be prepped. Include initials, date, job number, and sample ID's on the prep blank and LCS containers.
7.6.2 Acid wash all glassware and plasticware to be used according to Glassware SOP
7.6.3 Shake sample to obtain homogeneity.
7.6.4 Transfer a 100ml sample aliquot (using a 100ml graduated cylinder, pre-rinsed with sample) to a 250ml beaker. Record initial volume used under the column titled "Volume i (ml)".
7.6.5 Describe sample color and clarity under the column for "Physical Description". Color is described in the first column using the abbreviations listed here: R=red, B=blue, Y=yellow, G=green, O=orange, V=violet, W=white, C=colorless, BN=brown, GY=gray, BL=black. Clarity is described in the third column using the abbreviations listed here: CL=clear, CD=cloudy, O=Opaque
7.6.6 Add reagents: For ICP prep, add 2ml 1:1 HNO_3 and 10ml 1:1 HCl. Swirl to mix. For furnace prep, add 1ml 1:1 HNO_3 and 2ml of 30% H_2O_2. Swirl to mix.
7.6.7 Cover with a watch glass and heat on a hot plate for 2 hours at 95 C or until sample volume is reduced to between 25 and 50ml. Do not allow sample to boil. (Antimony is easily lost by volatilization from hydrochloric acid media).
7.6.8 When this volume is obtained, remove from heat. Allow sample to cool.
7.6.9 Filter sample (using pre-rinsed filter paper) into a 100ml volumetric flask. Rinse down beaker walls, and filter paper, with nano, to recover as much of digestate as possible.
7.6.10 Bring to volume with nanopure water (100ml).
7.6.11 Record on prep log the final volume under column "Volume f (ml)". Record the color of the sample in the second column under "Physical Description", and clarity of the sample in the fourth column under "Physical Description".
7.6.12 Transfer digestate to pre-rinsed and labeled 125ml plastic container. Sign and date sample prep log. Include the number on acid bottles used for that particular prep.
7.7 Place sample digestates on appropriate instrument work shelf and include completed prep log.
7.8 Remove samples prepped from computer.
7.8.1 Sign on to LIMS. Type "S MTL,134, METALS" <return>
7.8.2 Type "COMPLETE/G", <return>
7.8.3 Enter the full seven digit sample ID. A backslash "\" denotes all inclusive. For example: 0123001\0123010 will erase samples 1-10 of job 0123. Use a comma to seperate individual samples. <return>
7.8.4 Enter the test to be completed. Five tests may be entered. For example: Type in "ICAP-PREP" <return> defaults to 1, enter a numeral 2 if total and dissolved samples were prepped.<return>, "FURN-PREP" <return> 2X.
7.8.5 The sample ID's listed on the screen have been deleted from the file.
7.8.6 If more samples are to be deleted hit <return> and continue entering. Type "N" for no when all sample ID's have been entered and <return>.
7.8.7 Type "OFF" <return>, and turn off computer.
7.9 Filing
7.9.1 After the ICP or Furnace operator signs the water prep log, it is then signed by the group leader, and returned to the individual who did the prep. Make one copy for each job.
7.9.2 File in job folder

7.9.3 Fill out a chain of custody form, include job number and sample ID's completed, sign and date. Date it for the day that the samples were prepped.
7.9.4 The data within the job folder must follow a certain order to facilitate the review process. Chain of Custody, ICP data, Furnace data, Mercury data, Prep log, Traveler.
7.9.5 File the original Preparation Log in the black notebook marked "Waters".

8.0 QUALITY CONTROL (for each 20 samples prepped)

8.1 Preparation blank (ie. nanopure water)
8.1.1 A preparation blank must be prepared for each batch of waters digested or every 20 samples, whichever is more frequent.
8.1.2 Prep along with other samples exactly as above instructions indicate.
8.1.3 The blank is useful in determining if samples are being contaminated.

8.2 Laboratory control sample.
8.2.1 An aqueous LCS must be prepared for each batch of waters digested or every 20 samples, whichever is more frequent.
8.2.2 Spike 100ml of nanopure water, using Class "A" pipets with: 5ml of the ICP LCS (designated by the iea-w-#), or 1ml of the furnace LCS (designated by the iea-w-#), for all protocol with the exception of EPA case work. For EPA work, spike 100ml of nanopure water with 10 ml of ICV1 and 10ml of ICV3 solution for ICP preps. Spike 100ml of nanopure water with 5ml of ICV2 and 5ml of ICV3 solution for furnace preps.
8.2.3 Fill out prep sheet under "QC addition" with "IEA-W- and the number that appears on the LCS bottle", also include the amount spiked in ml. When using the ICV solutions include amount used in ml, appropriate solution, and the ICV ID.

8.3 Duplicate sample.
8.3.1 If a client wishes to designate a specific sample for duplication it will be indicated on the computer printout next to the sample ID as "DUP". If this is not indicated choose a sample to duplicate from the 20 being prepped.
Field blanks can not be used as QC.
8.3.2 Take the duplicate sample along the entire sample preparation proceedure.
8.3.3 The duplicate sample is used to determine precision.

8.4 Spiked sample.
8.4.1 The sample chosen for duplication should also be the sample chosen for spiking unless otherwise specified.
8.4.2 Before the acid addition :
Add 2.5ml (using Class "A' pipets) of the ICP spike solution (designated by the iea-w-#) to 100ml of the ICP sample designated as the spike.
Add 2ml (using class "A" pipets) of the furnace spike solution (designated by the iea-w-#) to 100ml of the furnace sample designated as the spike.
8.4.3 Take the spiked sample along the entire sample preparation proceedure.
8.4.4 The spike sample provides information about the effect of the sample matrix on the digestion and measurement methodology.

9.0 METHOD PERFORMANCE N/A

10.0 DATA HANDLING

10.1 All water prep sheets are pagenated and completed originals filed.
10.2 A copy of prep is filed in the appropriate job folder, and chain of custody signed and dated.

10.3 All corrections on forms must have a single line through them, initialed and dated.

11.0 SPECIAL PROCEDURES

11.1 Occassionaly we receive samples of an oily nature. These should be prepped with caution. Heat slowly and use stirring rods in the beaker. Additional acid may be needed. The oil layer forms an impenetrable film on the surface by which the water vapor can not escape causing a small explosion to occur, consequently sample is lost.

11.2 Performance evaluations (PE's).

11.2.1 Read instructions given by client.

11.2.2 Acid wash all glassware to be used. Use class "A" pipets. Label all volumetrics

11.2.3 To a 500ml volumetric, add enough nanopure water to cover bottom and acidify to 1% HNO3. Pipet in 5ml of sample, bring to volume with nano, shake to obtain homogeneity, and stopper.

11.2.4 Make a duplicate.

11.2.5 To a 50ml and 25ml volumetric, add enough nano to cover bottom and acidify with 1ml HNO3. Pipet in 1ml of sample, bring to volume with nano, shake and stopper.

11.2.6 Complete a water prep log and place it along with the PE's on the ICAP workshelf.

12.0 USE OF OTHER REQUIRED SOP'S

12.1 Documentation and Paperwork

13.0 DOCUMENTATION OF PROBLEMS

13.1 Corrective Action Reports.

13.1.1 Preps must be reprepped if the analysis of the prep blank indicated contamination problems, or the LCS has not been adequately digested and falls outside the specified percent recovery limits set by the EPA.

13.1.2 Notify sample control if a problem occurs with the sample while in your possession, i.e. sample container breaking, mislabled samples.

14.0 REFERENCES

14.1 SW846
14.2 1990 EPA contract
14.3 1989 N.Y. State Dept. of Environmental Conservation

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

STANDARD AND SAMPLE PREPARATION LOGS

AS:041891:0

300889

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

300889

AS:041891:0

STANDARD AND SAMPLE PREPARATION LOGS

ABSTRACT: This proceedure describes the inventory and tracking of sample preparation and standard preparation.
It is the intent of this procedure to comply with the IEA business ethics policy.

1.0 SCOPE AND APPLICATION

1.1 The standard preparation log book tracks receipt of standards, preparation, and expiration dates.

1.2 The sample preparation log book contains preprinted data sheets, which track sample preparation, dates, additions of acids, and analytes, and the preparation blank and LCS associated with each group of samples prepared together.

1.3 The percent solid log book tracks the determination of solid sample weights for calculation purposes.

1.4 The leachate log book is a record of leachates received from the wet chemistry group, and includes information on samples spiked, and their preservation.

1.5 The filtration log book is a record of all samples filtered and preserved in the metals laboratory before the sample is prepared for analysis.

1.6 The mercury sample preparation log book contains information on reagents, acids, and spikes added to samples being prepared for mercury. It also contains information on standard preparation, blanks, and LCS's used for each mercury run.

2.0 SUMMARY OF METHOD

2.1 Standard log book - the first section includes the stock standard solutions in which all pertinent information supplied by the manufacturer is recorded upon receipt. The second section is for recording the preparation of working standard solutions, such as, the ICP and Furnace spikes and LCS's, Mercury standards, etc.

2.2 Sample log book must be completed as samples are prepped, spiked, preserved, filtered, etc. Any test, preparation, or addition must be recorded in these log books.

2.3 All entries to the logbooks must be signed and dated by the analyst and the individual doing the work.

2.4 All entries to the logbooks must be completed in ink.

2.5 If an error is made, corrections shall be made by crossing a line through the error and entering the correct information. Initial and date the change.

3.0 INTERFERENCES N/A

4.0 APPARATUS AND MATERIALS

4.1 standard log book

4.2 sample log book
4.3 percent solids log book
4.4 leachate log book
4.5 filtration log book
4.6 mercury log book

5.0 REAGENTS

5.1 All acid and reagent bottles are number sequentially upon receipt. If a contamination occurs with sample preparation or standard preparation, the acid or reagent bottle may be traced.

6.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

6.1 All samples are to be refrigerated in sample control
6.2 Purchased stock standards and solutions can be stored as received at room temperature.
6.3 ICV solutions must be refrigerated when received
6.4 All prepared standards are acidified and stored at room temperature.

7.0 PROCEDURE

7.1 Stock standard log book. Refer to fig. 1. Record the following information from the bottle.
7.1.1 Stock ID (not found on bottle). This is a code specific to the laboratory and always begins with "MTL-S(for stock)-0001"(the last four digits are numerical entries generated in sequential order.
7.1.2 element name
7.1.3 manufacturer name
7.1.4 date received
7.1.5 element concentration
7.1.6 manufacturer lot number
7.1.7 expiration date (if no expiration date is given the analyst should assume a one-year expiration date from the date received)
7.2 Working standard log book. Refer to fig. 2.
7.2.1 Standard ID. This code is specific to the laboratory and always begins with "MTL-W(for working standard)-0001" (the last four digits are numerical entries generated in sequential order.
7.2.2 date standard is prepared
7.2.3 Under "element" list the elements being prepared.
7.2.4 Next for "stock/standard ID" list the stock or standard ID used to make the dilution.
7.2.5 For "concentration" list the concentration of the stock or standard being used.
7.2.6 Vi = initial volume in milliliters of stock or standard taken.
7.2.7 Vf = final volume in milliliters standard was diluted to.
7.2.8 "concentration f" = the final concentration in ppm or ppb of the standard.
7.2.9 For "chemist" sign your name and include type of acid used, %, and number found on bottle assigned by laboratory.
7.3 Water and TCLP sample preparation log book. Refer to fig. 3.
7.3.1 Circle "water" on top of form.
7.3.2 If using eppendorf for spiking additions calibrate using 1ml of water on top loading balance and record weight, ex. 1ml = 1.00g, otherwise indicate "class A pipets used".
7.3.3 "Case/SAS" enter case number or SAS number if doing EPA work.
7.3.4 "SDG #" used for EPA case work or CLP work.

7.3.5 Circle low or medium level for EPA work or Std (standard) for regular work.
7.3.6 "ICAP/Furnace" circle appropriate prep.
7.3.7 "IEA ID" column. List sample ID's.
7.3.8 "IFB ID" column, used for EPA work, list EPA's sample ID.
7.3.9 "Vol i (ml)" = initial volume in milliliters.
7.3.10 "Vol f (ml)" = final volume in milliliters.
7.3.11 "Phys. Desc. before/after" enter sample color using color key in column 1 as sample appears before prep begins. Enter sample color after sample is brought to volume. In column 2, enter clarity of sample using abbreviations from key before sample was prepped and after sample was brought to a final volume.
7.3.12 "QA/QC" - "PB" = preparation blank, only initial volume and final volume need to be completed.
7.3.13 "LCS" = laboratory control sample. Enter initial and final volumes. "QC Additions" enter standard stock ID and amount, ex. 2.5ml MTS-w-0001.
7.3.14 "D" = duplicate sample. Enter sample ID, initial and final volumes.
7.3.15 "S" = spike sample. Enter sample ID, initial and final volumes, standard stock ID and amount.
7.3.16 "Prepared by" individual preparing samples signature goes here.
7.3.17 "date" = date prepared
7.3.18 "Accepted by" individual analyzing samples signature and date.
7.3.19 "Supervisor" = supervisors' signature and date.
7.3.20 Record laboratory # given to acid and reagent bottles at bottom of page.

7.4 TCLP sample preparation log is similar to above, exceptions are: circle TCLP at top of form. Refer to fig. 4.

7.5 Soil sample preparation log. Refer to fig. 5. Similar to Water preparation log, differences are listed below.
7.5.1 Circle "soil" at top of form.
7.5.2 "Calibration" refers to the calibration of the top loading balance with class "S" weights. Record as, ex. 1.00g = 1.00g class "S" wt.
7.5.3 "Weight (g)" refers to the weight in grams of sample used in digestion.
7.5.4 "Volume (ml)" refers to the final volume digestate was diluted to.
7.5.5 Under the heading "Physical Description", the first column lists the color of the soil before digestion/ color of liquid at final volume. Column 2, list the texture of the soil before digestion procedure. Column 3, list the clarity of the liquid at final volume after digestion is complete. Column 4, list artifacts, ex. roots.

7.6 Percent solids log book. Refer to fig. 6.
7.6.1 "case/SAS/batch no" refers to EPA cases and SAS
7.6.2 "SDG no." refers to EPA and CLP work, record number.
7.6.3 "Calibration" record Class "S" wt. from top loading balance.
7.6.4 "Oven temp" record the oven temperature in degrees Celsius at the time the soil sample went in and the temperature at the time the soil was taken out.
7.6.5 "IEA ID" record IEA sample ID.
7.6.6 "IFB ID" record EPA sample ID.
7.6.7 "Dish Wt." record the aluminum tin weight in grams
7.6.8 "Sample wet weight" record the soil weight in grams, before placing in the oven.
7.6.9 "Sample dry weight plus dish" this is the weight of the soil after it has been dried in the oven and includes the dish weight.
7.6.10 "Sample dry weight minus dish weight", record here the weight of the soil after subtracting the dish weight from column 3.
7.6.11 "% solids" record here the final result by dividing the wet weight from column 4 by the dry weight minus the dish wt. in column 6.
7.6.12 "date in" record date soils were put into the oven.
7.6.13 "time in" record in military time, that soils were placed in the oven.
7.6.14 "date out" record date soils were taken out of the oven.

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7.6.15 "time out" record in military time, that the soils were taken out of the oven.
7.6.16 "prepared by" requires a signature
7.6.17 "date" record date results were calculated.
7.6.18 "witnessed by" requires signature and date.

7.7 Leachate log book. Bound notebook.
7.7.1 Record IEA job number, and blank associated with batch
7.7.2 Record IEA sample ID number(s).
7.7.3 Record date received from Wet Chemistry
7.7.4 Initials
7.7.5 Refrigerator location in which samples will be stored.
7.7.6 Comments: include, sample ID spiked and standard ID of spiking solution, preserved with HNO3 and acid bottle ID.

7.8 Filtration log book. Bound notebook.
7.8.1 Record IEA job number
7.8.2 Record IEA sample ID number(s).
7.8.3 Record date received from sample control, which must also be the date filtered.
7.8.4 Initials
7.8.5 Record refrigerator location in which samples will be stored.
7.8.6 Record any comments off the original bottle.

7.9 Mercury sample preparation log book. Refer to fig. 7.
7.9.1 Circle water or soil which ever matrix is applicable.
7.9.2 "top loading balance calibration" record weight of class "S" wt. off top loading balance.
7.9.3 "analytical balance calibration" record weight of class "S" wt. off the analytical balance.
7.9.4 "page __of __" record page number and number of pages in run.
7.9.5 "IEA ID" column, list the IEA sample ID.
7.9.6 "IFB ID" column, list the EPA sample ID.
7.9.7 "Wi(g)/Vi(ml)" record the weight in grams of the soil sample or the initial volume of the water sample in millileters.
7.9.8 "Vf(ml)" record the final volume in millileters the sample was diluted to.
7.9.9 "BOD ID" = the bottle ID into which the sample was poured.
7.9.10 "QA/QC ID" List the standards, CCV's, ICV's, CCB's, LCS'S, prep blanks, spikes, and duplicates for the run in this column.
7.9.11 "Wi/Vi" record the initial weight or initial Volume.
7.9.12 "QC add" record QC ID addition and amount added.
7.9.13 "Vf(ml)" record the final volume in millileters.
7.9.14 "BOD ID" record the bottle ID into which the sample was poured.
7.9.15 "standard ID =" enter the full standard ID for calibration standards.
7.9.16 "ICV ID" enter the EPA initial calibration verification standard ID used.
7.9.17 "CCV ID" enter the continuing calibration verification standard ID used.
7.9.18 "LCS ID" enter the EPA laboratory control solid sample ID used.
7.9.19 "spike ID" enter the spiking standard ID used.
7.9.20 "prepared by" signature of individual preparing samples.
7.9.21 "date" enter date samples prepared.
7.9.22 "lot #" enter the number given by the laboratory on the acid and reagent bottles listed.
7.9.23 "accepted by" signature and date of supervisor reviewing data.

8.0 QUALITY CONTROL
8.1 Sample preparation forms are signed, dated, and reviewed by supervisor as they are prepared.
8.2 Preprinted sample preparation forms are filed in numerical sequence to ensure easy accessabiliy when backtracking samples.

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8.3 A bound leachate log book is filled out immediately as leachates are received from the Wet Chemistry group for inventory purposes. This is also the case with the filtration log book.
8.4 Stock standards are recorded upon receipt to keep track of expiration dates.
8.5 Standards are recorded using the stock ID from which they came, to ensure ability to back track to the source if any contamination problems occur.
8.5 All samples, stock standards, standards, reagents, and acids are given in house laboratory ID's to ensure the ability to backtrack through everything if contamination problems occur.

9.0 METHOD PERFORMANCE

9.1 In the event of an LCS, or prep blank, failure on the insruments resulting in the batch of samples having to be reprepared, sequential numerical entry allows for an easy way to retreive information.
9.2 Likewise sequential numerical entry of standards in the log book allows for easy accesibility to the previous standard made, so that preparation may be uniform.
9.3 The leachate log book makes accessible the date when samples were leached from Wet Chemistry, so the individual preparing the samples for analysis may know which samples were leached.

10.0 DATA REDUCTION AND DATA HANDLING - N/A

11.0 SPECIAL PROCEDURES - N/A

12.0 USE OF OTHER REQUIRED SOP'S
12.1 The SOP that applies to the particular procedure should be consulted before sample or standard entry begins.

13.0 DOCUMENTAION OF PROBLEMS
13.1 Corrective action reports-any mislabeled samples or standards, any misreported data, or any incorrectly entered forms warrants generation of a corrective action report per the Atomic Spectroscopy Corrective Action Report Procedure.

14.0 REFERENCES
14.1 USEPA Contract Laboratory Program, SOW 3/90
14.2 Standard and Sample Logbooks.

Fig. 1

STOCK ID :
ELEMENT :
NUFACTURER :
DATE RECEIVED :

CONCENTRATION:
MANUFACTURER LOT # :
EXPIRATION DATE:

STOCK ID :
ELEMENT :
MANUFACTURER :
DATE RECEIVED :

CONCENTRATION:
MANUFACTURER LOT # :
EXPIRATION DATE:

STOCK ID :
ELEMENT :
MANUFACTURER :
DATE RECEIVED :

CONCENTRATION:
MANUFACTURER LOT # :
EXPIRATION DATE:

STOCK ID :
ELEMENT :
MANUFACTURER :
DATE RECEIVED :

CONCENTRATION:
MANUFACTURER LOT # :
EXPIRATION DATE:

STOCK ID :
ELEMENT :
MANUFACTURER :
DATE RECEIVED :

CONCENTRATION:
MANUFACTURER LOT # :
EXPIRATION DATE:

STOCK ID :
ELEMENT :
ANUFACTURER :
DATE RECEIVED :

CONCENTRATION:
MANUFACTURER LOT # :
EXPIRATION DATE:

Fig 2

STD ID: ____________________ DATE: ____________________

	ELEMENT(S)	STK/STD ID	CONC.	V_i (MLS)	V_f (MLS)	C.
1						
2						
3						
4						
5						
6						
7						

CHEMIST: ____________________

STD ID: ____________________ DATE: ____________________

	ELEMENT(S)	STK/STD ID	CONC.	V_i (MLS)	V_f (MLS)	$CONC._f$
1						
2						
3						
4						
5						
6						
7						

CHEMIST: ____________________

STD ID: ____________________ DATE: ____________________

	ELEMENT(S)	STK/STD ID	CONC.	V_i (MLS)	V_f (MLS)	$CONC._f$
1						
2						
3						
4						
5						
6						
7						

CHEMIST: ____________________

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Fig. 3

001173

METALS SAMPLE PREP LOG - WATER

Calibration of eppendorf
Case/SAS _____ Low/Med/Std ICAP/Furnace SDG #: _____

IEA ID	IFB ID	Vol i (ml)	Vol f (ml)	Phys. Desc.* before/after				Color Key*
				1-	/	2-	/	R = red
				1-	/	2-	/	B = blue
				1-	/	2-	/	Y = yellow
				1-	/	2-	/	G = green
				1-	/	2-	/	O = orange
				1-	/	2-	/	V = violet
				1-	/	2-	/	C = colorless
				1-	/	2-	/	Bn = brown
				1-	/	2-	/	Gy = gray
				1-	/	2-	/	Bl = black
				1-	/	2-	/	Clarity*
				1-	/	2-	/	CL = clear
				1-	/	2-	/	C = cloudy
				1-	/	2-	/	O = opaque
				1-	/	2-	/	
				1-	/	2-	/	
				1-	/	2-	/	
				1-	/	2-	/	
				1-	/	2-	/	
				1-	/	2-	/	

*Each physical description involves 1-Color and 2-Clarity both before and after

QA/QC

IEA ID		IFB ID	Volume i (ml)	Volume f (ml)	QC Addition
PB					
LCS					
	D				
	S				

Prepared by: _____ Supervisor: _____
Date: _____ Date: _____
Accepted by: _____ Date: _____
ICP: HNO_3 _____ HCl _____ H_2O_2 _____ Furnace: HNO_3 _____ H_2O_2 _____

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Fig 4

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METALS SAMPLE PREP LOG - TCLP

Calibration of eppendorf
Case/SAS_____ Low/Med/Std
ICAP/Furnace SDG #:_____

IEA ID	IFB ID	Vol i (ml)	Vol f (ml)	Phys. Desc.* before/after		Color Key*
				1- /	2- /	R = red
				1- /	2- /	B = blue
				1- /	2- /	Y = yellow
				1- /	2- /	G = green
				1- /	2- /	O = orange
				1- /	2- /	V = violet
				1- /	2- /	C = colorless
				1- /	2- /	Bn = brown
				1- /	2- /	Gy = gray
				1- /	2- /	Bl = black
				1- /	2- /	Clarity*
				1- /	2- /	CL = clear
				1- /	2- /	C = cloudy
				1- /	2- /	O = opaque
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	

*Each physical description involves 1-Color and 2-Clarity both before and after

QA/QC

IEA ID		IFB ID	Volume i (ml)	Volume f (ml)	QC Addition
PB					
LCS					
	S				
	S				
	S				

Prepared by:_____ Supervisor:_____
Date:_____ Date:_____
Accepted by:_____ Date:_____
ICP: HNO_3_____ HCl_____ H_2O_2_____ Furnace: HNO_3_____ H_2O_2_____

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Fig. 5

METALS SAMPLE PREP LOG - SOIL

Case/SAS _____ Low/Med/Std _____ SDG #: _____
Calibration: _____ ICAP/Furnace

IEA ID	IFB ID	Weight (g)	Volume (ml)	Physical Description* before/after				Color Key*
				1- /	2-	3-	4-	R = red
								B = blue
								Y = yellow
								G = green
								O = orange
								V = violet
								C = colorless
								Bn = brown
								Gy = gray
								Bl = black
								Clarity*
								Cl = clear
								C = cloudy
								O = opaque
								Texture*
								F = fine
								M = medium
								C = coarse

*Each physical description involves 1-Color (before and after), 2-Texture (before), 3-Clarity (after), and 4-Artifacts (yes or no description)

QA/QC

IEA ID		IFB ID	Weight (g)	Volume f (ml)	QC Addition
PB					
LCS					
	D				
	S				

Prepared by: _____ Supervisor: _____
Date: _____ Date: _____
Accepted by: _____ Date: _____
ICP: HNO_3 _____ HCl _____ H_2O_2 _____ Furnace: HNO_3 _____ H_2O_2 _____
Page ___ of ___

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Fig. 3

% SOLIDS DETERMINATION

IEA 200 Monroe Turnpike
Monroe, Connecticut 06468 (203) [illegible]

Case/SAS/Batch No: _____ SDG No: _____ Calibration: _____

% Solids = (Dry Wt / Wet Wt) x 100 Oven Temp (C): In: _____ Out: _____

IEA ID	IFB ID	Dish wt (g) Wds	Sample Wet Wt (g) ww (minus dish)	Sample Dry wt (g) plus dish	Sample Dry wt (g) minus dish	% Solids

Date in: _____ Time in: _____ AM/PM Date out: _____ Time out: _____ AM

* 1) A minimum of 5.00g of wet sample should be taken for this analysis.
2) Do not exceed a 24 hour drying time.
3) One sample must be determined in duplicate (see ICP prep for dup select

Prepared by: _____ Date: _____ Witnessed by: _____ Date

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Monroe, CT 06468 (203) 452-8200

METALS WATER/SOIL PREPERATION LOG - MERCURY

Top Loading Balance Calibration____________

Analytical Balance Calibration____________

Page ___ of ___

IEA ID	IFB ID	Wi(g) Vi(ml)	Vf(ml)	BOD ID	QA/QC ID	QA/QC Wi/Vi	QA/QC QC add	QA/QC Vf(ml)	QA/QC BOD ID

QA/QC

Standard ID = ____________

ICV ID = ____________

CCV ID = ____________

LCS ID = ____________

Spike ID = ____________

Prepared by:____________ Accepted by:____________

Date:____________ Date:____________

Lot #: Hydroxylamine Hydrochloride____ Stannous Chloride_____ KMnO4 ____

H2SO4________ HNO3________ K2O8S2________ NaCl________

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000008

iea
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FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

SAMPLE HANDLING AND

CHAIN OF CUSTODY

AS:022189:0

Miramar, Florida 305-989-0929
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-6161
Cary, North Carolina 919-677-0080
Essex Junction, Vermont 802-878-5138

STANDARD OPERATING PROCEDURE
SAMPLE HANDLING AND CHAIN OF CUSTODY

1.0 SAMPLE HANDLING/CHAIN OF CUSTODY

While samples are in a work-in-progress state, it is the responsibility of each analyst working on a sample(s) to do the following:

- Sign the chain-of-custody upon removing and returning each sample for preparation and analysis (Metals see Figure 1.0)
- Be sure to return the sample (unused portion--does not apply to volatiles) to the appropriate refrigerator/ freezer.

2.0 EXTRACT STORAGE

After analysis the extracts (prepared samples) may be stored for up to one month in the AS laboratory. After one month the samples are to be boxed and relocated to the basement storage area. All boxes must be labeled with the case/job # and date of disposal.

3.0 SAMPLE ARCHIVAL/DISPOSAL

Subsequent to analysis and final data review, the sample and any related extracts are archived and/or disposed of according to SOP SC:110386:1.

FIGURE 1.0

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CHAIN OF CUSTODY
ATOMIC SPECTROSCOPY DEPARTMENT

Job Number _____ Sample Numbers _____

WATER - SOIL - SLUDGE - EPTOX/TCLP

I confirm that I have performed the preparation below following SOP guidelines and authorize the release of this preparation:

Sample Prep _____ _____

_____ _____ ICP/FLME

_____ _____ FURN

_____ _____ MERCURY

Chemist Date(s)

I confirm that I have performed the analysis below following SOP guidelines and authorize the release of all associated data:

Analysis _____ _____ ICP

_____ _____ FLAME

_____ _____ FURN

_____ _____ MERCURY

Chemist Date(s)

I have reviewed and authorize the release of this job:

Complete _____ _____

Supervisor Date

Batch Assignment _____

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300904

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STANDARD OPERATING PROCEDURE

STORAGE AND DISPOSAL OF SAMPLES AND RELATED EXTRACTS

SC:042690:2

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-6181
Cary, North Carolina 919-677-0080
Essex Junction, Vermont 802-878-5138

300905

STANDARD OPERATING PROCEDURE
STORAGE AND DISPOSAL OF SAMPLES AND RELATED EXTRACTS

Abstract

This SOP addresses the storage and disposal of client samples (and related extracts) at IEA, Inc.-CT.

No aqueous or soil/sediments are to be disposed of by any other means than as expressly directed in this document and its revisions.

The general approach to waste disposal at IEA involves transfer of samples, acid waste, alkaline waste, solvent and extract waste and PCB waste to segregated labeled drums for disposal by EPA and State of Connecticut licensed (as appropriate) firms, through appropriate Resource Conservation and Recovery Act (RCRA) mandated methods.

This system ensures that all samples, related extracts and other laboratory chemicals/waste are disposed of in an environmentally acceptable manner and in accordance with Good Laboratory Practice (GLP).

Additionally, all waste disposal must be in accordance with State and Federal Laws and Regulation. See the Hazardous Waste Coordinator if you have questions.

1.0 STORAGE

Samples are segregated by preservative and stored in refrigerators. Volatiles are stored in separate refrigerators.

Sample refrigerator temperatures must be maintained between 4-6°C. Freezer temperatures are kept lower than -12°C.

A temperature log should be taken on a daily basis. The temperature log is kept in the sample control center. Sample refrigerators are located in sample control center. Sample control center is unlocked at 8:00 a.m. by the sample custodian and is kept locked after normal business hours.

2.0 SAMPLE DISPOSAL

Upon completion of all analyses, a report is generated and an invoice is prepared. At the time of invoicing, samples are removed from the refrigerators and the sample storage time begins. The length of time each set of samples is held varies by client and is as follows:

Normal holding time (most clients)	21 days
* USEPA	60 days
* NYSDEC	90 days
* USEPA Extracts	365 days
* NJDEP	1 year*

*Note: See Section 3.0

Samples are then placed in the appropriate drums for disposal. Personnel disposing of samples should be properly trained in safe handling procedures and should utilize proper personal protective equipment.

The disposal drums, when full, (target hauling frequencies are once/month) are manifested and hauled by an EPA licensed firm. A copy of the manifest is then placed in the central file.

3.0 DISPOSAL OF CONTRACT SAMPLES AND EXTRACTS

The laboratory's government contracts require specific archive storage times for samples, sample extracts, and sample digestates. These are as follows:

USEPA

Inorganic samples (metals and cyanide): 60 days after report (soil and sediments must be refrigerated during this time)

Inorganic Acid Digestates: 60 days after report submission

Organic Samples: 60 days after report submission

Organic Sample Extracts: 365 days after data submission

NJDEP

All samples and associated extracts must be held for 1 year after date of data submittal. The laboratory must notify the NJDEP at least 30 days prior to disposal in writing. Letters should be addressed to the individual requesting the work with a copy to the NJDEP Contract Administrator (A. Fishman)

Organic extracts must be kept refrigerated at less than 4°C. Unused sample volumes need not to be refrigerated.

NYSDEC

Unused sample volumes must be held for 90 days after data submission. Samples do not require refrigeration after analysis.

Organic sample extracts must be stored at less than 4°C for 365 days after data submission.

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STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDE/PCB ANALYSIS

EX:062389:2

300909

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

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Revision History

Code	Revision Date	Reason
EX:062389:0	6/23/89	Original Issue.
EX:062389:1	2/12/90	Update to NJDEP & NYSEDC89 requirements.
EX:062389:2	3/26/91	Change to IEA forms and minor text changes.

STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDE/PCB ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 The analyst is referred to the 02/88 version of the "USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media/Multi-Concentration for further information about these procedures.

1.2 This procedure is applicable to the analysis of water samples.

1.3 It is the policy of IEA, Inc. and of the Extractions Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the IEA, Inc. Policy Statement on Business Ethics and Conduct.

2.0 SUMMARY OF METHOD

Water samples are serially extracted in a separatory funnel using methylene chloride. The extract is dried using sodium sulfate, and concentrated using a Kuderna-Danish (K-D) apparatus.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus. This can lead to discrete artifacts and/or elevated baselines in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the running of reagent blanks. A specific interference that can cause a problem is the presence of phthalate esters, which are commonly found in plastics. Those interferences can be avoided by not using plastics in the laboratory.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

4.0 APPARATUS AND MATERIALS

4.1 Glassware

4.1.1 Separatory funnel, 2,000 ml with teflon stopcock.

4.1.2 Drying column, chromatographic column approximately 400 mm long x 19 mm ID.

4.1.3 Concentrator tube, K-D, 10 ml, graduated.

4.1.4 Evaporative flask, K-D, 500 ml.

4.1.5 Snyder column, three ball macro.

4.1.6 Vials, 10 to 20 ml capacity with teflon lined screw cap.

4.1.7 Alumina column, chromatographic column approximately 200 mm long x 8 mm ID.

4.1.8 Erlenmeyer flask, 250 or 500 ml.

4.1.9 Graduated cylinder, 1,000 ml.

4.2 Pyrex glass wool, extracted in ultrasonic cleaner with methylene chloride for 30 minutes and dried at 180°C.

4.3 Silicon carbide boiling chips, approximately 10/40 mesh, extracted in ultrasonic cleaner with methylene chloride for 30 minutes and dried at 180°C.

4.4 Water bath, heated, should be in a hood.

4.5 Balance, analytical, capable of weighing ±0.0001 g.

4.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.

4.7 Chlorine test kit, capable of detecting 0.1 ppm residual chlorine.

5.0 REAGENTS

5.1 Reagent water - Water in which an interferant is not observed at the method detection limit for each analytical parameter.

5.2 Acetone, hexane, methylene chloride, - Pesticide quality or equivalent.

5.3 Sodium sulfate - (ACS) granular anhydrous, purify by extracting in methylene chloride for 30 minutes and place in the drying oven overnight at 180°C.

5.4 Alumina, Super I Woelm or equivalent, prepare by adding 7 percent (v/w) reagent water and tumble for a minimum of 2 hours. There should be no lumps present.

5.4.1 Each batch must be assayed using the alumina equivalency test. If the batch does not meet criteria, it may be deactivated with up to 9 percent water.

5.5 Sodium hydroxide solution, (10N) - Dissolve 40 g NaOH in reagent water and dilute to 100 ml.

5.6 Tetrabutylammonium (TBA) - Sulfite reagent. Dissolve 3.39 g of TBA in 100 mls of reagent water. Extract three times with 20 ml portions of hexane. Discard the hexane extracts, and add 25 g of sodium sulfite to the water solution. The resulting solution can be stored at room temperature for at least one month in an amber bottle with a teflon screw cap.

5.7 Sulfuric acid solution (1+1) - (ACS) Slowly add 50 ml sulfuric acid to 50 ml of reagent water.

5.8 Dibutylchlorendate (DBC) surrogate solution (for waters) - Prepare DBC solution at a concentration of 1 ug/ml in methanol. The solution must be stored at 4°C (±2°C) in teflon sealed containers for a maximum of 12 months.

5.8.1 Add 1.0 ml of the DBC solution to all samples, blanks and matrix spikes.

5.8.2 The solution must be stored at 4°C (± 2°C) for a maximum of 12 months in a sealed container with a teflon lined cap.

5.9 Pesticide matrix spiking solution.

5.9.1 The spiking solution will contain the following pesticides at the specified concentrations in methanol:

Pesticide	Concentration, ppm
Lindane	0.2
Heptachlor	0.2
Aldrin	0.2
Dieldrin	0.5
Endrin	0.5
4,4'DDT	0.5

5.9.2 Matrix spikes and matrix spike duplicates will get 1.0 ml of the pesticide spiking solution.

5.9.3 The solution must be stored at 4°C (± 2°C) for a maximum of 12 months in a sealed container with a teflon lined cap.

6.0 SAMPLE PRESERVATION AND STORAGE

6.1 Preservation Requirements

6.1.1 Water samples must be protected from light and refrigerated at 4°C from the time of receipt until extraction and analysis.

6.2 Holding Times

6.2.1 CLP water samples shall be extracted within 5 days of Validated Time of Sample Receipt (VTSR), and SOP water samples shall be extracted within 7 days of collection. NYSDEC89 samples shall be concentrated within 5 days of VTSR.

a) If SOP water samples are not received within 1 day of collection, the Extractions Group Leader and the Organics Manager must be notified. A CAR will be issued to Client Services to contact the client for instructions on how to proceed.

7.0 SAMPLE ANALYSIS

7.1 Safety

7.1.1 Analysts shall treat all samples as if they are hazardous and take all appropriate safety precautions, including but not limited to safety glasses, lab coats and impervious gloves.

7.2 Chain of Custody Procedures

7.2.1 All analysts are responsible for maintaining sample custody in accordance with EPA guidelines.

7.2.2 All samples must be signed out on the chain of custody forms located in sample control.

7.3 Water Sample Extraction Procedure - Separatory Funnel

7.3.1 When the sample is brought to the laboratory from sample control, the extraction logbook for the batch is started. A copy of the logbook page is labeled Figure 1.0. The appropriate information is entered into blocks 1 and 3.

7.3.2 The pH of the sample is checked using wide range pH paper. If the pH is outside the range of 5-9, a Corrective Action Report (CAR) is filled out. A copy of a CAR is labeled Figure 2.0. The pH is adjusted to be in the range of 5-9 using 1:1 sulfuric acid or 10 N sodium hydroxide.

7.3.3 The chlorine residual of the sample is checked using the chlorine test kit. If a residual is detected, a CAR is filled out.

7.3.4 If the sample is submitted in a 1 liter bottle, the level of the sample is marked on the side of the bottle with an indelible marker so the volume can be determined. The sample is mixed to ensure homogeneity, and transferred to a 2 liter separatory funnel. If the sample is submitted in a larger volume bottle, a pre-cleaned graduated cylinder is used to measure 1 liter of well mixed sample. The appropriate information is entered into block 4 of the

extraction logbook.

7.3.5 With every batch of samples processed, a blank must be run. The blank will consist of 1 liter of reagent water.

7.3.6 Tracking labels (see Figure 3.0) or an equivalent system must be used for every sample and blank.

7.3.7 Add 1,000 ul of DBC for Waters surrogate to every blank, sample and matrix spike/matrix spike duplicate (MS/MSD).

7.3.8 If an MS/MSD is to be run with a batch, add 1,000 ul of pesticide spike solution to the MS and the MSD.

a) For the NYSDEC 89 protocol prepare a blank MS every time a MS/MSD is extracted by adding 1000 ul of Pesticide Matrix spike solution to a liter of reagent water.

7.3.9 Fill in the appropriate information in block 5 of the extraction logbook.

7.3.10 Add 60 ml of methylene chloride directly to the separatory funnel. Extract the sample by shaking the funnel for 2 minutes with periodic venting to release the excess pressure. Allow the organic layer to separate from the water phase for at least 10 minutes. If an emulsion forms at the interface between the two layers, and it is greater than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to break the emulsion.

a) Acceptable mechanical techniques for breaking an emulsion include: stirring, filtering the emulsion through glass wool, or centrifugation.

b) If the emulsion cannot be broken, it will be necessary to use a continuous liquid-liquid extractor. Details on the procedure can be obtained in the liquid-liquid extraction procedure in section 7.4.

7.3.11 The organic layer is then collected in a labeled 250 ml erlenmeyer flask.

a) The label should include the sample number, the parameter analyzed and the date. Colored tape is used for the label.

7.3.12 The extraction is repeated twice more, using fresh 60 ml portions of methylene chloride. The extracts are combined in the erlenmeyer flask.

7.3.13 Assemble a K-D concentrator by attaching a 10 ml concentrator tube to a 500 ml evaporative flask. Transfer the tape label to the flask.

7.3.14 Prepare a drying column by placing a plug of glass wool in the bottom of the column and filling it with approximately 10 cm of anhydrous granular sodium sulfate. Rinse the column with methylene chloride, and discard the rinse.

7.3.15 Pour the combined extract through the drying column into the K-D concentrator. Rinse the erlenmeyer with 20-30 mls of methylene chloride to quantitatively transfer the sample.

7.3.16 Add one or two clean boiling chips to the K-D and attach a three ball snyder column. Pre-wet the snyder column with about 1 ml methylene chloride. Place the K-D in a hot water bath (80-90°C). The temperature should be such that the concentration is completed within 15 minutes. When the apparent volume reaches 2 - 3 mls, remove the K-D.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

7.3.17 Add 50 mls of hexane through the top of the Snyder column, and concentrate as above. When the apparent volume reaches 2 - 3 mls, remove from the bath and allow the K-D to cool for at least 10 minutes.

7.3.18 Remove the Snyder column, and rinse the flask and its lower joint into the concentrator tube with hexane. If sulfur crystals are noted, it will be necessary to perform a sulfur cleanup (as detailed below) on the sample.

a) Place the concentrator tube on the N-Evap and blow the sample down to 1 ml.

b) Transfer the 1 ml to a 22 ml vial with a teflon lined screw cap and rinse the concentrator tube with 1 ml of hexane. Transfer the washings to the bottle.

c) Add 1 ml of TBA-Sulfite reagent and 2 ml of 2-propanol. Shake for 1 minute. If the solution clears, add crystalline sodium sulfite (in approximately 100 mg portions) until a solid residue remains.

d) Add 5 ml of distilled water and shake for at least 1 minute. Allow the sample to stand for at least 10 minutes. Transfer the hexane layer (top) to a concentrator tube and continue with the procedure.

7.3.19 If the sample is CLP level, prepare the sample for alumina cleanup by placing the concentrator tube in the N-Evap which has a water bath maintained at 35°C. Evaporate the solvent volume to 0.5 ml using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the bath. The extract must never be allowed to go dry. Dilute the extract to 1 ml with acetone,and follow the procedure outlined below. If the sample is SOP, bring the extract to a final volume of 10 mls with hexane in a receiving vessel.

a) Add 3 g activity III alumina to the 10 ml chromatographic column. Tap the column to settle the alumina. Do not pre-wet the alumina. Place a clean 10 ml concentrator tube beneath the alumina column to collect the eluate.

b) Transfer the acetone/hexane extract to the top of the alumina using a disposable pasteur pipet. Add 1 ml of hexane to the column, and rinse the original concentrator tube with hexane and transfer the rinsings to the top of the alumina column. Elute the column with additional hexane to make a final volume of 10 mls. Do not allow the column to go dry during the elution.

c) If necessary, place the concentrator tube on the N-Evap and blow the sample down. Bring up to a final volume of 10 mls in a receiving vessel.

7.3.20 Transfer the extract to a labeled teflon lined screw cap vial.

7.3.21 Fill in the appropriate spaces in block 6 of the extractions logbook.

7.3.22 The PCB/Pesticide extract is now ready for analysis. Place the extract in the appropriate freezer in sample control. Fill out block 8 of the extraction logbook. A copy of labels with the appropriate information is included as Figure 4.0.

7.4 Sample Extraction - Continuous Liquid/Liquid Extraction

7.4.1 Transfer the emulsified sample to the continuous extractor and add 120 mls methylene chloride. A reagent water blank with surrogates added must accompany each batch of samples extracted.

7.4.2 Add 500 mls of methylene chloride to the boiling flask and extract for 18 hours. Allow the flask to cool and remove the labeled boiling flask.

7.4.3 Dry and concentrate the sample as in 7.3 above.

8.0 QUALITY CONTROL

8.1 A blank will be run with every batch. A batch is defined as a group of samples for an analysis run within a 24 hour period. A batch can have a maximum of 20 samples.

8.2 A matrix spike/matrix spike duplicate will be run at a frequency of 1 sample in 20. However, if more than 14 days elapses and 20 samples have not been run, it will be necessary to run a MS/MSD. For samples extracted using the NYSDEC 89 protocol it is also necessary to extract a Blank MS every time a MS/MSD is extracted.

8.3 The extractions Batch Approval Sheets must be filled out on a daily basis. They are located on the back of the extractions logbook page. A copy of the sheet is included as Figure 5.0

8.4 A case narrative must be filled out for every batch assigned on a daily basis. There is space provided on the Batch Approval Form for the case narrative. If the information requires transmission to other groups, a CAR should be used. Final case narratives will also be included in the client job folder. An example of a Final Case Narrative is provided as figure 6.0.

8.5 The Extractions Logbook and the tracking log must be completely filled out on a daily basis for every batch extracted or concentrated.

8.6 The Extractions Group Leader or his designee will fill out the Extractions QCAR (Figure 7.0) for inclusion in each clients job folder.

9.0 METHOD PERFORMANCE

9.1 The method performance is determined by the final measurement results of each sample and the accuracy of surrogate and MS/MSD measurement results.

10.0 DATA REDUCTION AND HANDLING

Not Applicable

BLOCK 1

BLOCK 2

BLOCK 3 | BLOCK 4 | BLOCK 5 | BLOCK 6 | BLOCK 7 | BLOCK 8

FIGURE 1.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX: (203) 268-5348

Reported By: _____

Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE REPORT

Client: _____ Job Number: _____ Sample Number(s): _____

Problem Reported By: _____

Category of Problem: _____
Poor Communication With ... Sample Matrix Instruc...

Description of Problem: _____

Use back of CAR/CNR or attached sheet if more space is required

Recommended Corrective Action(s): _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline For CAR: _____ Reply to Originator Required? [ ]

Actual Corrective Action(s) Taken: _____
Document Actual Corrective Actions On T...

Form and Send a Copy of Completed CAR/CNR to QA Officer and Job Fold...

_____
Signature and Date

Is this a recurring Problem? _____ Should SOP be Modified or Updated? ___

Initial Lab manager, Marketing Manager, Branch Manager, QA Offic...
Distribution: Client Services, Job Folder, Other: _____
SECONDARY DISTRIBUTION: ORIGINATOR, QA OFFICER, OTHER: _____

Document Problem and Corrective Action(s) In Case Narrative: [ ]
Attachments: Telephone Log, Services Change Request, Memo, Other:

FIGURE 2.0

Air Liquid...
New Jersey
201-428-6181

Schaumburg,
Illinois
708-705-0740

Miramar,
Florida
407-889-0828

Essex Junction,
Vermont
802-878-5138

N. Billerica,
Massachusetts
617-272-5212

Cary,
North Caro...
919-677-...390

300922

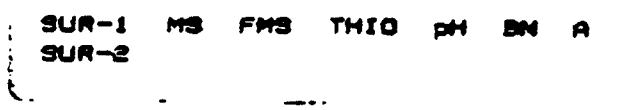
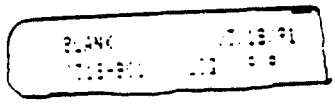


FIGURE 3.0



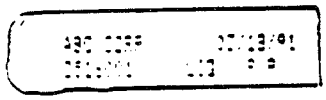


FIGURE 4.0

IEA CT

EXTRACTIONS BATCH APPROVAL FORM

Parameter _____ Matrix _____

Blank ID. _____

		Extraction Init/Date	Concentrate Init/Date
1)	All required QA/QC was performed or referenced	_____	_____
2)	Extraction logbook was completed	_____	_____
3)	The sample tracking log has been updated	_____	_____
4)	Case narrative was completed	_____	_____
5)	Was a corrective action form submitted?	_____	_____
6)	The assignment was performed according to the SOP and all exceptions have been documented	_____	_____

CASE NARRATIVE

_____

_____

_____

_____

_____

_____

_____

Final Approval: _____

FIGURE 5.0

IEA - CT
FINAL
EXTRACTIONS CASE NARRATIVE

CLIENT NAME : XYZ Corp. CASE NUMBER: 3091-0573

DATE: 3/19/91 SAMPLE NUMBERS:0573001 - 0

B/P/P

All samples were extracted and concentrated without any apparent problems, except as noted below. Sample 0573015 screened with low acid surrogates. The sample was run by Mass Spec, and the screening results were confirmed. The sample was re-extracted on 2/20/91, which was within holding time. The results of the re-extraction confirmed matrix problems.

CASE NARRATOR J Burtt DATE 3/15/91

APPROVED BY J Burtt DATE 3/15/91

Be as specific and detailed as possible. Please be sure to include any conversations with the client

FIGURE 6.0

300926

IEA CT

EXTRACTIONS QUALITY CONTROL APPROVAL REPORT

Client: _____ Job Number: _____

Parameter: _____ Samples: _____

		Init / Date
1)	Batch Approval Form is complete	_____
2)	Holding times are met	_____
3)	Case Narrative has been completed	_____
4)	Copies of corrective actions made	_____
5)	Copies of any required data made	_____
6)	Logbook has been checked	_____

These extractions have been performed in accordance with the extractions SOP unless otherwise noted in the case narrative.

Final Approval: _____ Date: _____

FIGURE 7.0

300927

STANDARD OPERATING PROCEDURE
FOR THE PREPARATION OF AQUEOUS SAMPLES
BASE-NEUTRAL/ACID EXTRACTABLE ORGANIC COMPOUNDS

1.0 SCOPE AND APPLICATION

1.1 The analyst is referred to the 02/88 version of the "USEPA Contract Laboratory Program Statement of Work for Organics Multi-Media, Multi-Concentration" for further information about these procedures.

1.2 This procedure is applicable to the analysis of water samples.

1.3 It is the policy of IEA, Inc. and of the Extractions Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the IEA, Inc. Policy Statement on Business Ethics and Conduct.

2.0 SUMMARY OF METHOD

Water samples are serially extracted in a separatory funnel using methylene chloride at a pH >11 and again at a pH of <2. The extract is dried using sodium sulfate, and concentrated using a Kuderna-Danish (K-D) apparatus.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus. This can lead to discrete artifacts and/or elevated baselines in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the measurement of reagent blanks. Interferences that can cause contamination problems are the presence of phthalate esters, which are commonly found in plastics. Those interferences can be avoided by using clean solvents and not permitting samples to contact plastics in the laboratory.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

4.0 APPARATUS AND MATERIALS

4.1 Glassware

4.1.1 Separatory funnel, 2000 ml with teflon stopcock.

4.1.2 Drying column, chromatographic column approx. 400 mm long x 19 mm ID.

4.1.3 Concentrator tube, K-D, 10 ml, graduated.

4.1.4 Evaporative flask, K-D, 500 ml.

4.1.5 Snyder column, three ball macro.

4.1.6 Vials, 1.8 ml capacity with teflon lined screw cap.

4.1.7 Erlenmeyer flask, 250 or 500 ml.

4.1.8 Graduated cylinder, 1,000 ml.

4.2 Pyrex glass wool, extracted in ultrasonic cleaner with methylene chloride for 30 minutes and dried at 180°C.

4.3 Silicon carbide boiling chips, approximately 10/40 mesh, extracted in ultrasonic cleaner with methylene chloride for 30 minutes and dried at 180°C.

4.4 Water bath, heated, should be in a hood.

4.5 Balance, analytical, capable of weighing ±0.0001 g.

4.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.

4.7 Chlorine test kit, capable of detecting 0.1 ppm residual chlorine.

5.0 REAGENTS

5.1 Reagent Water - Water in which an interferant is not observed at the method detection limit for each analytical parameter.

5.2 Acetone, Methanol, Methylene Chloride - Pesticide quality or equivalent.

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF AQUEOUS SAMPLES FOR BNA ANALYSIS

EX:062689:2

Miramar, Florida 305-949-0929
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-879-5138

TABLE OF CONTENTS

Revision History

Code	Revision Date	Reason
EX:062689:0	6/26/89	Original Issue.
EX:062689:1	2/12/90	Update to NJDEP & NYSEDC89 requirements.
EX:062689:2	4/2/91	Change to IEA forms and minor text revisions.

5.3 Sodium Sulfate - (ACS) granular anhydrous, purify by extracting in methylene chloride for 30 minutes and place in the drying oven overnight at 180°C.

5.4 Sodium Thiosulfate - (ACS) - 10% solution.

5.5 Sodium Hydroxide Solution, (10N) - Dissolve 40 g NaOH in reagent water and dilute to 100 ml.

5.6 Sulfuric Acid Solution (1+1) - (ACS) Slowly add 50 ml sulfuric acid to 50 ml of reagent water.

5.7 Base Neutral/Acid Surrogate Solution.

5.7.1 The BNA surrogate will contain the following compounds at the specified concentrations in methanol:

Surrogates	Concentration, ppm
Nitrobenzene-d_5	100
Terphenyl-d_{14}	100
2-fluorobiphenyl	100
2,4,6-tribromophenol	200
2-fluorophenol	200
Phenol-d_5	200

5.7.2 Add 100 ul of the BNA surrogate solution to all samples, blanks, and matrix spike/matrix spike duplicates.

5.7.3 The solution must be stored at 4°C (± 2°C) for a maximum of 12 months in a sealed container with a teflon lined cap.

5.8 Base Neutral/Acid Matrix Spiking Solution.

5.8.1 The BNA spiking solution will contain the following compounds at the specified concentration in methanol.

Compound	Concentration, ppm
1,2,4-trichlorobenzene	100
Acenaphthene	100
2,4-dinitrotoluene	100
Pyrene	100

N-nitroso-di-n-propylamine	100
1,4-dichlorobenzene	100
Pentachlorophenol	200
Phenol	200
2-chlorophenol	200
4-chloro-3-methylphenol	200
4-nitrophenol	200

5.8.2 The matrix spike and the matrix spike duplicate (MS/MSD) will each get 1.0 ml of the BNA matrix spiking solution.

5.8.3 The solution must be stored at 4°C (± 2°C) for a maximum of 12 months in a sealed container with a teflon lined cap.

6.0 SAMPLE PRESERVATION AND STORAGE

6.1 Preservation Requirements

6.1.1 Water samples must be protected from light and refrigerated at 4°C from the time of receipt until extraction and analysis.

6.2 Holding Times

6.2.1 CLP water samples shall be extracted within 5 days of receipt, and SOP water samples shall be extracted within 7 days of collection. NYSDEC 89 samples shall be concentrated within 5 days VTSR.

a) If SOP water samples are not received within 1 day of collection, the Extractions Group Leader and the Organics Manager must be notified. A CAR will be issued to Client Services to contact the client for instructions on how to proceed.

7.0 SAMPLE ANALYSIS

7.1 Safety

7.1.1 Analysts shall treat all samples as if they are hazardous and take all appropriate safety precautions, including but not limited to safety glasses, lab coats and impervious gloves.

7.2 Chain of Custody Procedures

7.2.1 All analysts are responsible for maintaining sample custody in accordance with EPA guidelines.

7.2.2 All samples must be signed out on the chain of custody forms located in sample control.

7.3 Water Sample Extraction\Concentration Procedure-Separatory Funnel

7.3.1 When the sample is brought to the laboratory from sample control, the extraction logbook for the batch is started. A copy of the logbook page is labeled Figure 1. The appropriate information is entered into blocks 1 through 3.

7.3.2 The pH of the sample is checked using wide range pH paper. If the pH is outside the range of 5-9, a Corrective Action Report (CAR) is filled out. A copy of a CAR is labeled Figure 2.

7.3.3 The chlorine residual of the sample is checked using the chlorine test kit. If a residual is detected a CAR is filled out, and client services is notified.

a) For SOP samples, the chlorine residual is neutralized using 500 ul of a 10 percent sodium thiosulfate solution. If after the thiosulfate is added, a chlorine residual still exists a second 500 ul aliquot of thiosulfate is added to the sample. If that does not neutralize the chlorine, this is noted on the CAR and in the case narrative. The extraction is continued.

b) For CLP level samples, a second 1 liter aliquot of the sample will be extracted concurrently with the original sample.

7.3.4 If the sample is submitted in a 1 liter bottle, the level of the sample is marked on the side of the bottle with an indelible marker so the volume can be measured at a later time. The sample is mixed to ensure homogeneity, and transferred to a 2 liter separatory funnel. If the sample is submitted in a larger volume bottle, a pre-cleaned graduated cylinder is used to measure 1 liter of sample. The appropriate information is entered into block 4 of the extraction logbook.

7.3.5 With every batch of samples processed, a blank must be run. The blank will consist of 1 liter of reagent water.

7.3.6 Tracking labels (see Figure 3) or an equivalent system must be used for every sample and blank.

7.3.7 Add 1,000 ul of BNA surrogate to every blank, sample and MS/MSD.

7.3.8 If an MS/MSD is to be run with a batch, add 1,000 ul of BNA spike solution to the MS and the MSD.

a) For the NYSDEC 89 protocol prepare a blank MS every time a MS/MSD is extracted by adding 1000 ul of BNA spike solution to a liter of reagent water.

7.3.9 Fill in the appropriate information in block 5 of the extraction logbook.

7.3.10 Using 10N sodium hydroxide adjust the samples to a pH >11. Check the pH with wide range pH paper.

7.3.11 Add 60 ml of methylene chloride directly to the separatory funnel. Extract the sample by shaking the funnel for two minutes with periodic venting to release the excess pressure. Allow the organic and aqueous phases to separate for at least 10 minutes. If an emulsion forms at the interface between the two layers, and it is greater than one-third the volume of the solvent layer, mechanical techniques must be used to break the emulsion.

a) acceptable mechanical techniques for breaking an emulsion include: stirring, filtering the emulsion through glass wool, or centrifugation.

b) If the emulsion cannot be broken, it will be necessary to use a continuous liquid-liquid extractor. Details on the procedure can be obtained in the liquid-liquid extraction section following.

7.3.12 The organic layer is then collected in a labeled 250 ml erlenmeyer flask. Colored tape is used for the label.

a) The label should include the sample number, the parameter analyzed and the date.

7.3.13 The extraction is repeated twice more, using fresh 60 ml portions of methylene chloride. The extracts are combined in the erlenmeyer flask.

7.3.14 Adjust the pH of the aqueous phase to <2 using 1-1 sulfuric acid. Check the pH with wide range pH paper.

7.3.15 The extraction is repeated three more times using fresh 60 ml portions of methylene chloride. The acid extracts are combined in a separate, labeled erlenmeyer flask.

7.3.16 Assemble a K-D concentrator for each fraction by attaching a 10 ml concentrator tube to a 500 ml evaporative flask, and transfer the tape label.

7.3.17 Prepare a drying column for each fraction by placing a plug of glass wool in the bottom of the column and filling it with approximately 10 cm of anhydrous granular sodium sulfate. Rinse the column with methylene chloride, and discard the rinse.

7.3.18 Pour the extract from each fraction through its drying column into its K-D concentrator. Rinse the erlenmeyer with 20-30 mls of methylene chloride to quantitatively transfer the sample.

7.3.19 Add one or two clean boiling chips to the K-D and attach a three ball snyder column. Pre-wet the Snyder column with about 1 ml methylene chloride. Place the K-D in a hot water bath (80 to 90°C). The temperature should be such that the concentration is completed within 15 minutes. When the apparent volume reaches 2 - 3 mls, remove the K-D and allow it to drain and cool for 10 minutes.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

7.3.20 Remove the Snyder column, and rinse the flask and its lower joint into the concentrator tube with methylene chloride. Transfer the label to the recieving vessel. Place the concentrator tube in the N-Evap which has a water bath maintained at about 35°C. Evaporate the solvent volume to <1 ml using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the bath. The extract must never be allowed to go dry. Bring up to a final volume of 1.0 ml in the recieving vessel using methylene chloride.

7.3.21 Transfer the extract from each fraction to a separate labeled teflon lined screw cap vial. A copy of labels with the appropriate information are included as Figure 4.0.

7.3.22 Fill in the appropriate spaces in block 6 of the extractions logbook.

7.3.23 The base neutral and acid extracts are now ready for analysis. Place the extracts in the appropriate freezer in sample control. Fill out block 8 of the extraction logbook.

7.4 Sample Extraction - Continuous Liquid/Liquid Extraction

7.4.1 Transfer the emulsified base neutral fraction to the continuous extractor and add 120 mls methylene chloride. A reagent water blank with surrogates added, and the pH adjusted to >11 must accompany each batch of samples extracted.

a) The initial attempt at extraction in a separatory funnel is not mandatory. A sample may be extracted initially using this procedure. Surrogates must be added, and the pH must be adjusted to >11 prior to beginning the extraction process.

7.4.2 Add 500 mls of methylene chloride to the boiling flask and extract for 18 hours. Allow the flask to cool and remove the labeled boiling flask.

7.4.3 Add 500 ml of methylene chloride to a clean boiling flask and attach to the extractor.

7.4.4 Adjust the pH of the aqueous phase to <2 using 1-1 sulfuric acid, and extract for 18 hours.

7.4.5 Dry and concentrate the base neutral and acid fractions as in 7.3 above.

8.0 QUALITY CONTROL

8.1 A blank will be run with every batch. A batch is defined as a group of samples for an analysis run within a 24 hour period. A batch can have a maximum of 20 samples.

8.2 A matrix spike/matrix spike duplicate will be run at a frequency of 1 sample in 20. However, if more than 14 days elapses and 20 samples have not been run, it will be necessary to run a MS/MSD. For samples extracted using the NYSDEC 89 protocol it is also necessary to extract a Blank MS every time a MS/MSD is extracted.

8.3 The extractions Batch Approval Sheets must be filled out on a daily basis. They are located on the back of the extractions logbook page. A copy of the sheet is included as Figure 5.0

8.4 A case narrative must be filled out for every batch assigned on a daily basis. There is space provided on the Batch Approval Form for the case narrative. If the information requires transmission to other groups, a CAR should be used. Final case narratives will also be included in the client job folder. An example of a Final Case Narrative is provided as figure 6.0.

8.5 The Extractions Logbook and the tracking log must be completely filled out on a daily basis for every batch extracted or concentrated.

8.6 The Extractions Group Leader or his designee will fill out the Extractions QCAR (Figure 7.0) for inclusion in each clients job folder.

9.0 METHOD PERFORMANCE

9.1 The method performance is determined by the final measurement results of each sample and the accuracy of surrogate and MS/MSD measurement results.

10.0 DATA REDUCTION AND HANDLING

Not Applicable

BLOCK 1

ANALYSIS ____________

CORRESPONDING QS/QSD ____________

IEA

ORGANIC EXTRACTION LOG

AQUEOUS/SOLID MATRIX

PAGE #

BLOCK 2

EXTRACTION DATE ____________

FINISH DATE ____________

SURROGATE CODE ____________

SPIKE CODE ____________

APPROVED BY ____________

BLOCK 3				BLOCK 4				BLOCK 5					BLOCK 6					BLOCK 7						BLOCK 8				
																			% SOLIDS DATA				LOCATION					
LOG ENT	CLIENT	LAB ID	SIGN OUT COC	CL2 RES	INIT pH H2O	INIT WT/VOL	SAMPLE COLOR & TEXTURE	SURR INIT	SPIKE INIT	EXT METHOD	METH 01 SCAN	EXT BY	C/N	EXT TO DATE	UE	COLOR OF EXT	CONC/ SCORED BY	pH	TARE	WET WT	DRY WT	% SOLIDS	FIR	TRAY	SLOT	METH 02 SCAN	COMMENTS

FIGURE 1.0

an environmental testing company
320 Monroe Turnpike
Monroe, Connecticut 06468
203) 261-4458
FAX: 203) 268-5348

Reported By: _____ Group

Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE REPO.

Client: _____ Job Number: _____ Sample Number(s): _____

Problem Reported By: _____

Category of Problem: _____
Poor Communication With ... Sample Matrix Instruc...

Description of Problem: _____

Use back of CAR/CNR or attached sheet if more space is required

Recommended Corrective Action(s): _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline For CAR: _____ Reply to Originator Required? [ ]

Actual Corrective Action(s) Taken: _____
Document Actual Corrective Actions On Th...

Form and Send a Copy of Completed CAR/CNR to QA Officer and Job Folder

_____
Signature and Date

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Initial Lab manager, Marketing Manager, Branch Manager, QA Officer
Distribution: Client Services, Job Folder, Other: _____
SECONDARY DISTRIBUTION: ORIGINATOR, QA OFFICER, OTHER: _____

Document Problem and Corrective Action(s) In Case Narrative: [ ]
Attachments: Telephone Log, Services Change Request, Memo, Other:

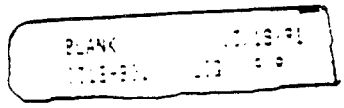
FIGURE 2.0

300941

SUR-1 MS FMS THIO pH BN A
SUR-2

FIGURE 3.0

300942



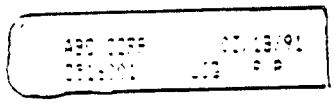


FIGURE 4.0

IEA C.

EXTRACTIONS BATCH APPROVAL FORM

Parameter _____ Matrix _____

Blank ID. _____

		Extraction Init/Date	Concentrate Init/Date
1)	All required QA/QC was performed or referenced	_____	_____
2)	Extraction logbook was completed	_____	_____
3)	The sample tracking log has been updated	_____	_____
4)	Case narrative was completed	_____	_____
5)	Was a corrective action form submitted?	_____	_____
6)	The assignment was performed according to the SOP and all exceptions have been documented	_____	_____

CASE NARRATIVE

_____

_____

_____

_____

_____

_____

_____

Final Approval: _____

FIGURE 5.0

IEA - CT
FINAL
EXTRACTIONS CASE NARRATIVE

CLIENT NAME : XYZ Corp. CASE NUMBER: 3091-0573

DATE: 3/19/91 SAMPLE NUMBERS:0573001 - 00

B/P/P

All samples were extracted and concentrated without any apparent problems, except as noted below. Sample 0573015 screened with low acid surrogates. The sample was run by Mass Spec, and the screening results were confirmed. The sample was re-extracted on 2/20/91, which was within holding time. The results of the re-extraction confirmed matrix problems.

CASE NARRATOR J Butt DATE 3/18

APPROVED BY J Butt DATE 3/18/91

Be as specific and detailed as possible. Please be sure to include any conversations with the client

FIGURE 6.0

300945

IEA CT

EXTRA ONS QUALITY CONTROL APPROVAL REPORT

Client: _____ Job Number: _____

Parameter: _____ Samples: _____

		Init / Date
1)	Batch Approval Form is complete	_____
2)	Holding times are met	_____
3)	Case Narrative has been completed	_____
4)	Copies of corrective actions made	_____
5)	Copies of any required data made	_____
6)	Logbook has been checked	_____

These extractions have been performed in accordance with the extractions SOP unless otherwise noted in the case narrative.

Final Approval: _____ Date: _____

FIGURE 7.0

300946

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX: (203) 268-5346

STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF SOIL/SEDIMENT SAMPLES FOR BNA/PESTICIDE/PCB ANALYSIS

EX:061689:2

300947

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

TABLE OF CONTENTS

Revision History

Code	Revision Date	Reason
EX:061689:0	6/16/89	Original Issue.
EX:061689:1	2/12/90	Update to NJDEP & NYSEDC89 requirements.
EX:061689:2	3/18/91	Change to IEA forms and minor text changes.

IEA - CT
FINAL
EXTRACTIONS CASE NARRATIVE

CLIENT NAME : XYZ Corp. CASE NUMBER: 3091-0573

DATE: 3/19/91 SAMPLE NUMBERS: 0573001 - 20

B/P/P

All samples were extracted and concentrated without any apparent problems, except as noted below. Sample 0573015 screened with low acid surrogates. The sample was run by Mass Spec, and the screening results were confirmed. The sample was re-extracted on 2/20/91, which was within holding time. The results of the re-extraction confirmed matrix problems.

CASE NARRATOR J Butt DATE 3/11/91

APPROVED BY J Butt DATE 3/11/91

Be as specific and detailed as possible. Please be sure to include any conversations with the client

300949

FIGURE 6.0

IEA CT

EXTRACTIONS QUALITY CONTROL APPROVAL REPORT

Client: _____ Job Number: _____

Parameter: _____ Samples: _____

		Init / Date
1)	Batch Approval Form is complete	_____
2)	Holding times are met	_____
3)	Case Narrative has been completed	_____
4)	Copies of corrective actions made	_____
5)	Copies of any required data made	_____
6)	Logbook has been checked	_____

These extractions have been performed in accordance with the extractions SOP unless otherwise noted in the case narrative.

Final Approval: _____ Date: _____

FIGURE 7.0

STANDARD OPERATING PROCEDURE FOR THE PREPARATION OF SOIL/SEDIMENT SAMPLES FOR BNA/PESTICIDE/PCB ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 The analyst is referred to the 2/88 version of the "USEPA Contract Laboratory Program Statement of Work for Organics analysis Multi-Media, Multi-Concentration for further information about these procedures.

1.2 This procedure is applicable to the analysis of soil/sediment samples.

1.3 It is the policy of IEA, Inc. and of the Extractions Group to ensure that we administer contracts and orders for goods and services in a manner that is fully compliant with governmental laws and regulations, as well as the IEA, Inc. Policy Statement on Business Ethics and Conduct.

2.0 SUMMARY OF METHOD

Soil/sediment samples are serially extracted using an ultrasonic probe with 1:1 methylene chloride/acetone. The extract is concentrated using a Kuderna-Danish (K-D) apparatus. The extract can be split and analyzed for semi-volatiles as well as pesticide/PCB's.

3.0 INTERFERENCES

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing apparatus. This can lead to discrete artifacts and/or elevated baselines in the gas chromatograph. All these materials must be demonstrated to be free from interferences by the measurement of reagent blanks. Interferences that can cause contamination problems are the presence of phthalate esters, which are commonly found in plastics. Those interferences can be avoided by using clean solvents and not permitting samples to contact plastics in the laboratory.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source.

4.0 APPARATUS AND MATERIALS

4.1 Glassware

4.1.1 Glass beakers, 400 ml

4.1.2 Side arm flask, 500 ml.

4.1.3 Concentrator tube, K-D, 10 ml, graduated.

4.1.4 Evaporative flask, K-D, 500 ml.

4.1.5 Snyder column, three ball macro

4.1.6 Vials, 1.7 to 15 ml capacity with teflon lined screw cap.

4.1.7 Alumina column, chromatographic column approximately 200 mm long x 8 mm ID.

4.1.8 Buchner funnels, 100 mm diameter.

4.1.9 Disposable pasteur pipets, cotton plugged.

4.2 Pyrex glass wool, extracted in ultrasonic cleaner with methylene chloride for 30 minutes and dried at 180°C.

4.3 Silicon carbide boiling chips, approximately 10/40 mesh, extracted in ultrasonic cleaner with methylene chloride for 30 minutes and dried at 180°C.

4.4 Water bath, heated, should be in a hood

4.5 Balance, analytical, capable of weighing ±0.0001 g.

4.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40°C.

4.7 Sonicator, equipped with 3/4" and 1/8" disrupter horns and a minimum of 375 watt capability.

4.8 Oven, drying, capable of maintaining 104 ± 2°C.

4.9 Desiccator

4.10 Weighing dishes

4.11 Whatman GFA Filter Paper, 100 mm (or equivalent)

5.0 REAGENTS

5.1 Reagent water - Water in which an interferant is not observed at the method detection limit for each analytical parameter.

5.2 Acetone, hexane, methylene chloride, methanol - Pesticide quality or equivalent.

5.3 Sodium sulfite - (ACS) reagent grade.

5.4 Sodium sulfate - (ACS) granular - Prepare by covering sodium sulfate with methylene chloride in a beaker and placing in ultrasonic cleaner. Sonicate for 30 minutes, drain off the excess methylene chloride and place in drying oven.

5.5 Alumina, Super I Woelm or equivalent, prepare by adding 7 percent (v/w) reagent water and tumble for a minimum of 2 hours. There should be no lumps present.

5.5.1 Each batch must be assayed using the alumina equivalency test. If the batch does not meet criteria, it may be deactivated with up to 9 percent water.

5.6 Tetrabutylammonium (TBA) - Sulfite reagent Dissolve 3.39 g of TBA in 100 mls of reagent water. Extract three times with 20 ml portions of hexane. Discard the hexane extracts, and add 25 g of sodium sulfite to the water solution. The resulting solution can be stored at room temperature for one month in an amber bottle with a teflon screw cap.

5.7 Dibutylchlorendate (DBC) surrogate solution (for soils) - Prepare DBC solution at a concentration of 20 ug/ml in methanol. The solution must be stored at 4°C (±2°C) in teflon sealed containers for a maximum of 12 months.

5.7.1 Add 100 ul of the DBC solution to all samples, blanks and matrix spikes/matrix spike duplicates (MS/MSD). If the sample requires GPC cleanup, the surrogate amount will be doubled.

300953

5.8 Base Neutral/Acid Surrogate Solution

5.8.1 The BNA surrogate will contain the following compounds at the specified concentrations in methanol:

Surrogates	Concentration, ppm
Nitrobenzene-d_5	100
Terphenyl-d_{14}	100
2-fluorobiphenyl	100
2,4,6-tribromophenol	200
2-fluorophenol	200
Phenol-d_5	200

The solution must be stored at 4°C (±2°C) in teflon sealed containers for a maximum of 12 months.

5.8.2 Add 500 ul of the BNA surrogate solution to all samples, blanks, and matrix spike/matrix spike duplicates. If the sample requires GPC cleanup, the amount of surrogate will be doubled.

5.9 Pesticide Matrix Spiking Solution.

5.9.1 The spiking solution will contain the following pesticides at the specified concentrations in methanol:

Pesticide	Concentration, ppm
Lindane	2.0
Heptachlor	2.0
Aldrin	2.0
Dieldrin	5.0
Endrin	5.0
4,4'-DDT	5.0

The solution must be stored at 4°C (±2°C) in teflon sealed containers for a maximum of 12 months.

5.9.2 Matrix spikes and matrix spike duplicates will get 400 ul of the pesticide spiking solution. If the sample requires GPC cleanup, the amount of matrix spike will be doubled.

5.10 Base Neutral/Acid Matrix Spiking Solution

5.10.1 The BNA spiking solution will contain the following compounds at the specified concentration in methanol.

Compound	Concentration, ppm
1,2,4-trichlorobenzene	100
Acenaphthene	100
2,4-dinitrotoluene	100
Pyrene	100
N-nitroso-di-n-propylamine	100
1,4-dichlorobenzene	100
Pentachlorophenol	200
Phenol	200
2-chlorophenol	200
4-chloro-3-methylphenol	200
4-nitrophenol	200

The solution must be stored at 4°C (±2°C) in teflon sealed containers for a maximum of 12 months.

5.10.2 The matrix spike and the matrix spike duplicate will each get 1.0 ml of the BNA matrix spiking solution. If the sample requires GPC cleanup, the amount of spiking solution will be doubled.

6.0 SAMPLE PRESERVATION AND STORAGE

6.1 Preservation Requirements

6.1.1 Soil/sediment samples must be protected from light and refrigerated at 4°C from the time of receipt until extraction and analysis.

6.2 Holding Times

6.2.1 CLP level soil/sediment samples shall be extracted within 10 days of VTSR (Validated Time of Sample Receipt), and SOP soil/sediment samples shall be extracted within 14 days of collection. NYSDEC 89 soil/sediment samples shall be concentrated within 5 days of VTSR.

a) If SOP soil/sediment samples are not received within 1 day of collection, the Extractions Group Leader and the Organics Manager must be notified. A CAR will be issued to Client Services to contact the client for instructions on how to proceed.

7.0 SAMPLE ANALYSIS

7.1 Safety

7.1.1 Analysts shall treat all samples as if they are hazardous and shall take all appropriate safety precautions, including but not limited to safety glasses, lab coats and impervious gloves.

7.2 Chain of Custody Procedures

7.2.1 All analysts are responsible for maintaining sample custody in accordance with EPA guidelines.

7.2.2 All samples must be signed out on the chain of custody forms located in sample control.

7.3 Low Level Soil Extraction/Concentration Procedure

7.3.1 When the sample is brought to the laboratory from sample control, the extraction logbook for the batch is started. A copy of the logbook page is labeled Figure 1. The appropriate information is entered into blocks 1 through 3.

a) If both BNA and pesticide/PCB analyses are requested on a sample, a separate logbook page will be started for each fraction.

7.3.2 If there is free water present in the sample, decant and discard the excess water on the sample. Record the approximate percentage of free water in the comments section of the extractions logbook.

7.3.3 Thoroughly mix the sample and discard any foreign objects (eg. sticks, rocks, etc.). Transfer 50 g of sample to a 150 ml beaker and add 50 ml of water. Stir for 1 hour, and read the sample pH using a pH meter equipped with a glass electrode. Record this information in block 7 of the extractions logbook. Discard this portion of the sample.

a) If the pH is outside the range of 5-9, a Corrective Action Report (CAR) is filled out. A copy of the CAR is labeled Figure 2. The client will be notified, and his instructions will be noted in the case narrative.

7.3.4 Weigh 30.0 g. ±0.1 g. into a labeled 400 ml beaker, add 60 g. of anhydrous sodium sulfate, and mix well. The sample should have a sandy texture at this point. If the sample is still wet at this point, add up to an additional 30 g. of sodium sulfate. Immediately add 100 mls of 1:1 methylene chloride\acetone to the sample. The appropriate information is entered into block 4 of the extraction logbook.

7.3.5 With every batch of samples processed, a blank must be run. The blank will consist of blank sand.

a) If the sample is from the NJDEP or for X-408 protocols the blank will consist of solvent only.

7.3.6 Tracking labels (see Figure 3) or an equivalent system must be used for every sample and blank.

7.3.7 Add 100 ul of DBC for soils and 500 ul of BNA surrogate solution to every blank, sample and matrix spike/matrix spike duplicate (MS/MSD). If the sample requires GPC cleanup, double the amount of surrogates.

a) If only one parameter is requested, it will only be necessary to add the appropriate surrogate to the sample.

7.3.8 If a MS/MSD is to be run with a batch, add 1000 ul of BNA matrix spike solution and 400 ul of pesticide matrix spiking solution to the MS/MSD samples. If the sample requires GPC cleanup, double the amount of matrix spiking solution.

a) If only one parameter is requested, it will only be necessary to add the appropriate spiking solution to the samples.

b) For the NYSDEC 89 protocol prepare a blank MS every time a MS/MSD is extracted by adding the appropriate spiking solution(s) to a sand blank.

7.3.9 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared weighing dish and dry overnight at 105°C. Allow the sample to cool in a desiccator before weighing. Record this information in block 7 of the extractions logbook.

7.3.10 Sonicate the samples for 3 minutes using the 3/4" horn with the output control knob set at 10 and a pulse rate of 50 percent. The bottom of the sonicator horn should be about 1/2" below the surface of the solvent, but above the sediment layer.

a) It may be necessary to break up lumps in the sodium sulfate using a clean spatula prior to each extraction.

7.3.11 Decant the extracts and filter through Whatman glass fiber filter paper using vacuum filtration into a labeled side arm flask.

a) The label should include the sample number, the parameter analyzed and the date. Colored tape is used for the label.

7.3.12 The extraction is repeated twice with 2 additional 100 ml portions of 1:1 acetone - methylene chloride. Decant off the solvent after each extraction.

7.3.13 After the final sonication, the entire sample is poured into the Buchner funnel and rinsed with 30 - 40 mls 1:1 acetone\ methylene chloride. Fill in the appropriate information in block 5 of the extractions logbook.

7.3.14 If the sample is to be screened for low/medium level analysis, take 5 ml of the total extract and concentrate to 1 ml using the nitrogen blowdown technique. After screening, the remainder of the extract is re-combined with the rest of the extract.

7.3.15 Transfer the extract to a K-D concentrator. Rinse the side arm with 20-30 mls of methylene chloride to quantitatively transfer the sample. Transfer the tape label to the K-D flask.

7.3.16 Add one or two clean boiling chips to the K-D and attach a three ball snyder column. Pre-wet the Snyder column with about 1 ml methylene chloride. Place the K-D in a hot water bath (80 to 90°C). The temperature should be such that the concentration is completed within 15 minutes. When the apparent volume reaches 2 - 3 ml, remove the K-D and allow it to drain and cool for 10 minutes.

a) At the proper rate of distillation, the balls in the column will actively chatter but the chambers will not flood with condensed solvent.

7.3.17 Remove the Snyder column, and rinse the flask and its lower joint into the concentrator tube with methylene chloride. Transfer the tape label to the recieving vessel. If the sample requires GPC cleanup, place the concentrator tube in the N-Evap and concentrate to 0.5 ml to remove the majority of the acetone. In all cases make up to a final volume of 10 mls using methylene chloride. For information on performing GPC cleanup, see the latest version of the "Standard Operating Procedure for Gel Permeation Chromatography".

a) If the sample is from the NJDEP or for X-408 protocols written permission must be obtained prior to performing GPC cleanup on the BNA fraction.

7.3.18 The extract is split for semi-volatile and PCB/pesticide analysis at this point. Transfer 0.5 ml of the extract to a clean labeled concentrator tube to prepare for pesticide/PCB analysis. The remaining 9.5 mls is prepared for semi-volatile analysis.

a) If the sample is for semi-volatiles only, the extract will not be split.

7.3.19 Place the concentrator tube containing the semi-volatile fraction in the N-Evap which has a water bath maintained at about 35°C. Evaporate the solvent volume to approximately 0.5 ml using a gentle stream of clean, dry nitrogen. During the concentration step, the solvent level must be kept below the water level of the of the bath. The extract must never be allowed to go dry.

7.3.20 If the extract was split, the semi-volatile portion is brought to a volume of 0.95 ml. If the extract was not split, the sample is brought to a final volume of 1.0 ml.

7.3.21 Add 5 ml of hexane to the 0.5 ml portion of the extract that was split out for pesticide/PCB analysis and mix well. Use the nitrogen blowdown technique to concentrate to 0.5 ml.

7.3.22 Prepare the sample for alumina cleanup by diluting the extract to 1 ml with acetone.

7.3.23 Add 3 g of activity III alumina to a 10 ml chromatographic column. Tap the column to settle the alumina. Do not pre-wet the alumina. Place a clean 10 ml concentrator tube beneath the alumina column to collect the eluate.

7.3.24 Transfer the acetone/hexane extract to the top of the alumina using a disposable pasteur pipet. Add 1 ml hexane to the column, and rinse the original concentrator tube with 1 ml hexane. Transfer the rinsings to the top of the alumina column and elute the column with an additional 8 mls of hexane.

a) Do not allow the column to go dry during the elution.

7.3.25 Place the concentrator tube on the N-Evap and blow the sample down to 1.0 ml.

7.3.26 Transfer the 1 ml to a 15 ml vial with a teflon lined screw cap and rinse the concentrator tube with 1 ml of hexane. Transfer the washings to the bottle.

7.3.27 Add 1 ml of TBA-Sulfite reagent and 2 ml of 2-propanol. Shake for 1 minute. If the solution clears, add crystalline sodium sulfite (in approximately 100 mg portions) until a solid residue remains.

7.3.28 Add 5 ml of reagent water and shake for at least 1 minute. Allow the sample to stand 5-10 minutes. Transfer the hexane layer (top) to a concentrator tube and concentrate to approx. 0.5 ml. using the N-Evap. Bring up to a final volume of 1.0 ml in a volumetric flask.

7.3.29 Transfer the semi-volatile and pesticide/PCB extracts to labeled separate teflon lined screw cap vials. A copy of the labels with the appropriate information is included as Figure 4.0.

7.3.30 Fill in the appropriate spaces in block 6 of the extractions logbook.

7.3.31 The extracts are now ready for analysis. Place the extract in the appropriate freezer in sample control, and fill out block 8 of the extractions logbook.

7.4 Semi-volatile Medium Level Sample Preparation

7.4.1 In a fume hood weigh 1.0 ±0.1 g sample into a 22 ml vial. Wipe the mouth of the vial to remove any excess material and record the exact weight in the extractions logbook. Cap the vial to avoid cross contamination.

7.4.2 If necessary, determine the pH and percent solids on the sample as in section 7.3 above.

7.4.3 Weigh out 1.0 g blank soil for each batch of samples. For NJDEP samples or for X-408 protocol samples the blank will consist of solvent only.

7.4.4 Add 2.0 g of anhydrous sodium sulfate to the sample and the blank, and mix well.

7.4.5 Add 1.0 ml of BNA surrogate to all samples and blanks, and MS/MSD.

7.4.6 If a MS/MSD is to be run with a batch of samples, add 1.0 ml of BNA matrix spiking solution to the sample.

a) For NYSDEC89 samples, it will be necessary to extract a Blank MS every time a MS/MSD is extracted.

7.4.7 Immediately make up to 10 mls with methylene chloride. Sonicate the sample for 2 minutes using the 1\8" tapered microtip horn. The output control knob is set at 5 in the continuous mode.

a) Prior to starting the extraction, make sure that the sodium sulfate is free flowing. It may be necessary to break up the lumps with a spatula.

7.4.8 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect 5 ml in a clean concentrator tube.

7.4.9 Concentrate the extract to 1.0 ml using the nitrogen blowdown technique.

7.4.10 Transfer the extract to a labeled vial, fill out the extractions logbook, and place the extract in a freezer in sample control.

7.5 Pesticide/PCB Medium Level Sample Preparation

7.5.1 In a fume hood weigh 1.0 ±0.1 g sample into a 22 ml vial. Wipe the mouth of the vial to remove any excess material and record the exact weight in the extractions logbook. Cap the vial to avoid cross contamination.

7.5.2 If necessary, determine the pH and percent solids on the sample as in section 7.3 above.

7.5.3 Weigh out 1.0 g blank soil for each batch of samples. For NJDEP samples or X-408 protocol samples the blank will consist of solvent only.

7.5.4 Add 2.0 g of anhydrous sodium sulfate to the sample and the blank, and mix well.

7.5.5 Add 50 ul of DBC for soils surrogate to all samples and blanks, and MS/MSD.

7.5.6 If a MS/MSD is to be run with a batch of samples, add 1.0 ml of Pesticide matrix spiking solution for soils to the samples chosen.

a) For NYSDEC89 samples it is necessary to extract a Blank MS every time a MS/MSD is extracted.

7.5.7 Immediately make up to 10 mls with hexane. Sonicate the sample for 2 minutes using the 1\8" tapered microtip horn. The output control knob is set at 5, and the pulse rate is at 50 percent.

a) Prior to starting the extraction, make sure that the sodium sulfate is free flowing. It may be necessary to break up the lumps with a spatula.

7.5.8 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extract through the glass wool and collect 5 ml in a clean concentrator tube.

7.5.9 Transfer 1.0 ml to a concentrator tube and concentrate the extract to 0.5 ml using the nitrogen blowdown technique. Add 0.5 ml of acetone and clean up the sample using the alumina and sulfur clean up procedures detailed in section 7.3.

7.5.10 Transfer the extract to a labeled vial, fill out the extractions logbook, and place the extract in a freezer in sample control.

8.0 QUALITY CONTROL

8.1 A blank will be run with every batch. A batch is defined as a group of samples for an analysis run within a 24 hour period. A batch can have a maximum of 20 samples.

8.2 A matrix spike/matrix spike duplicate will be run at a frequency of 1 sample in 20. However, if more than 14 days elapses and 20 samples have not been run, it will be necessary to run a MS/MSD. For samples extracted using the NYSDEC 89 protocol it is also necessary to extract a Blank MS every time a MS/MSD is extracted.

8.3 The extractions Batch Approval Sheets must be filled out on a daily basis. They are located on the back of the extractions logbook page. A copy of the sheet is included as Figure 5.0

8.4 A case narrative must be filled out for every batch assigned on a daily basis. There is space provided on the Batch Approval Form for the case narrative. If the information requires transmission to other groups, a CAR should be used. Final case narratives will also be included in the client job folder. An example of a Final Case Narrative is provided as figure 6.0.

8.5 The Extractions Logbook and the tracking log must be completely filled out on a daily basis for every batch extracted or concentrated.

8.6 The Extractions Group Leader or his designee will fill out the Extractions QCAR (Figure 7.0) for inclusion in each clients job folder.

9.0 METHOD PERFORMANCE

The method performance is determined by the final measurement results of each sample and the accuracy of surrogate and MS/MSD measurement results.

10.0 DATA REDUCTION AND HANDLING

Not Applicable

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IEA
ORGANIC EXTRACTION LOG
AQUEOUS/SOLID MATRIX

PAGE #

BLOCK 1

ANALYSIS ______________
CORRESPONDING QS/QSD ______________

BLOCK 2

EXTRACTION DATE ______________
FINISH DATE ______________
SURROGATE CODE ______________
SPIKE CODE ______________
APPROVED BY ______________

BLOCK 3				BLOCK 4				BLOCK 5					BLOCK 6					BLOCK 7					BLOCK 8					
																			% SOLIDS DATA				LOCATION				
LAB EXT	CLIENT	LAB ID	SIGN OUT COC	CL2 RES	INIT pH H2O	INIT WT/VOL	SAMPLE COLOR & TEXTURE	SURR INIT	SPIKE INIT	EXT METHOD	METH BS SCAN	EXT BY	C/D	EXT TO DAT	WT	COLOR OF EXT	CONC/ SCORED BY	pH	TARE	WET WT	DRY WT	% SOLIDS	FZR	TRAY	SLOT	METH 02 SCAN	COMMENTS

FIGURE 1.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

Reported By: _____ Group

Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE REPORT

Client: _____ Job Number: _____ Sample Number(s): _____

Problem Reported By: _____

Category of Problem: _____
Poor Communication With ___ Sample Matrix Instrument

Description of Problem: _____

Use back of CAR/CNR or attached sheet if more space is required

Recommended Corrective Action(s): _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline For CAR: _____ Reply to Originator Required? [ ]

Actual Corrective Action(s) Taken: _____
Document Actual Corrective Actions On This

Form and Send a Copy of Completed CAR/CNR to QA Officer and Job Folder

_____
Signature and Date

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Initial Lab manager, Marketing Manager, Branch Manager, QA Officer.
Distribution: Client Services, Job Folder, Other: _____
SECONDARY DISTRIBUTION: ORIGINATOR, QA OFFICER, OTHER: _____

Document Problem and Corrective Action(s) In Case Narrative: [ ]
Attachments: Telephone Log, Services Change Request, Memo, Other:

FIGURE 2.0

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BLOCK 1

ANALYSIS ____________

CORRESPONDING RS/RSD ____________

BLOCK 2

EXTRACTION DATE ____________

FINISH DATE ____________

SURROGATE CODE ____________

SPIKE CODE ____________

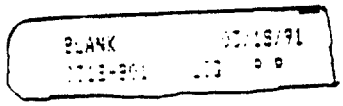
APPROVED BY ____________

IEA
ORGANIC EXTRACTION LOG
AQUEOUS/SOLID MATRIX

PAGE #

BLOCK 3				BLOCK 4				BLOCK 5					BLOCK 6					BLOCK 7					BLOCK 8				
																			% SOLIDS DATA				LOCATION				
LAB EXT	CLIENT	LAB ID	SIGN OUT COC	CL2 RES	INIT pH H2O	INIT WT/VOL	SAMPLE COLOR & TEXTURE	SURR INIT	SPIKE INIT	EXT METHOD	METH 01 SCRN	EXT BY	C/D	EXT TO DAT	BY	COLOR OF EXT	CONC/ SCORED BY	pH	TARE	WET WT	DRY WT	% SOLIDS	FZR	TRAY	SLOT	METH 02 SCRN	COMMENTS

FIGURE 1.0



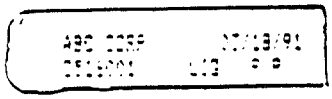
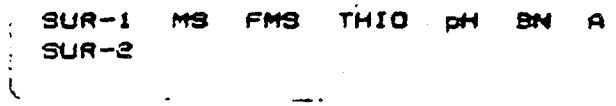


FIGURE 4.0



300970

FIGURE 3.0

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IEA CT

EXTRACTIONS BATCH APPROVAL FORM

Parameter _____ Matrix _____

Blank ID. _____

		Extraction Init/Date	Concentrate Init/Date
1)	All required QA/QC was performed or referenced	_____	_____
2)	Extraction logbook was completed	_____	_____
3)	The sample tracking log has been updated	_____	_____
4)	Case narrative was completed	_____	_____
5)	Was a corrective action form submitted?	_____	_____
6)	The assignment was performed according to the SOP and all exceptions have been documented	_____	_____

CASE NARRATIVE

_____

_____

_____

_____

_____

_____

_____

Final Approval: _____

FIGURE 5.0

300972

an environmental testing company
300 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

THE ANALYSIS OF PESTICIDES/PCB'S IN ENVIRONMENTAL EXTRACTS

GC:010687:6

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-6181
Cary, North Carolina 919-677-0090
Essex Junction Vermont 802-878-5138

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TABLE OF CONTENTS

List of Tables

1.0 CONVENTIONS

For organizational purposes all activities in the GC section have strict conventions for coding standards, data files, peak reports, analysis methods and instrument nomenclatures.

1.1 Ethics Policy - IEA

It is the policy of IEA, Inc.-Connecticut and the GC group to ensure that analyses of samples fully comply with governmental laws and regulations as well as the IEA, Inc. Ethics Policy.

1.2 Standards

All standards are traceable to EPA and are ordered from the EPA Repository. All standards, upon receipt, are logged in the appropriate card file with the date of receipt, source and lot numbers.

All standards preparations are logged and coded (see Pesticide/PCB standards prep SOP).

1.3 Computer System Conventions

At the time of preparation of this SOP the Perkin Elmer 7500 Computer with Chrom 3 software is utilized for the collection of data.

Under this mode of operation each GC has an operating environment with its own methods. Currently four environments are utilized. The following table lists the methods utilized for data collection and handling. Method 106 is used for raw data collection. Other methods are then calibrated against associated peak files for re-reports of standard peak files.

Data files are named depending on the particular analysis being run, and the column used. The following table gives an example of file naming. Only ten digits are allowed. For confirmation runs, add a "C" or Conf to the file name.

Client(s)	File Naming Convention
EPA	"epa nnnn" (the numbers (n) being the EPA case number)
Miscellaneous Clients	pestn (add a number (n) if this name has been used so as not to overwrite previous data)

STANDARD OPERATING PROCEDURE
THE ANALYSIS OF PESTICIDES/PCB'S IN ENVIRONMENTAL EXTRACTS

Abstract

This Standard Operating Procedure (SOP) addresses the analysis of aqueous and solid sample extracts for Pesticides and PCB's as defined by EPA Method 608 (10/84) for priority pollutants, EPA Method 8080 SW846 3rd edition, and USEPA CLP SOW 2/88 Hazardous Substance List. Extracts are analyzed using Gas Chromatography with Electron Capture Detection (GC/ECD). Specific protocols should be reviewed for complete descriptions of sample analysis.

The intention of this SOP is to consolidate all aspects of the analyses in one document isions as required) for the purposes of consistency in data quality.

The basic methodology involves solvent extraction of waters or soils followed by concentration to required volume. The extract is then injected into gas chromatographs equipped with electron capture detectors. If target compounds are tentatively identified then the extract is injected on second column for confirmation.

If the column has not been used recently, heat up slowly (approximately 10°C/min) by temperature programming the GC. If column is new then it must be conditioned. Columns are conditioned by temperature programming. Setting the initial temperature at 40°C for 15 minutes to stabilize flow then heating at 2°C/min up to 220°C holding at this temperature for approximately 18 hours.

(3) When desired oven temperature is obtained and detector is above 250°C, prime column by injecting approximately 10 ul of a 10 ppm PCB 1254 standard or high concentration of mixed pesticide standard (i.e. stock standard of Mix A or B). Turn power on at detector control box for PE SIGMA GC. For HP GC check that detector is on (Reference GC Manual).

(4) When all temperatures are reached, wait 15 minutes for equilibration. Turn ECD to current setting = 4 and let equilibrate for 15 to 20 minutes (for PE SIGMA only).

When utilizing Chrom 3 software the signal from detector should be stabilized between 8-12 millivolts. This can be adjusted by the zero control on ECD control box (PE SIGMA only).

2.1.1 Daily Instrument Maintenance

Injection port septa should be changed daily or after approximately 60 injections. To do this do the following: Turn ECD to "Balance," cool all GC temperatures to 30°C. When GC is cool shut off gas flow at toggles and let bleed until back pressure is zero. Replace septum. Turn carrier gas on and heat to required temperatures. Record all maintenance activity in GC log book.

2.1.2 Routine Preventive Maintenance

The following is a list of tasks to be performed on a routine basis as preventive maintenance for the instruments used by the GC group. Record all maintenance in appropriate log.

1) Change diverter valve septa in Perkin-Elmer autosampler every 200 injections.

2) Check flushing efficiency of Perkin-Elmer autosampler each time a run is started. Adjust helium/air pressure as needed. Clean syringe when indicated.

3) Check syringe on the Hewlett-Packard autosampler for clogs each time a run is started. Clean with solvents once a month or sooner to prevent freezing of the syringe.

2.0 SAMPLE ANALYSIS

2.1 Instrument Preparation

(1) Verify that flow to column is off and no back pressure is present. Change injection port septa, and install appropriate column for

TABLE I
METHODS OF ANALYSIS

Application	Chrom 1	Chrom 2	Chrom 4
Column:	1.5% SP2250/1.95% SP2401	3% OV-1	1.5% SP2250/1.95% SP2401
Raw Data File Collection	Method 206	Method 206	Method 206
Re-reports:			
Evaluation Mixes	Method 100	Method 100	Method 100
Ind A	Method 820	Method 810	Method 820
Ind B	Method 821	Method 811	Method 821
DDT Series	N/A	Method 102	N/A

analysis. When using on column injection columns for PE SIGMA, install leaving approximately one quarter inch from end of injection port. Turn on carrier gas and establish flow at 60 ml/min for 4 mm ID columns and 30 ml/min for 2 mm ID columns. Ensure flow through detector is approximately 60 ml/min. Check nitrogen pressure in the gas cylinder. Do not allow pressure to go below 300 psig. Leak check all fittings.

(2) If GC is off, turn instrument power on. Set instrument parameters as follows:

Column	1.5% SP2250/1.95% SP2401	3% OV-1
***Oven Temp**	210°C	190°C
Zones (Detector & Injector)	325°C	325°C

Pesticide/PCB analysis by packed column is isothermal, therefore, initial and final temperatures are the same. These may also vary to meet EPA/CLP criteria for 4,4'DDT retention time.

*Note: 2.2.2 may not be performed on CLP samples without permission from client.

2.2.3 AS100B Set Up

Change septum in autosampler diverter valve. This should be done after approximately 100 injections. Check to see that AS-100B solvent or gas lines are not crimped and that syringe moves freely. (Reference Section 5 of AS-100B manual.) Place autosampler on bracket and position it approximately 1/2" from the injection port.

Check the alignment by carefully moving the syringe towards the injection port, piercing the septum and making sure that the needle moves freely into the injection port. Slide sample tray into the carriage assembly and plug in the interface cable.

Turn on the autosampler controller. The autosampler will now be pressurized, so do not attempt to move the syringe. Position the tray to the flush vials and manually flush the syringe by pressing the flush button. Do not flush each vial more than twice. Check flow through the sample, waste lines and into the waste vessel (15-20 drops per flush).

2.3 Autosampler Set Up

2.3.1

Remove samples to be analyzed from their respective freezers and sign appropriate chain of custody sheets. The chain of custody sheets are to be signed with the date and time when removing extracts from the freezer and when they are returned to the freezer. If it is necessary to sign the chain of custody at a later time, fill in the correct information then initial and date that information with the current date. Remove standards necessary for the run. Let all samples and standards equilibrate to room temperature. If using the AS100B autosampler, flushes can be prepared at this time. Fill each flush vial with hexane and place in the odd numbered slots in the autosampler tray.

2.3.2

Properly complete injection log book for the respective tray (See Figure 1). Initiate QC Batch Approval Form. Note any deviations occurring throughout the run, initial and date each task performed. (See Figure 2) Clearly label each vial for standards and samples. For soil samples use the autosampler vials which are designed for small volume analysis. Place the vials in the tray as noted on the autosampler log sheet. For AS100B, place 2-3 vials before the first

4) Check solvent flush vials and waste vials on the Hewlett-packard autosampler at the start of each run.

5) Clean by sonication or replace ECD anode in Perkin-Elmer GC when linearity exceeds criteria and standard prep is correct.

6) When breakdown criteria exceeds 18 percent change glass wool at injection end of the column and inspect for breakage.

7) When breakdown exceeds 20 percent change glass wool and silanize injection port.

8) Maintain adequate directory space in the data station by archiving and deleting data every week.

2.2 Extract Cleanups

2.2.1

Check the extractions log book for the type of clean-up performed on the samples. Certain clean-ups are required depending on matrix and protocols required. Some samples are required to be extracted by EPA-CLP protocols. Sulfur and alumina column clean-ups are performed in this extraction procedure. If after running the samples extracted as per EPA SW846, the scans still indicate the presence of interferences, a florisil column cleanup may be required. Reference Table II for the elution of pesticides in the Florisil Fractions. Highly colored samples may require dilution.

Soils and some waters may contain sulfur which can be removed by the addition of mercury. A few drops are added to the sample, which should then be shaken for 1-2 minutes. If sulfur is present a black precipitate will form. If this is the case, let the precipitate settle out and carefully pipet sample to another vial. Repeat above until no precipitate forms.

2.2.2

If an extract is to be analyzed for PCB's only, then an acid clean up may be done. This is to remove any organic compounds which may interfere with the analysis. The ratio for the clean up is 1 part acid (Conc. H_2SO_4) to 2 parts extract. The acid is added to the sample then shaken. The acid layer should be at the bottom layer. Carefully pipet extract layer to another vial. Repeat until acid layer is light brown to orange. End with a sulfur cleanup to remove any residual acid.

TABLE II
DISTRIBUTION AND RECOVERY OF CHLORINATED PESTICIDES
AND PCB'S USING FLORISIL COLUMN CHROMATOGRAPHY

Parameter	Percent Recovery by Fraction* 1 (6%)	2 (15%)	3 (50%)
Aldrin	100		
a-BHC	100		
b-BHC	97		
d-BHC	98		
g-BHC (Lindane)	100		
Chlordane	100		
4,4'-DDD	99		
4,4'-DDE	98		
4,4'-DDT	100		
Dieldrin	0	100	
Endosulfan I	37	64	
Endosulfan II	0	7	91
Endosulfan Sulafte	0	0	106
Endrin	4	96	
Endrin Aldehyde	0	68	26
Heptachlor	100		
Heptachlor Epoxide	100		
Methoxychlor	100		
Toxaphene	96		
PCB 1016	97		
PCB 1221	97		
PCB 1232	95	4	
PCB 1242	97		
PCB 1248	103		
PCB 1254	90		
PCB 1260	95		

*Percentage of ether in hexane

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required injection as flushes to clean out the syringe before initiating the run. Reference Table VI for the correct analytical sequence. Load the autosampler tray according to this sequence.

2.3.3

Analyze an instrument blank after standard sequence every time a new sequence is started on each GC. This is prepped with DBC at a concentration of 0.1 ug/mL.

If everything functions properly proceed to the "set-up" as per the autosampler method (Reference Table III). Be sure that the helium tank has more than 500 psig for correct autosampler operation.

Also check that the house air is on.

2.2.4 HP7673A

If HP GC is ready then prep the autosampler sequence via the HP integrator. Using the shift key hit [Prep] [Seq]. This will then prompt you a series of questions. Reference Table IV for prompts. Enter any changes, then hit the [Enter] key. If no changes are required just hit [Enter].

To end or cancel sequence use the [Break] key. Any changes made will be stored.

Now check to make sure vial trays are in the autosampler and that the tray holder is set all the way forward towards the autosampler tower. Make sure tower is straight. Empty solvent waste vials and fill solvent flush vial.

To start sequence run, hit [Shift] [Seq] and then [Start]. The autosampler program entered will now start running.

3.0 DATA ACQUISITION

Data is acquired using the Chrom 3 software and each run is processed into a data file and a peak file. Set up of the system is required before data can be processed. The following steps outline the set-up procedure. Data may also be acquired utilizing integrators but cannot be archived.

3.0.1

Check the system configuration by selecting "config" key. The configuration page will show whether the devices are linked to chromatographic environments. If changes are required, refer to the Chrom 3 manual.

3.0.2

To proceed to set up, press the "select" key and choose the appropriate chromatograph. Select the "set up" key under the operate section of the keyboard.

TABLE III
AS100B AUTOSAMPLER KEYS

Programs

Key Illuminated	Meaning	Entry
Set-Up	Program set-up	Program (#1-4)
Auto	Starts program	
Man	Manual manipulation of autosampler exit set-up	Use flush key, position or sample; exit set-up program
	SET-UP PROCEDURE	
P	First sample	00-99 (2 digits required)
Run time	Time between injection (minutes)	045 (3 digits required)
Time/Flush	Sample flushing time	12 sec (09 if soil vials are on sequence)
Time/Sample	Needle residence time	02 (secs)
Sample	Number of replicates on each vial	1
Flush	Flush interval	2 (every other vial is flush)
Stop	Last vial	00-99 (2 digits required)

3.0.3

To set up the method of choice select "set up method" under the soft key section. Enter appropriate responses for set-up page, then press "accept set up".

Prompt	Response
Analyst:	Initials
Method:	Analysis method
Sample:	Data file name
Descr:	Column, ul injected, GC#
Save Files	All
Application	None
Read BCD	No
Flag	Increment

3.0.4

Press the "plot" key to bring the plot to the screen. Select the "scale y" key to adjust scaling applicable to the GC environment. Select "monitor" soft key to monitor the detector output for PE SIGMA. Adjust to 0.01 with detector zero knob. Exit the monitor after the signal is stable. If the system indicates ready then the run may be started via the autosampler or integrator.

4.0 DATA REDUCTION

4.1 Initial Data Review

Check the continuing standards while the run is in progress. Initial and comment on any criteria. If any standard is out of QC requirements then samples following that standard must be reanalyzed. If run was stopped at that standard, try reinjecting standard. If QC is still not met, then a new 72 hour sequence period must be started. Reference Table V for required analytical QC criteria. After a run is complete or while it is still in progress check each chromatogram to ensure an appropriate injection was made. This can be done by checking for the presence of 1) solvent peak and 2) DBC surrogate. Reanalyze any sample that looks suspicious (i.e. no DBC peak or DBC peak with <30 percent the area of the average area of the standards). Before reanalysis see Group Leader. If scan indicates interferences, check area integration for that data file by selecting the data analysis section of the Chrom 3 and checking integration with the "interactive reintegration" mode. If necessary go into "method development" to adjust integration algorithm for proper integration. See Group Leader for approval to change the integration method. Check for dilutions at this point. Complete the analysis secion of the QC Batch Approval Form.

TABLE IV
HP 7673A AUTOSAMPLER PROGRAM

Prompt	Meaning	Entry
ALS Information INET Sampler Control	7673A Controlled by INET	Y
7673A Sampler Loop Address: 8	Communications	No Entry
Front Injector		
Inj/Bottle 1→	Injections/Bottle	1
First Bottle 13→	Starting Vial	# to start at
Last Bottle 13→	Last Vial	# to end seq.
# of Sample Washes 3→	Rinses Syringe with sample to waste	3
# of Pumps 5→		5
Viscosity 3→	Solvent Viscosity	3
Volume 2→	Injection Volume	2
# of Solvent A Washes 6→	Solvent Rinses	6
# of Solvent B Washes 0→	Solvent Rinses	0
Priority Sample (1=Yes) 0→		0
Capillary On-Column 0→	Capillary vs. Packed	0
Equilibrium Time in Seconds [10]		10
Method [M:CLP1.MET]		Anal. Method
Sample Information Table		
Bottle or Run Sample Indexed [R/B*]		B
Bottle #	Bottle or Run # (can be labeled at this point)	Enter*

*This will end sequence program

For samples analyzed according to the NJDEP contract, TIC must be done on pesticide/PCB (Task IV) samples. This is done by recording on appropriate forms the retention times for the ten largest peaks that have a response greater than 2x the background noise leve. For external standard calculation only retention times are required. If using internal standard concentrations, it should be calculated from the nearest internal standard.

4.3 Confirmation

Any samples with possible target pesticides/PCB hits must be run on the confirmatory column (3 percent OV-1). Associated method blanks must also be run.

Toxaphene is always quantified using this column and requires three levels of toxaphene standards be run. If DDT is to be quantified using this column and the percent RSD for DDT in the evaluation mixes is greater than 10 percent, then the DDT series standards must be analyzed and the percent RSD calculated for each compound. The DDT compounds (4,4'-DDT, 4,4'-DDD and 4,4'-DDE) must also have a calibration curve plotted by graphing area vs. ng injected. This is done by using the Super Plot Program on the PE-7500 computer. If the percent RSD is greater than 10 percent for a compound, then the concentration of that compound in the sample is read from the curve.

Only standards of compounds to be confirmed are to be analyzed at both the beginning and the end of the run. The analytical sequence for the samples is the same as the primary run (Reference Table VI).

Data reduction is performed in the same manner as the primary column data. A compound is confirmed if it is tentatively identified on primary analysis and if the retention time falls within the retention time window of a corresponding standard on the confirmation run. See Group Leader if you are not confident on a peak.

5.0 DATA CALCULATIONS, REPORTING AND VALIDATION

5.1 Subsequent to analysis (data acquisition), final results are calculated using the LIMS-2000 (correct areas should be in the standards data set). Results are then put on the appropriate report forms (See Figures 28-42) (i.e. Priority Pollutants, TCL/CLP). The following indicates the type of documentation required for the different levels of reporting:

. Level 1 - requires only tabular results given to client, with all other documentation (standards and sample scans) placed in client job file.

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After the analytical run is finished the GC instrument log book should be completed with the information required on the page. It should be signed by the Group Leader and copied to be put with the raw data. A copy should also go with EPA.

4.2 Final Data Review

After evaluation of data, re-reports against appropriate methods can be done for standards and matrix spikes. Any replots of scans should also be done at this time. The methods for Pesticide Individual Mix A and B should be calibrated and updated against the corresponding peak files for that run. When doing this be certain that all compounds in the mixes are recognized, if not the method must be modified to do so. All samples should have a raw area re-report (Method 206) on a single page. If reintegration was necessary a 206 report should be pulled for job and raw data file. Results can then be compared against calculated RT windows for possible hits.

Examine the raw data and calculate the required QC acceptance criteria for the run. This should be done on the LIMS-2000 computer. Fill out the appropriate QC forms (See Figures 24-27) continue filling out QC Batch Approval Form. Reference Figures 21-23 if LIMS is not operable and QC must be done manually. To fill out forms refer to Section B of the EPA CLP Manual. If QC is done on LIMS, then forms are printed by the computer. Reference SOP for LIMS. Have another analyst fill out Forms Reviewed Section of the QC Batch Approval Form.

Sample analysis is done by reviewing the scans, comparing peak retention times to the retention time windows calculated on Form 9 for that analytical sequence. Multi-responding compounds should be identified by pattern recognition (i.e. Aroclors). After sample analysis is complete then a confirmation list should be filled out (primary analysis only), refer to Figure #4. Associated Method Blanks must also be analyzed on the confirmatory column. Submit form to Group Leader.

If sample analysis is to be done on a confirmatory run, then only those compounds identified in the primary analyses are looked for. At this time the QC Batch Approval Form should be completed and approved by Group Leader. A copy should be made for each job analyzed in the batch.

The analytical run is then labeled with appropriate information (client. sample information, column, GC, uL injected). Standard peaks should also be labeled with compound identification and nanograms injected. The run is then copied and packaged. All raw data used for NJDEP x408 work must be signed. If continuous data, then (it is only necessary to sign) the beginning and end.

This program is on the LIMS computer and should be entered for each new column. Column identifications are as follows: Qn (n being the next number of column installed) for mixed phase column and Cn for confirmatory column.

GC:010687:6

2 - requires tabular results, QC Forms DBC recovery and retention time shift, method blank summary sheet, MS/MSD forms and data, standard and sample scans and integration reports.

4 2/88 - requires IFB deliverables: method blank summary, S/MSD forms, surrogate recovery, pesticide Form X, EPA QC orms VIII, IX, Form I, Page 3.

<408 - requires IFB deliverables plus TIC form.

1989 SOW - see Table Va.

tarting to complete a job package, a GC Packaging Review Form e 50) should be started. Each item should be checked off as it pleted. Any deviations from protocols should be anotated under ts section and in the case narrative. All information required obtained from the traveler. This form, along with the corre-ng QC Batch Approval Forms, should be handed in to the Group with the complete job package. At this time the Group Leader gnate will complete the Quality Control Approval Report (Fig-). The QCBAF forms are reviewed for deviations from criteria, kaging review form is checked to make sure comments and forms d match requested protocols and all deviations are noted in the arrative. This form will be placed in the job folder along he data package. The other forms will be on file with the Leader.

OCEDURES

ibration: When a new column is installed or detector is chang-ration curve of the compounds of interest is run. At least s of each compound are to be analyzed. The three pesticide mix standards must be run prior to starting curve and all r these must be met (Reference Table V).

ime Windows: Upon initial set up of a column, a standard f component retention times must be established. This is done jections of all single component pesticide mixtures, multi-sticides and PCB's over a 72-hour period. Retention time shift t be less than 2 percent difference (packed column). A stand-on of the three retention times is calculated. Plus or minus the standard deviation is used to calculate a daily window.

NCE CRITERIA

	Level of Reporting
	All
xed phase ied for 3%	All
	All
nt column firmatory	All
in sam- ntinuing l within	All
	All
(a 50% ory on-	All
ry only	CLP, Level II
	NYSDEC 89

mixed phase column, then any ould be calculated from the 3

les to be considered accept

300991

TABLE VI
72 HR. ANALYTICAL SEQUENCE: PRIMARY ANALYSIS

EPA CLP

Pesticide Evaluation Mix A
Pesticide Evaluation Mix B
Pesticide Evaluation Mix C
Pesticide Individual Mix A
Pesticide Individual Mix B
Toxaphene
PCB 1221*
PCB 1232*
PCB 1242
PCB 1248
PCB 1016/1260
PCB 1254
5 Samples
Pesticide Evaluation Mix B
5 Samples
Pesticide Individual Mix A or B
5 Samples (etc.)

Analytical sequences may continue until one or more of the criteria is not met.

Sequence must end with both Individual Mix A and Individual Mix B.

*PCB 1221 and PCB 1232 are required once per month, or whenever the column or detector have been changed.

7.0 EPA METHOD 608 PROTOCOLS

Analysis of Pesticides/PCB's using EPA Method 608 protocols varies with the external standard calibration.

7.1

Calibration standards at a minimum of three levels are analyzed for each parameter of interest. From the data calculate a calibration factor and percent RSD for each compound. If the percent RSD is ≤10 percent, then an average C_f may be used for quantitation. If the RSD >10 percent, results should be calculated from a calibration curve (area vs. mass injected). This curve must be checked daily. Percent difference for any analyte should not exceed 15 percent difference from the original curve. If this is the case, then a new curve must be run.

7.2

Quality control consists of a method blank to be analyzed for each set of samples extracted or change of reagents. Ten percent of all samples should be spiked at the time of extraction. Quality control samples should also be analyzed at the same frequency. Before analyzing any samples, all quality control set by EPA (Reference Federal Register 10/26/84) should be met.

8.0 CORRECTIVE ACTIONS

The following are some of the problems which may be encountered with analysis of pesticides/PCB extracts. If any problems occur a corrective action report must always be handed in (Reference Figure 3) to Group Leader. If the problem should be noted in the case narrative then a CAR/Case Narrative Report (QA:111086:4) should be filled out and submitted with the job checking off the appropriate box (see Figure 3a).

8.1

ODT and endrin breakdown is caused by active sites either within the column or injection port. The first thing to check is the glass wool in the front of the column. If the glass wool is discolored, then it must be changed. Also check for discoloration of the packing. The first few inches may require changing.

If using off column injection system then silanizing the injection port area will be necessary. If none of these solutions correct the problem, then you must replace the column. See Group Leader before changing column.

8.2

All compounds in the continuing calibration standards must fall within calculated retention time windows. This is an occasional problem if the septa is worn or not pierced correctly. Restarting the 72 hour sequence is the solution in this case. If this becomes a repeated problem over a few analytical runs, then new SD must be determined (see 6.2).

8.3

Linearity criteria is critical for all analytical runs. If this is out of criteria, re-inject standards. If it is still out, new standards should be prepped. If the percent RSD's are still consistently out, the anode on the ECD may require changing (P.E. Sigma Series) or detector replacement may be necessary.

8.4

DBC is added as a surrogate for pesticide analysis. The retention time of the compounds must not shift more than 2 percent from the initial evaluation mix A. If DBC is greater than 2 percent for a sample, then the sample must be reanalyzed.

If the DBC retention time shift is still out, then the sample matrix is probably affecting the retention time. If the sample is not from EPA no further analysis is required. If the sample is from EPA, inform the Group Leader who will contact SMO for instructions.

9.0 PESTICIDES/PCB STANDARD PREPARATION

9.1 Calibration

Due to the coelution of Pesticides/PCB's on packed columns, two separate individual mixes of pesticides are required, as well as separate standards for all multi-response compounds. All standards are prepped from EPA stocks for traceability. Dibutylchlorendate is added to all solutions as surrogate at a concentration of 0.1 ug/ml. Tables 9.1 through 9.6 list the preparation instructions for all standards used. All standards are prepared using hexane as the solvent. They are to be stored under refrigeration at all times. For calibration, each stock is diluted as required. (See following Tables.)

All standards are to be prepared using Class A volumetric glassware and stored in silanized vials with teflon lined caps. A new vial is to be used each time a standard is prepared. Two separate vials of pesticide

an environmental testing company
200 Monroe Turnpike
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STANDARD OPERATING PROCEDURE

THE ANALYSIS OF SAMPLE EXTRACTS FOR SEMI-VOLATILE ORGANICS BY USEPA CLP PROTOCOLS

MSSV:120586:7

300995

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-706-0740
N. Billerica, Massachusetts 617-272-6212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0080
Essex Junction, Vermont 802-878-5138

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STANDARD OPERATING PROCEDURE
ANALYSIS OF SAMPLE EXTRACTS FOR
SEMI-VOLATILE ORGANICS BY USEPA CLP PROTOCOLS

Abstract

This SOP addresses analysis of aqueous and solid matrix samples for semi-volatile (Base-Neutral/Acidic) organics as defined by the USEPA TCL List (EPA CLP). Samples are extracted, screened using capillary GC/FID, and then analyzed by capillary GC/MS. The specific protocols - the USEPA Contract Lab Program IFB. SOW 2/88 and updates are to be reviewed for complete descriptions including extraction, screening, sample analysis, data review, standard preparation and calibration protocols.

The intention of this SOP is to consolidate all aspects of the analyses in one working document (to be revised as necessary) for purposes of consistency in data quality.

The basic methodology involves extraction of samples with suitable solvent and concentration of the extract to obtain acceptable recoveries and detection limits for target compounds. A portion of the extract is then injected into the gas chromatograph for compound separation, and subsequently an electron Impact Ionization Mass Spectrometer for identification/quantitation.

1.0 CONVENTIONS

For organizational purposes all activities in the semi-volatile section have strict conventions for coding standards, data files, archive tapes, ID files, quantitation output files, header information and custom programs.

1.1 Standards

All standards are traceable to EPA and are either purchased through Supelco, Inc. or obtained from the EPA Repository. All standards, upon receipt, are logged in the appropriate log book with the date of receipt, source and lot number(s).

All standard preparations (see semi-volatile standard preparation SOP) are logged and coded. Our convention for coding all semi-volatile standards is as follows:

SV:xxxxxx:N:N

where SV = semi-volatiles
where xxxxxx = date of preparation
where the first N = page # of standard prep. log
where the second N = preparation log book #

On all vials labeled for storage, this code must appear along with a description (i.e., 40 ng/ul IS, etc.). All injection log entries of DFTPP, calibration checks, or calibration curve points must be annotated with this code.

1.2 Miscellaneous

The following naming conventions are utilized to consistently identify various files, programs, etc. to the respective instrument they are either utilized or generated on.

a) Data files
>Nxxxx

b) Quantitation output files
Nxxxx

c) ID files
I_N

d) Calibration files
C_N

e) Batch sequence files
S_Nxx

f) Tuning files
T_N

where N = Instrument ID
where x = Alpha numeric characters used in sequencing

2.0 SAMPLE ANALYSIS

2.1 Preliminary Activities

1) Check that the helium tank (carrier gas) has more than 250 psi. If it does not, please change the tank before proceeding.

2) Execute the command "FREE" to check that the data cartridge you are using and the QT cartridge have enough available space. If available space is <25 percent, purge files before proceeding.

3) If soils had been analyzed in the previous day's run, cool the oven and injection port (oven to 30°C, injection port to 50°C). Remove and replace the injection port septum. Replace the injection port liner and cut off the first 2 inches of the capillary column. A clean injection port liner should always be available that has been heated gently in chromic acid and then pre-silanized to deactivate the surfaces. If exceptionally dirty soils or waters have been run, deactivate the injection port with silanizing reagent. The silanizing reagent used is Sylon-CT, purchased from Supelco, Inc. Please follow the directions supplied with the Sylon-CT bottle explicitly when silanizing.

4) After analysis of water samples, step 3 may not be necessary and its necessity can only be determined after injection of the DFTPP 50 ppm/Calibration Check I mixture. If the 50 ppm DFTPP/Calibration Check standard does not meet USEPA criteria because of low acidic analyte recoveries, this step is mandatory.

5) Once the above has been completed inject 10 ul of 200 ug/ml acid shock mixture and heat the oven to 290°C and allow it to remain there for 15 minutes. Toggle purge B on for 10 seconds then turn off. During this same period of time, heat the injection port to 265°C.

6) Once the system has "baked" for 15 minutes, set it for the injection of 1.0 ul of the tuning check compound DFTPP/Calibration Check I at a concentration of 50 ng/ul. This is done in the following manner:

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a) Remove from the refrigerator the current DFTPP/Calibration Check I standard mixture. This contains DFTPP, the acid semi-volatile compounds and 3,3'-dichlorobenzidine. Allow it to temperature equilibrate at room temperature for 5 minutes.
b) Place in autosampler position #1.
c) Using the Aquarius II program "BEDIT," set the run to the following standard parameters (consult Aquarius II manual for further information and more detail:

- Sample size = 1 (1 ul)
- Bottle number = 1
- Archive the data file, quant identification file, and quant output file (answer "Y" to the appropriate prompts).
- Consult the injection log for the proper data file name and codes for standard and internal standard mix.
- Insert the proper identification file.
- Insert the proper method file.
- Dilution factor = 1.0

7) Once the run is finished (or at elapsed time = 25 minutes or longer), the DFTPP spectrum must be examined to determine if analysis can proceed.

a) Use the custom transfer file EZ to evaluate this. This will automatically evaluate the maximum scan of the DFTPP peak with averaging of the spectra (-2, +2 scans).
b) If not in criteria first manually examine other straight forward averaging to ensure a proper distribution of the mass ranges throughout the peak has been obtained.
c) If the instrument is not within EPA criteria, the system must be manually tuned. After tuning, DFTPP must be injected again and the mass spectrum evaluated again (See manual tuning section). Please save copies of all peak profiles for future reference in the appropriate instrument logbook.
d) If the DFTPP mass spectrum is in criteria, please print the following report.

- Put the log bottom soft key on and type MSH to pring the mass spectral header information.
- Take log bottom off and type DR,11,X,A to draw the mass spectrum. Press the graph display key and examine it to see if it is what you want to print. Press the graph copy key after this check.

- Type in NRM,100, which normalizes all masses to the base peak, which is set at 100 percent. Then, put log bottom on and type in:

  TAB to list the masses.
- Fill out the DFTPP Form V and place in a daily tuning/calibration folder (these will be filed once completed).

8) If DFTPP meets criteria, analysis can proceed. If running an initial calibration curve, nine additional data points need to be collected. Please consult the BNA standards SOP for preparation and calibration range. For sample analysis, proceed directly to step 9b.

9a) Once the quantitation report is produced, a combined calibration check report must be generated and evaluated, using the Aquarius command CBCHK. All calibration check compounds (CCC) must have a percent difference less than 25 percent. All system performance check compounds (SPCC) must have a response factor equal to or greater than 0.050. Furthermore, all TCL compounds in the initial calibration must be present in the 50 ppm calibration check mixture. The BNA 50 ppm Calibration Check II (which contains the base-neutral compounds) solution must next be run. This is set up in BEDIT in the same manner as the DFTPP/Calibration Check I run.

9b) Once the quantitation report is produced, a calibration check report must be generated and evaluated, using the Aquarius command CBCHK. All calibration check compounds (CCC) must have a percent difference less than 25 percent. All system performance check compounds (SPCC) must have a response factor equal to or greater than 0.050. Furthermore, all TCL compounds in the initial calibration must be present in the 50 ppm calibration check mixture.

10) For EPA CLP work, DFTPP and the calibration check standards must be performed for each 12-hour analysis period.

2.2 Batch Acquisition of Sample Data

1) Make sure that the methanol rinse vial is full and any waste containers necessary for the autosampler are empty. Additionally, inspect syringe for proper alignment and freedom of movement of the plunger.

2) Please be sure that all samples to be run are correctly entered into the injection log.

a) For NJDEP contract work, the injection time must be recorded next to each data file acquired in the instruments injection log.
b) All logs used for NJDEP contract work must be permanently bound.
c) All NJDEP contract injection logs must contain each analysts signature, not just the initials.
d) The unused areas at the bottom of every injection log page must be crossed out with a "Z" or "X" to prevent any further entries at a later date. The crossouts must be initialed and dated by the analyst performing the crossout.

3) Before starting up the "batch," use the QCAL command to update response factors from that day's calibration check mixture for the identification files which will be employed in the "batch."

4) Please ensure that the sequence file for the day's run contains the following standard items.

a) Sample size = 1 (1 ul)
b) Bottle numbers are sequenced 1 through the number of samples to be run.
c) All data files and output files need to be sequenced correctly.
d) Archive all data files, quant files, and identification files.
e) Employ the appropriate method and tune file.
f) Do a double check to make sure that the dilution factors are properly entered.
g) The header and miscellaneous information sections should contain the Case #, sample number and York Labs sample ID at a minimum.

5) Check that there is sufficient printer paper for the number of reports that will be hard copied.

6) Make sure that the printer is on-line and that the report terminal is logged off (to allow Aquarius to dump reports).

7) As the data comes off the printer, check surrogate and internal standad area criteria and enter in injection logbook. After examining the chromatography place any additional information necessary in the comments section.

a) If either internal standard areas or surrogate recoveries do not meet EPA CLP criteria, please notify the section leader so that reanalysis or re-extraction reanalysis may be scheduled as necessary.

2.3 Aqueous Sample Analysis Preparation - Low Level

Combine 300 ul of the base/neutral fraction, 300 ul of the acid fraction and 6 ul of the working internal standard mix (4,000 ng/ul) in an amber screw-cap type vial. Transfer 100 ml to a 110 ml autosampler vial. Enter the appropriate quantitation factor, calculated as follows:

$$\frac{1.000}{\text{amount (extracted (ml)}} \times \frac{1.000}{500} = QF$$

Please note that this quantitation factor is for the purposes of calculation only -- when reporting results, the dilution is 1.0, unless a 2-fold, 3-fold, or other dilution was necessary due to high viscosity and/or high analyte concentrations.

2.4 Soil Sample Analysis Preparation - Low Level

Place 300 ul of the soil extract in an amber screw-cap vial, along with 3 ul of the working internal standard mix. Enter the appropriate quantitation factor, calculated as follows:

$$\frac{1.000\ g/kg}{\text{dry weight (g)}} = QF$$

dry weight = (x g extracted) (% solids (decimal notation)

Please note that this quantitation factor is for the purposes of calculation only. As in aqueous samples, when reporting results, the dilution is 1.0, unless a higher level of dilution was necessary because of high viscosity and/or high analyte levels. If GPC clean up is used the QF must be multiplied by a factor of 2 and the CRQL adjusted accordingly.

2.5 Soil Sample Analysis Preparation - Medium Level

Place 300 ul of the soil extract in an autosampler vial, along with 3 ul of the internal standard mix. Enter the appropriate quantitation factor, calculated as follows:

$$\frac{1.000\ g/kg}{\text{dry weight (g)}} \times \frac{10\ ml}{5\ ml} = QF$$

dry weight = (x g extracted) (% solids (decimal notation)

Please note that this quantitation factor is for the purposes of calculation only. The same holds true for the reporting of dilution factors as for aqueous and low level soil samples.

3.0 MANUAL TUNING PROCEDURE

If you cannot achieve DFTPP criteria or the source has been cleaned, the following procedure can be followed to achieve DFTPP criteria.

1) Perform a full autotune after opening PFTBA valve.

2) With the PFTBA valve still open, go to manual tune and select EDIT. Press "scan parameters" and set the tune masses to 131, 219 and 502. Return to the top level of manual tune, press "execute" then press "parameter ramp" and the "ion focus." From the display note the ion focus setting which corresponds to the intersection of the 131 and 219 plots. Place the cursor at this intersection and press "set to cursor."

3) Confirm that the abundances of masses 131 and 219 are equal by returning to the top level, pressing "execute" and then "profile scan." Make a hard copy of this using the graph copy key. Turn off PFTBA.

4) At this point save the tune parameters by returning to the top level and pressing "store tune." Save the file as T_N (where N = instrument ID). Then exit Mtune.

5) Verify MS performance by injecting DFTPP and evaluating the spectrum.

6) Additional adjustment of lens voltages may be necessary if DFTPP is not in criteria at this point.

4.0 110 ul AUTOSAMPLER VIAL CLEANING PROCEDURE (other vials are discarded)

1) In the extraction lab, in a hood, and while wearing gloves - remove and dispose of the septum cap;

2) Place vials in a 400 ml beaker of acetone;

3) Place beaker in ultrasonic cleaner water bath for 10-15 minutes with heat applied to the water bath;

4) Decant solvent into waste solvent drum;

5) Add sufficient methylene chloride to the beaker to cover the vials and resonicate for 20 minutes with heat;

6) Decant solvent to waste solvent drum. Repeat 5 again;

7) Add sufficient methanol to the beaker to cover the vials and sonicate for 10 minutes;

8) Decant methanol into water solvent drum.

9) Oven dry for one hour @ 103°C.

5.0 SCHEDULE OF QA/QC VALIDATION CHECKS

1) All working solutions must be traced to EPA reference standards when prepared and once per quarter if not purchased from a supplier who performs this.

2) Blind and double blind QC checks must be run as required by the organics QA/QC officer.

6.0 INITIAL DATA REVIEW

Once the data acquisition for a batch of samples has been started, the GC/MS operator immediately completes initial data review for the data collected the preceding shift. This data review examines six key criteria for semi-volatiles:

- 12-hour Clock Criteria
- Surrogate Recoveries
- Internal Standard Areas
- Chromatography
- Target Analyte Concentrations
- Updated Response Factors (QCAL)
- Method Blank Criteria

These criteria are examined and summarized on the semi-volatile injection logbook and batch summary sheet (Figures 2.0 and 2.1). These will be verified and countersigned within one working day by the section leader or his designate. The preliminary data rejection criteria for a RERUN flag on the injection log are as follows:

- Surrogates - recoveries are evaluated using the following criteria (EPA CLP, SOW 2/88):

	Surrogates	Water %	Soil %
Base/Neutral Surrogates	Nitrobenzene-d_5	35-114	23-120
	2-Fluorobiphenyl	43-116	30-115
	Terphenyl-d_{14}	33-141	18-137

	Surrogates	Water %	Soil %
Acid Surrogates	Phenol-d_5	10-94	24-113
	2-Fluorophenol	21-100	25-121
	2,4,6-Tribromophenol	10-123	19-122

1) All surrogates for method blanks must be within the above ranges.

2) For samples no more than one surrogate can be outside the above ranges in either the Base/Neutral or Acid fraction. (If out after reanalysis a corrective action must be submitted to the Extractions Group to schedule re-extraction of the sample.)

3) Recoveries must be ≥10 percent for all surrogates.

- Internal Standards - must not be <50 percent or >100 percent of the EICA (extracted ion current area) of the corresponding calibration check internal standards (DFTPP/Calibration Check I). (If the same internal standard is out on reanalysis both must be submitted.)

- Chromatography - there should be no "ghost" peaks present in the chromatogram.

- Target compound concentrations - For a diluted sample all samples must have the analyte of the highest concentration under 160 ppb (times the dilution factor). They must also be above 80 ppb (times the dilution factor).

A case folder is then prepared and all data applicable to that particular case is placed in it to await further reduction. The case folder must include the following.

- Case narrative sheet describing all irregularities
- Copy of injection log
- Form V DFTPP and spectral tab for DFTPP for initial calibration curve
- Initial calibration curve (Form VI)* plus medium quant reports for individual standards if CLP.
- DFTPP for "batch" of samples (Form V and spectral tab)
- Continuing calibration check report (Form VII)*
- Continuing calibration quant report (medium)
- Copies of the injection logbook
- Sample quant reports (full)
- Matrix spike/matrix spike duplicate quant reports (medium)

*Please note these are the Hewlett-Packard generated forms.

Method Blank Criteria

Method Blanks must contain less than the CRQL of the TCL List of compounds excepting common phthalateesters which must be less than 3X the CRQL (See Figure 8.0).

7.0 FINAL DATA REVIEW

All final data judgment/review for target compounds and tentatively identified compounds is done according to USEPA CLP protocols (SOW 2/88).

The target analytes are flagged appropriately in accordance with the following conventions. The flags are placed directly to the right of the rounded-off value. All semi-volatile data is reported to two significant figures except when values are less than 10, then one significant figure is used.

Flag Conventions:

See Sheet

Surrogates are calculated according to USEPA CLP protocols (SOW 2/88). If a dilution of the extract is made flag the surrogate recovery as (DL). Record surrogate recoveries to the right of the concentration columns of the quant report (see Figure 3.0).

Sign and date all quantitation reports when reviewing data (see Figure 3.0). All mass spectra which do not meet EPA CLP criteria must be crossed out with a clean single line drawn through the quant report hit and concentration for that compound. Only the quantitation reports, chromatograms and positive hits are reported. All other spectra are saved to be placed in the case purge file. Subsequent to final data review, the package is signed and dated and given to the data reporting/reduction section. Every page of raw data must be signed and dated by the data reviewer for NJDEP contract work.

7.1 Quality Control Approval Report (QCAR)

A QCAR is completed for each deliverables report prepared and submitted along with the package. Please refer to Figure 4.0.

The QCAR consists of ten check points which are carefully reviewed by the person performing the initial data review and/or the final data reviewer. The ten approval check points are as follows:

7.1.1 Analysis QCAR's are complete

Each QC batch associated with the deliverable report should have an associated analysis QCAR (Figure 8.0), which has been completed and filed with the QC batch.

7.1.2 Client and sample ID's match

The deliverables report is checked to ensure that the client ID's reported match the lab ID's from the traveler reports.

7.1.3 Raw data has been sorted

All raw data which is not required in the deliverables report must be removed and placed in an extra data file which will accompany the report.

7.1.4 All samples and QC present

A review is made to ensure that all required samples, reanalyses, blanks, duplicates and matrix spikes/matrix spike duplicates are present and reported.

7.1.5 All raw ata present and legible

A review is performed to ensure that all original and/or photocopies of the data are legible and present.

7.1.6 All required forms completed

A review is performed of the deliverables package to ensure that all forms are completed which are required for the level of report requested.

7.1.7 Case narrative completed

Every deliverables report must have a completed case narrative which documents any deviations from the SOP which were required or any problems which were encountered.

7.1.8 Result transcriptions checked

The final reviewer checks all reported results to ensure that no transcription errors have occurred.

7.1.9 Analysis holding times meet criteria

Each sample is checked by the final reviewer to ensure that the protocol required holding times have been met and that any deviations have been documented in the case narrative.

7.1.10 Deliverables in correct order

The final reviewer places the deliverables report in its proper order before submission to data management.

The final reviewer signs the bottom of QCAR as final approval to release the data to data management.

7.2 Qualitative and quantitative identification of target compounds

7.2.1 Qualitative determination

1) Relative intensities of major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

2) The relative intensities of the major ions should agree within ± 20 percent. (Example: For an ion with an abundance of 50 percent in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

3) Molecular ions present in the reference spectrum should be present in sample spectrum.

4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. NOTE: Data system library reduction programs can sometimes create these discrepancies.

If a compound cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the contractor shall report that identification, with comment.

1) Target compounds identified shall be quanitified by the internal standard method. The internal standard used shall be the one nearest the retention time to that of a given analyte. The EICP area of the characteristic ions for the target compounds are used.

2) Internal standard responses and retention times in all standards must be evaluated during or immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system must be inspected for malfunctions and corrections made as required. The extracted ion current profile (EICP) of the internal standards must be monitored and evaluated for each sample, blank, matrix spike and matrix spike duplicate. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometric system must be inspected for malfunction and corrections made as appropriate. If the analysis of a subsequent sample or standard indicates that the system is functioning properly, then corrections may not be required. The samples or standards with EICP areas outside the limits must be reanalyzed and treated according to sections 3) and 4) below. If corrections are made, then the laboratory must demonstrate that the mass spectrometric system is functioning properly. This must be accomplished by the analysis of a standard or sample that <u>does</u> meet the EICP criteria. After corrections are made, the reanalysis of samples analyzed while the system was malfunctioning is required.

3) If after reanalysis, the EICP areas for all internal standards are inside the contract limits (-50% to +100%), then the problem with the first analysis is considered to have been within the control of the laboratory. Therefore, <u>only submit</u> data from the analysis with EICP's within the contract limits. This is considered the <u>initial</u> analysis and must be reported as such on all data deliverables.

4) If the reanalysis of the sample does not solve the problem, i.e. the EICP areas are outside the contract limits for both analyses, then submit the EICP data and sample data from both analyses. Distinguish between the initial analysis and the reanalysis on all data deliverables. Document in the case narrative all inspection and corrective actions taken.

The relative response factor (RRF) from the daily standard analysis is used to calculate the concentration in the sample. Secondary ions may be used if interferences are present. The area of a

secondary ion cannot be substituted for the area of a primary ion unless a relative response factor is calculated using the secondary ion. When target compounds are below required quantitation limits but the spectra meets the identification criteria, report the concentration with a "J". For example, if CRQL is 10 ug/L and concentration of 3 ug/L is calculated, report as "3J".

Water

Concentration $$ug/L = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_o)(V_i)}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured
A_{is} = Area of the charactierstic ion for the internal standard
I_s = Amount of internal standard added in nanograms (ng)
V_o = Volume of water extracted in milliliters (mL)
V_i = Volume of extract injected (uL)
V_t = Volume of total extract

(Use 2000 uL or a factor of this when dilutions are made. The 2000 uL is derived from combining half of the 1 mL BN extract and half of the 1 mL A extract.)

Sediment/Soil

Concentration (dry weight basis) $$ug/Kg = \frac{(A_x)(I_s)(V_t)}{(A_{is})(RRF)(V_i)(W_s)(D)}$$

Where:

A_x, I_s, A_{is} = same as given for water, above
V_t = Volume of low level total extract (use 1000 uL or a factor of this when dilutions are made). If GPC cleanup is used, the volume is 2000 uL. The 1000 uL is derived from concentrating the 9.5 mL extract to 0.95 mL.)

- OR -

V_t = Volume of medium level extract (use 2000 uL or a factor of this when dilutions are made. The 2000 uL is derived from concentrating 5 mL of the 10 mL extract to 1 mL.).
V_i = Volume of extract injected (uL)
D = $\frac{100 - \text{percent moisture}}{100}$
W_s = Weight of sample extracted (g)

7.3 Tentatively identified compounds

A library search is executed when requested for up to 20 non-target compounds of greatest apparent concentration for the purpose of tentative identification and quantitation. Compounds with responses less than 10 percent of the nearest internal standard are not required to be searched. Aldol condensation product should not be counted toward the 20 non-target compound total.

7.3.1 The following guidelines are for making tentative identification

1) Relative intensities of major ions in the reference spectrum (ions greater than 10 percent of the most abundant ion) should be present in the sample spectrum.

2) The relative intensities of the major ions should agree within ±20 percent. (Example: For an ion with an abundance of 50 percent in the standard spectra, the corresponding sample ion abundance must be between 30 and 70 percent.)

3) Molecular ions present in reference sectrum should be present in sample spectrum.

4) Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

5) Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting compounds. NOTE: Data system library reduction programs can sometimes create these discrepancies.

7.3.2 If in the technical judgment of the mass interpretation spectral specialist, no valid tentative identification can be made, the compounds should be reported as unknown. The mass spectral specialist should give additional classification of the unknown compound, if possible (i.e., unknown phthalate, unknown hydrocarbon, unknown acid type, unknown chlorinated compound). If probable molecular weights can be distinguished, include them.

7.3.3 An estimated concentration for the tentatively identified compounds is quantified by the internal standard method assuming a relative response factor of one. The value obtained is qualified as estimated using the "J" qualifier.

8.0 DATA REPORTING/COLLATING

Subsequent to final data review, all forms must be generated on the applicable PC and a diskette produced (See Diskette SOP). Once all forms are generated the case narrative materials which include corrective action reports (Figure 5.0) and comments noted on the semi-volatile job breakdown sheet (Figure 6.0) are collated and a case narrative for semi-volatiles is written. This is then dated and signed (see Figure 7.0).

9.0 DATA VALIDATION

Once a case has been reported, it is then double checked by a second person. All criteria listed on the semi-volatile quality approval form must be countersigned.

Once complete, the case is submitted to the semi-volatile Section Leader for final approval. The case is then submitted to the Project Manager.

FIGURE 1.0

SEMI-VOLATILE MAINTENANCE CHECKLIST - MS

/ /

	Freq	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Change Linear	WN															
Change Septa	D															
Cut Column	WN															
Acid Shock(s)	D															
Change Syringe	WN															
Change He Tank	WN															
Change Column	WN															
Clean Source	WN															

	Freq	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31
Change Linear	WN															
Change Septa	D															
Cut Column	WN															
Acid Shock(s)	D															
Change Syringe	WN															
Change He Tank	WN															
Change Column	WN															
Clean Source	WN															

Comments: _____

301014

FIGURE 5.1.2

GC/MS INJECTION LOG BOOK — EA LABORATORY: CONNECTICUT

STANDARD CODES:	ROUTINE MAINTENANCE PERFORMED:	QC BATCH:
/		DATE:
/		MATRIX:
/		_____ IDFILE QCALED @ ___
/		
/		

Calibrated for the following (check) :

Fraction= (circle) VOA BNA PROTOCOL= LIST= TAL ( ) PP ( ) Other _____ CLP 2/88 ( ) CLP 3/90 ( ) Other _____

DATA FILE	CLIENT JOB NUMBER	SAMPLE ID	INJ. TIME	ALS POS.	DF	QF	ANALYST'S SIGNATURE	COMMS
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
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>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					
>			:					

WITNESSED BY:	Date:	Book No.	Page No.

301015

[illegible] on the date of 07/05/89

FIGURE 2.1

Reviewed By: [signature] Date: 7/5/89
Checked By: [signature] Date: 7/6/89

Sample #	Data File	Internal Standards 1 4 area	2 5 area	3 6 area	Surrogates NBZ FBP PHL %	TPH ZFP TBP %		Flags	Chrom. Acceptable Y or N	Fraction. Data Acceptable Y or Refun	
CAL CHECK	SSPPH STRL	XC0812	55332	210156	116905	NR	NR	NR		Y or N	Y or Refun
			263120	252932	217618	NR	NR	NR			
	1153081	XC4197	40946	155677	103798	32*	76	169*		Y or N	Y or Refun RRP
			220824	159881	102432*0	27	32	84			
	06/30-B01	XC4198	42549	165701	98797	82	91	137		Y or N	Y or Refun
			238431	195019	124018	36	52	96			
	1153002BE	XC4199	45773	177858	111488	41	86	87		Y or N	Y or Refun Inj LES a-L Re.
			227371	196372	139486	28	56	72			
	1153005BE	XC4200	41392	158791	105590	82	88	123		Y or N	Y or Refun
			219708	193467	132532	28	29	61			
	0630-B04	XC4201	46676	178135	103969	88	96	144*		Y or N	Y Refun Surr
			238343	191476	148161	84	74	97			
	1143002	XC4202	55788	271227	1011135	74	103	166*	*0	Y or N	Y or Refun RRL:100
			136775	50185*0	32619*0	77	74	37			
	06/29-B02	XC4203	47177	175482	99588	92	105	113		Y or N	Y or Refun
			245158	208156	128927	82	78	70			
	1159001	XC4204	47298	187958	108934	53	106	116		Y or N	Y or Refun
			265112	212778	165718	84	70	46			
	1159001MS	XC4205	43811	170683	100888	90	95	121	*0	Y or N	Y or Refun
			211785	205388	150839	85	81	66			
	1159001MSD	XC4206	51436	202481	120626	96	102	119	*0	Y or N	Y or Refun
			283929	229658	187615	88	86	85			
	1159002	XC4207	51404	196821	110988	94	106	114		Y or N	Y or Refun
			257646	211819	158615	89	84	56			

DFTPP Inj. Time is 13:13 Cal Check Calibration Time :06/30/89 18:38
Last Inj. Time is 00:00 Blank IB Calibration Time :07/05/89 15:03

SURROGATE LIMITS	WATER	SOIL
S1 (NBZ) = Nitrobenzene-d5	(35-114)	(23-120)
S2 (FBP) = 2-Fluorobiphenyl	(43-116)	(30-115)
S3 (TPH) = Terphenyl-d14	(33-141)	(18-137)
S4 (PHL) = Phenol-d5	(10-94)	(24-113)
S5 (ZFP) = 2-Fluorophenol	(21-100)	(25-121)
S6 (TBP) = 2,4,6-Tribromophenol	(10-123)	(19-122)

*= Values outside of contract required QC Limits

+4 = Area is) 200% or (50% of Cal Check
+0 = RT is Plus or Minus .5 min. of Cal Check
+6 = There is a compound in the sample over 100

301016

FIGURE 3.0

QUANT REPORT

Operator ID: MSC Quant Rev: 6 Quant Time: 890616 1
Output File: ^C4050::QT Injected at: 890616 1
Data File: >C4050::C4 Dilution Factor: 2.0
Name: ;;;SBLKC4050
Misc: 06/12-802 INST.ID:C;;061289;LLW;1;;SEPF BTL# 1

ID File: I_C::N2
Title: SEMIVOLATILE BNA COMPOUNDS
Last Calibration: 890616 12:53

	Compound	R.T.	Q ion	Area	Conc	
1)	*1,4-Dichlorobenzene-d4	11.87	151.8	56630	40.00	ug
4)	2-Fluorophenol	8.90	111.8	78549	92.81	ug
6)	Phenol-d5	11.13	98.8	66378	57.61	ug
20)	*Naphthalene-d8	15.10	135.9	204782	40.00	ug
21)	Nitrobenzene-d5	13.33	81.8	65213	66.48	ug
35)	*Acenaphthene-d10	19.71	163.9	115166	40.00	ug
39)	2-Fluorobiphenyl	17.96	171.8	127440	67.64	ug
52)	Diethylphthalate	20.96	148.8	2702	.97	ug
56)	2,4,6-Tribromophenol	21.79	329.6	78585	135.97	ug
57)	*Phenanthrene-d10	23.56	187.9	294030	40.00	ug
68)	*Chrysene-d12	30.66	240.0	175385	40.00	ug
71)	Terphenyl-d14	27.75	244.0	259516	72.61	ug
~~72)~~	~~Butylbenzylphthalate~~	~~29.09~~	~~149.0~~	~~941~~	~~.33~~	~~ug~~
76)	bis(2-Ethylhexyl)phthalate	30.81	149.8	15706	3.72	ug
77)	*Perylene-d12	36.87	264.0	106391	40.00	ug

* Compound is ISTD

DS 6/19/89

FAX (203) 268-5348

FIGURE 4.0

QUALITY CONTROL APPROVAL REPORT

GC/MS Group Volatiles _____ Semivolatiles _____

Job Number:_____ Client:_____

Case Number:_____ SDG Number:_____

Deliverables Requested:_____

QC Batches:_____ _____ _____ _____ _____

_____ _____ _____ _____ _____

	Initial Approval		Final Approval	
	Initials	Date	Initials	Date
1. The Analysis QCAR's are complete	_____	_____		
2. Client and sample ID's match	_____	_____		
3. Raw data has been sorted	_____	_____		
4. All samples and QC present	_____	_____	_____	_____
5. All raw data present and legible	_____	_____	_____	_____
6. All required forms completed	_____	_____	_____	_____
7. Case Narrative completed	_____	_____	_____	_____
8. Result transcriptions checked			_____	_____
9. Analysis holding times meet criteria			_____	_____
10. Deliverables in correct order			_____	_____

This data meets the requirements of the GC/MS SOPs unless otherwise documented in the case narrative.

Final Approval
to Release Data:_____Date:_____

301018

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4466
FAX (203) 268-3348

QUALITY CONTROL APPROVAL REPORT

GC/MS Analysis Checklist

QC Batch: _____

	Approvals	
	Initials	Date
1. Chain-of-Custody Forms have been completed	_____	_____
	_____	_____
2. Initial Calibration meets the following criteria CLP-02/88 ( ) CLP-03/90 ( ) Other_____ ( )	_____	_____
2A. Continuing Calibration meets the following criteria CLP-02/88 ( ) CLP-03/90 ( ) Other_____ ( )	_____	_____
3. Retention times have been verified in the standards	_____	_____
4. QC Batch folder is complete as per SOP	_____	_____
5. Form III generated (if required)	_____	_____
6. IDRS criteria met for all accepted analysis and is entirely complete	_____	_____
7. All raw data is present with correct header information and quantitation factors	_____	_____
8. Required TIC's have been qued for generation on the TIC request form	_____	_____
9. All data has been captured on the PC	_____	_____
10. Sample tracking/breakdown sheets have been entirely updated for all batch results	_____	_____
11. All Required TIC's have been generated	_____	_____
12. Archive Directory has been generated	_____	_____

This data meets the requirements of the GC/MS SOPs unless otherwise documented in a Corrective Action Report:

_____ Date: _____

Authorizing Signature

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4468
FAX (203) 268-5348

Reported By: _____

Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE Re

Client: _____ Job Number: _____ Sample Number(s): _____

Problem Reported By: _____

Category of Problem: _____

Poor Communication With ... Sample Matrix Inf

Description of Problem: _____

See back of CAR/CNR or attached sheet if more space is requir

Recommended Corrective Action(s): _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline For CAR: _____ Reply to Originator Required?

Actual Corrective Action(s) Taken: _____

Document Actual Corrective Actions Ta

Form and Send a Copy of Completed CAR/CNR to QA Officer and Job Fo

_____
Signature and Date

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Initial Lab manager, Marketing Manager, Branch Manager, QA Offi

Distribution: Client Services, Job Folder, Other: _____

SECONDARY DISTRIBUTION: ORIGINATOR, QA OFFICER, OTHER: _____

Document Problem and Corrective Action(s) In Case Narrative: [ ]

Attachments: Telephone Log, Services Change Request, Memo, Other:

301020

CLIENT JOB NUMBER : PROTOCOL : COMPOUND LIST :

ID	Loc.	pH	Solid	Init. Wt./ Vol.	Final Vol.	GPC YorN	Dil. Fac.	Quant. Factor	Final Quant. Factor	Rerun Comments	Data File Analysis	Data File Reanalysis

Comments:

3010214

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4468
FAX (203) 268-6348

CASE NARRATIVE

GC/MS Group

Volatiles:_____
Semivolatiles:_____

IEA Job No.: _____
Client : _____
Case No.:_____
SDG No.:_____

Signature : _____ Date :_____

301022

FIGURE 8.0
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)*

	Semi-Volatiles	Case Number	Precision	Accuracy	Quantitation Limits (water ug/L a.b CRQL	
1.	Phenol	108-95-2	±6	37	10	
2.	bis(2-Chloroethyl)ether	11-44-4	±14	82	10	
3.	2-Chlorophenol	95-57-8	±11	77	10	2
4.	1,3-Dichlorobenzene	541-73-1	±13	67	10	
5.	1,4-Dichlorobenzene	106-46-7	±13	68	10	
6.	Benzyl alcohol	100-51-6	±21	77	10	
7.	1,2-Dichlorobenzene	95-50-1	±13	71	10	
8.	2-Methylphenol	95-48-7	±11	72	10	5
9.	bis(2-Chloroisopropyl)ether	108-60-1	±14	81	10	1
10.	4-Methylphenol	106-44-5	±10	66	10	4
11.	N-Nitroso-di-n-dipropylamine	621-64-7	±14	81	10	1
12.	Hexachloroethane	67-72-1	±14	60	10	1
13.	Nitrobenzene	98-95-3	±12	81	10	1
14.	Isophorone	78-59-1	±14	87	10	1
15.	2-Nitrophenol	88-75-5	±11	83	10	2
16.	2,4-Dimethylphenol	105-67-9	±15	69	10	8
17.	Benzoic acid	65-85-0	±20	27	50	16
18.	bis(2-Chloroethoxy)methane	111-91-8	±13	87	10	1
19.	2,4-Dichlorophenol	120-83-2	±12	84	10	2
20.	1,2,4-Trichlorobenzene	120-82-1	±14	75	10	1
21.	Naphthalene	91-20-3	±12	77	10	1
22.	4-Chloroaniline	106-47-8	±36	65	10	1
23.	Hexachlorobutadiene	87-68-3	±15	67	10	1
24.	4-Chloro-3-methylphenol (para-chloro-meta-cresol)	59-50-7	±15	82	10	2
25.	2-Methylnaphthalene	91-57-6	±11	72	10	1
26.	Hexachlorocyclopentadiene	77-47-4	±11	28	10	1
27.	2,4,6-Trichlorophenol	88-06-2	±12	88	10	2
28.	2,4,5-Trichlorophenol	95-95-4	±14	90	50	6
29.	2-Chloronaphthalene	91-58-7	±10	83	10	1
30.	2-Nitroaniline	88-74-4	±16	91	50	1
31.	Dimethylphthalate	131-11-3	±23	49	10	1
32.	Acenaphthylene	208-96-8	±9	85	10	1
33.	2,6-Dinitrotoluene	606-20-2	±13	97	10	2
34.	3-Nitroaniline	99-09-2	±19	109	50	3
35.	Acenaphthene	83-32-9	±9	86	10	1
36.	2,4-Dinitrophenol	51-28-5	±30	74	50	4
37.	4-Nitrophenol	100-02-7	±20	22	50	4
38.	Dibenzofuran	132-64-9	±10	91	10	1
39.	2,4-Dinitrotoluene	121-14-2	±19	104	10	1
40.	Diethylphthalate	84-66-2	±21	80	10	2
41.	4-Chlorophenyl-phenyl ether	7005-72-3	±10	94	10	1
42.	Fluorene	86-73-7	±12	93	10	1
43.	4-Nitroaniline	100-01-6	±42	90	50	4
44.	4,6-Dinitro-2-methylphenol	534-52-1	±18	70	50	2
45.	N-nitrosodiphenylamine	86-30-6	±15	99	10	2

FIGURE 8.0
TARGET COMPOUND LIST (TCL) AND
CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)*(Continued)

	Semi-Volatiles	Case Number	Precision	Accuracy	Quantitation Limits (water ug/L a,b ** CRQL
46.	4-Bromophenyl-phenylether	101-55-3	±9	91	10
47.	Hexachlorobenzene	118-74-1	±10	93	10
48.	Pentachlorophenol	87-86-5	±19	68	50
49.	Phenanthrene	85-01-8	±10	90	10
50.	Anthracene	120-12-7	±9	87	10
51.	Di-n-butylphthalate	84-74-2	±12	88	10
52.	Fluoranthene	206-44-0	±13	88	10
53.	Pyrene	129-00-0	±13	91	10
54.	Butylbenzylphthalate	85-68-7	±15	93	10
55.	3,3'-Dichlorobenzidine	91-94-1	±6	68	20
56.	Benzo(a)anthracene	56-55-3	±11	91	10
57.	Chrysene	218-01-9	±11	95	10
58.	bis(2-Ethylhexyl)phthalate	117-81-7	±13	96	10
59.	Di-n-octylphthalate	117-84-0	±16	102	10
60.	Benzo(b)fluoranthene	205-99-2	±14	88	10
61.	Benzo(k)fluoranthene	207-08-9	±30	86	10
62.	Benzo(a)pyrene	50-32-8	±11	87	10
63.	Indeno(1,2,3-cd)pyrene	193-39-5	±13	98	10
64.	Dibenz(a,h)anthracene	53-70-3	±12	94	10
65.	Benzo(g,h,i)perylene	191-24-2	±13	94	10

a. Low soil/sediment contract required quantitation limits (CRQL and MDL's) for semi-volatile TCL compounds are 33 times the individual water CRQL and MDL.

b. Medium soil/sediment contract required quantitation limits (CRQL and MDL) for semi-volatile TCL compounds are 60 times the individual low soil/sediment CRQL and MDL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

+ The reference to the MDL calculation is Federal Register 40 CFR, Part 136 Appendix B October 26, 1984.

The reference to the Precision and Accuracy is SW846, 3rd Edition, Chapter One, Section 1.1.8.

individual Mix A and B at Level II (See Table 9.3) are prepared to 25 ml volumes as needed. These are used for the 72 hour run sequence. (250 ul of the stock is diluted to 25 ml hexane). For multi-response compounds any one of the working standards may be used.

9.2 Standard Preparation Frequency

The following table directs the frequency with which all Pesticide/PCB standards are to be prepared.

Upon preparation of a standard, it is to be labeled with date, initials, concentration, and code. This information is also entered into the Standards Preparation Log Book. EPA stocks are to be dated upon receipt and coded and dated when opened.

Standard	Preparation Replenishment Frequency	Storage Containers
EPA Individual Stocks	Open new stock every 6 months	2 ml amber vials - silanized
Mixed Stocks	every 6 months	15 ml silanized vials
Working Standards	every 6 months	silanized vials for volume size

9.3

The convention for coding Pesticide/PCB standards is as follows:

GC:xxxxxx:n

Where GC = GC Group
Where xxxxxx = date of preparation
Where n = page of standards prep log book

On all vials labeled for storage, this code must appear along with a description (i.e. PCB 1242 2.0 ug/ml). Codes shall be annotated in injection log book. Standard vials should always be marked at the bottom of the meniscus to detect any evaporation of the standard.

9.4 Stock Standard Log-In

When standards are received they are logged-in on a 4x6 index card. Compound identification, concentration and solvent determine the heading of the card. If the new standard has the same concentration and solvent, it is tagged with the next ampule number and a sample seal stamped with the date received and ampule number.

The index card has further information about the lot number of the standard location of the standard, date received and by whom, date used and by whom. All of these must be filled in for each standard received. If no card exists for the standard a new card is made up. This is illustrated in Figure 52.

10.0 MISCELLANEOUS PESTICIDE ANALYSIS

10.1

Analysis of Pesticides not on the PP list or TCL list is frequently requested. An example of this would be Pesticides from the Appendix IX list such as Isodrin, Kepone and Chlorobenzilate. Any of the miscellaneous pesticide standards may be added to the existing 72-hour sequence as long as EPA/CLP samples are not being analyzed.

10.2

Prior to sample analysis, an initial-5 point calibration should be run on the GC and column the samples will be analyzed on. The date from the curve should be one file so C_f can be referred to. Reference Table XIV for concentration ranges for miscellaneous pesticides currently analyzed for. If not on this list then retention time and response must be established prior to the curve.

10.3

If samples are analyzed independent of the 72-hour sequence, then a 3 point standard curve must be analyzed. Compare the average C_f to that of the original 5-point curve. If the percent difference is $\leq \pm 20$ percent then proceed with samples if not the 5 point curve must be reanalyzed every 10 samples and must be $\leq \pm 15$ percent difference from the daily 3point curve before proceeding with any more samples.

TABLE VII
PESTICIDE INDIVIDUAL MIX A - STOCK

Compound	Initial Concentration	Amount Added	Final Volume	Final Concentration (ug/ml)
Lindane	5,000 ug/ml	4 ul	10 ml	2.0
Heptachlor	5,000 ug/ml	4 ul	10 ml	2.0
Aldrin	5,000 ug/ml	4 ul	10 ml	2.0
Heptachlor Epoxide	5,000 ug/ml	10 ul	10 ml	5.0
Endosulfan I	5,000 ug/ml	10 ul	10 ml	5.0
Dieldrin	5,000 ug/ml	10 ul	10 ml	5.0
4,4'-DDT	5,000 ug/ml	40 ul	10 ml	20.0
Endrin Aldehyde	5,000 ug/ml	40 ul	10 ml	20.0
Endosulfan II	5,000 ug/ml	20 ul	10 ml	10.0
Methoxychlor	5,000 ug/ml	50 ul	10 ml	25.0
DBC (Surrogate)	5,000 ug/ml	20 ul	10 ml	10.0

TABLE VIII
PESTICIDE INDIVIDUAL MIX A - DILUTIONS

Compound	Concentration Stock (ug/ml)	Concentrations Level I (50 ul)	Level II (100 ul)	Level III (200 ul)	Level V (500 ul)
Lindane	2.0	0.01	0.02	0.04	0.10
Heptachlor	2.0	0.01	0.02	0.04	0.10
Aldrin	2.0	0.01	0.02	0.04	0.10
Heptachlor Epoxide	5.0	0.025	0.05	0.10	0.25
Endosulfan I	5.0	0.025	0.05	0.10	0.25
Dieldrin	5.0	0.025	0.05	0.10	0.25
4,4'-DDT	20.0	0.10	0.20	0.40	1.0
Endrin Aldehyde	20.0	0.10	0.20	0.40	1.0
Endosulfan II	10.0	0.05	0.10	0.20	0.50
Methoxychlor	25.0	0.125	0.25	0.50	1.25
DBC (Surrogate)	10.0	0.05	0.10	0.20	0.50

Final volumes are 10 ml.

TABLE IX
PESTICIDE INDIVIDUAL MIX B - STOCK

Compound	Initial Concentration	Amount Added	Final Volume	Final Concentration (ug/ml)
Alpha BHC	2,500 ug/ml	8 ul	10 ml	2.0
Beta BHC	2,500 ug/ml	20 ul	10 ml	5.0
Delta BHC	5,000 ug/ml	4 ul	10 ml	2.0
Aldrin	5,000 ug/ml	4 ul	10 ml	2.0
Gamma Chlordane	5,000 ug/ml	20 ul	10 ml	10.0
Alpha Chlordane	5,000 ug/ml	20 ul	10 ml	10.0
4,4'-DDE	5,000 ug/ml	10 ul	10 ml	5.0
Endrin	5,000 ug/ml	20 ul	10 ml	10.0
4,4'-DDD	5,000 ug/ml	20 ul	10 ml	10.0
Endosulfan Sulfate	5,000 ug/ml	100 ul	10 ml	50.0
Endrin Ketone	2,000 ug/ml	50 ul	10 ml	10.0
DBC	5,000 ug/ml	20 ul	10 ml	10.0

TABLE X
PESTICIDE INDIVIDUAL MIX B - DILUTIONS

Compound	Concentration Stock (ug/ml)	Concentrations Level I (50 ul)	Level II (100 ul)	Level III (200 ul)	Level IV (500 ul)
alpha BHC	2.0	0.01	0.02	0.04	0.10
beta BHC	5.0	0.025	0.05	0.10	0.25
delta BHC	2.0	0.01	0.02	0.04	0.10
gamma Chlordane	10.0	0.05	0.10	0.20	0.50
alpha Chlordane	10.0	0.05	0.10	0.20	0.50
Aldrin	2.0	0.01	0.02	0.04	0.10
4,4'-DDE	5.0	0.025	0.05	0.10	0.25
Endrin	10.0	0.05	0.10	0.20	0.50
4,4'-DDD	10.0	0.05	0.10	0.20	0.50
Endosulfan Sulfate	50.0	0.25	0.50	1.0	2.5
Endrin Ketone	10.0	0.05	0.10	0.20	0.50
DBC	10.0	0.05	0.10	0.20	0.50

Final volumes are 10 ml.

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
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STANDARD OPERATING PROCEDURE

PERKIN-ELMER 3030 ATOMIC ABSORPTION SPECTROMETER (GRAPHITE FURNACE)

AS:070987:2

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

STANDARD OPERATING PROCEDURE
PERKIN-ELMER 3030 ATOMIC ABSORPTION SPECTROMETER (GRAPHITE FURNACE)

INTRODUCTION

This method outlines the operation of the Perkin-Elmer 3030 for graphite furnace analysis of environmental samples.

It is the intent of this procedure to comply with the IEA business ethics policy.

1.0 SCOPE OF APPLICATION

1.1 This procedure describes the analysis of water and soil samples using the Perkin-Elmer Model 3030 Atomic Absorption Spectrophotometer.

1.2 The analytes determined using this procedure are:

Arsenic Thallium Selenium Lead

2.0 SUMMARY OF METHOD

2.1 Samples are digested with acid and peroxide according to the sample preparation standard operating procedure for furnace.

2.2 Appropriate quality control check standards are measured according to EPA or routine protocols at the beginning, throughout, and at the end of the analysis to assure measurement is accurate.

3.0 INTERFERENCES

Refer to manufacturer's instructions regarding specific elements.

4.0 APPARATUS AND MATERIALS

4.1 Perkin-Elmer Model Zeeman/3030 Atomic Absorption Spectrophotometer

4.2 Perkin-Elmer Autosampler Model AS-60

4.3 Perkin-Elmer HGA-600 Graphite Power Unit

4.4 Perkin-Elmer PR-100 Printer

4.5 1,000 uL variable Eppendorf pipette

4.6 2.0 mL autosampler cups

4.7 20 uL fixed Eppendorf pipette

4.8 Furnace stock standards as described in the Furnace Standards and Solutions Preparation Logbook (Appendix A)

4.9 EPA Quality Control Standard Solutions as described per EPA in the Atomic Spectroscopy Reagents Logbook (Appendix C)

4.10 Matrix modifier solutions as described in Furnace Standards and Solutions Preparation Logbook (Appendix D)

5.0 REAGENTS

5.1 Furnace working standards are made fresh daily from a standard stock solution and recorded in the Furnace Standards and Solutions Preparation Logbook (Appendix B).

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 Samples should be preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection.

6.2 Holding times for furnace analysis are 180 days for both the current EPA SOW 2nd SW846 protocols.

7.0 PROCEDURE

7.1 Start up of the Zeeman 3030

Warning: Not to be operated in proximity to heart patients wearing electronic pacemakers. This instrument contains powerful magnets which may affect pacemakers, watches and magnetic storage media, such as recording tape and computer disks.

A) Prior to startup, the operator should be familiar with the user's manual and reference manual supplied by Perkin-Elmer.

B) Before starting the instrument check the exhaust vent above the furnace to be sure it is working. The switch is on the wall next to the telephone. It should always be on. Also, check to make sure the cooling water is on and draining at a rate of approximately 1.0 liter/minute.

C) Check that the pressure of the argon supply gas is at 50 psi and that there is ample argon in the tank.

D) Check flush water to capillary system. Be sure rinse reservoir is full (0.5 percent HNO_3 solution) and that waste reservoir is empty.

E) Check that there is plenty of paper feeding to the printer and that it is aligned at the top of the page.

7.2 Turn instrument on in this order:

A) Depress power button on the HGA-600 power unit to "on" position.

B) Turn on power to spectrometer and wait until "element select mode" page is presented.

C) Switch on PR-100 printer and make sure the green "on-line" indicator is lit.

D) Turn on EDL power supply by lifting toggle switch up to the "on" position.

1) Select appropriate lamp. Connect lamp to channel cable. Light the lamp by holding ignitor in front of lamp while turning appropriate channel knob fully clockwise (meter should go full scale). Now turn knob counterclockwise to required wattage printed on lamp. Let lamp warm up for approximately 30 minutes, making sure to maintain proper wattage.

2) Press the "user index" key to see a listing of programs for the elements. Select the program for the element you wish to run by entering the method number and pressing blue "recall" key (Appendix E). Depress "prog" key to view the 3 pages of the element program.

3) On page 3, enter the position number of the last cup you want to run by cursoring down to "sample 05 to _ '_" and hitting the enter key (Appendix E).

4) Store the program by depressing blue "store" key.

5) Put the spectrometer into "set-up" mode by pressing blue "set-up" key. Adjust wavelength to proper setting by turning first the coarse (inner), then the fine tuning (outer) adjustment knob on the far left of the instrument. A bar graph on the computer screen indicates proper optimization. If bar goes off scale, hit the "gain" button.

6) Optimize lamp position by turning both adjustment screws (on lamp turret) back and forth until a maximum energy is achieved. (Again, depress "gain" button if bar graph goes off scale.)

7) Clean all windows on the furnace. Take out windows on both sides of the furnace compartment and remove any fog or dirt with Kimwipes. Unscrew the left-hand side of the furnace housing and remove the quartz windows to clean.

   Turn (either way) the small window on the right side of the furnace housing and pull out, cleaning both sides of this window with a cotton swab and Kimwipe.

8) Open furnace by pressing "furnace" button on the HGA-600 power unit. Remove graphite tube and clean inner and outer portions of the contact rings with a cotton swab and a Kimwipe. Tubes and platforms last approximately 16 to 20 hours, therefore check the "daily run and maintenance logbook" (Appendix F) to see if it needs replacing.

9) Insert the L'vov platform into the graphite tube using the following method:

   a) Place platform with cavity downward onto flat surface of insertion tool.

   b) Hold tube horizontally so that the grooves are at the top and the sample introduction hole is on the bottom.

   c) Guide the edges of the platform into the grooves and push into tube with the insertion tool. While inserting the platform, squeeze the handle of the tool so that the edges of the platform are pressed into the grooves in the tube. Shake lightly to ensure that the platform is securely seated.

10) Take the graphite tube, with grooved-end facing left, and place it into the left-hand contact cylinder so that the sample introduction hole in the tube lines up with the sample port in the contact cylinder. Close the furnace by pressing the "furnace" button.

11) Align the sampler arm through the sample introduction hole of the graphite tube using the following method.

   a) Enter "continuous mode" by pressing "cont" hard key.
   b) Depress "as standby" soft key to release sampling arm.
   c) Use the knob on the sampling arm assembly to swing the arm to the furnace and over the sample introduction port.

d) Release lock control by turning the red knob on the autosampler assembly, counterclockwise.

12) If necessary, use the front knob (gray), to adjust the arm backward or forward, and use the side knob to adjust the arm side to side, so that the capillary tip can be lowered into the sample port. Lock the tip into place by turning (red) lock control knob clockwise.

13) Adjust the penetration depth to 2-3 mm above the L'vov platform by turning the front knob on top of the sample arm assembly (knob closest to sampling tray). The tip is at the correct height when the sample droplet contacts the platform just before leaving the end of the capillary tip.

14) Bring the sampling arm back to "run" position by depressing "as home" soft key.

a) Condition the graphite tube as follows:

Depress "run" key to go into run mode. Type in "900" using the key pad on the right side of the computer, and hit the "manual temp" soft key on the computer screen. You should see a faint orange glow after a few seconds, hit "manual temp" again to deactivate. Type 1400 and hit "manual temp". You should see a brighter orange glow. Depress "manual temp" again to deactivate this. Repeat this sequence going next to 1800, 2200, and then 2600°C. Repeat this procedure three times to ensure the tube is fully conditioned.

15) Hit "cont" key then depress "AZ" soft key to zero on instrument absorbance display.

16) Enter program mode, to check user method, by depressing "program" key. Check the number and concentration of standards to be used for analysis (see Appendix E).

17) Hit "program" key again and check the temperature conditions. Confirm there are at least five steps being utilized (see Appendix E).

18) Hit "program" key again and check run sequence. Make sure that there is a sample cup position assigned to the blank (always location 1) and all standards are being analyzed as part of the initial calibration. Also check that a "matrix modifier cup" position is assigned. Be

sure that sample location range is entered and that the matrix modifier ID is entered (i.e. one or two) (see Appendix E).

19) Enter "run mode" by depressing "run" key.

20) Load the tray always starting with a reagent blank in cup #1 and appropriate standards in ascending concentration in next cups (see Appendix E).

21) Enter ID number for first cup after calibration and hit "print" key (light should come on).

22) Be sure autosampler is at position "0" on computer screen by depressing "autosampler reset" soft key, then depress "check" soft key and display will change to "sampler on/off". Depress "sampler on/off" soft key. Analysis is now initiated.

7.3 Sample Run Log (Appendix G)

A) Analysis starts with initial calibration as follows, with double injections of all standards and samples the result being the average of the two burns.

1) Cups 1-4 are for reagent blank and three standards.

2) Cup 5 ICV (initial calibration verification)

3) Cup 6 ICB (initial calibration blank)

4) Cup 7 CRA - this standard is equivalent to the CRDL for the element being analyzed.

B) Depending on protocol, a continuing calibration verification (CCV) and continuing calibration blank (CCB) are run immediately after the CRA, or nine samples can be run, followed by another CCV and CCB.

C) Each sample must be immediately followed by its analytical spike and cannot be separated by QC. An analytical spike is not required on the actual pre-digest spike sample.

D) Monitor the run frequently during the analysis. Check the precision to assure that the replicates are within acceptable limits (RSD ≤20 percent) for samples greater than the CRDL. Check that initial and continuing QC passes and analytical spike recoveries are acceptable. If any of these fail, it may be an indication that a problem exists. Stop the run,

identify the problem and make the appropriate corrective action before proceeding.

7.4 System Shut Down

A) Turn EDL channel knob fully counterclockwise
B) Turn off EDL power supply
C) Turn off power to 3030 spectrometer
D) Turn off power to HGA-600
E) Turn off printer

7.5 The 3030 has a bound maintenance logbook that is filled out every time an analysis is to be performed. The book is kept underneath the furnace on top of the HGA-600 power unit. The first column is for the date of analysis. The second column is for the analyst's initials. The next column is for recording maintenance performed, such as changing the graphite tube and platform, contact rings, or sampling capillary. The next column is for how many hours the instrument was operating and the last column is for the element and the job/case number that was analyzed (Appendix F).

A) A separate folder in the main filing cabinet is maintained for repair work which required a service call from a certified Perkin-Elmer technician. There is a folder labeled "3030" with the completed service tags, describing problem, along with the corrective action.

B) A furnace auto ample logbook is also kept under the 5100 PC desk. Every time analysis is performed - a copy of the furnace data sheet goes in the book, the elements that were analyzed for are written in the "element" space on the upper left hand corner (Appendix G).

8.0 QUALITY CONTROL

When the run is complete, check that all quality control (ICV's, prep blanks and laboratory control samples) meets acceptable criteria. If contamination is evident in prep blanks or LCS's fail to recover, refer to protocol to determine corrective action. If necessary submit a "corrective action report" with a copy of the data and prep sheet attached explaining the problem, to the supervisor for immediate action (Appendix H).

9.0 METHOD PERFORMANCE

9.1 Instrument detection limits are determined quarterly per the current EPA contract as stated in Exhibit E, Section II, page E-14.

9.2 Control charts are kept for tracking of accuracy (LCS and spike recoveries), and precision (RPD on the duplicate analysis).

10.0 DATA REDUCTION AND DATA HANDLING

If data meets all criteria, calculate results and keep track of any reruns or dilutions needed. Once a job has been fully completed, sign and date all relevant chain of custody sheets. At the end of each completed analysis, report all sensitivities for analyses that were run, in the appropriate logbook for standards (Appendix I). For non-CLP work, report results and quality control on final report sheets. CLP work should go into the appropriate job folder.

11.0 SPECIAL PROCEDURES - N/A

12.0 USE OF OTHER REQUIRED SOP'S

Glassware cleaning SOP, standards and sample preparation SOP, standards and sample prep log SOP and corrective action SOP.

13.0 DOCUMENTATION OF PROBLEMS

13.1 Corrective Action Reports

If any discrepancy occurs during analysis that requires attention such as standard or instrument problem, sample prep problem, etc., a corrective action report is warranted. This report should be prepared per the atomic spectroscopy corrective action report SOP (AS:040590:0).

13.2 All CLP-type reports and regular reports require case narratvies that specifically address any anomolies and their possible causes that may have occurred during analysis.

14.0 REFERENCES

14.1 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December, 1982.

14.2 USEPA Contract Laboratory Program, SOW 7/88

14.3 Test Methods for Evaluating Solid Waste, Volume 1A, USEPA SW846, 3rd Edition, November, 1986.

APPENDIX A

Stock Standard Solution

Pipette 1.0 ml each of 1,000 mg/L (ppm) of arsenic, selenium, thallium, and lead standards into a 200 ml volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 5,000 ppb of each element.

CRA Stock Standard Solution

Pippette 1.0 ml each of 1,000 mg/L (ppm) arsenic and thallium, 0.5 ml of 1,000 mg/L (ppm) selenium and 0.3 ml of 1,000 mg/L (ppm) lead into a 200 ml volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 5,000 ppb arsenic and thallium, 2,500 ppb/selenium and 1,500 ppb lead.

Analytical Spike Solution

Pipette 9.4 ml each 1,000 mg/L (ppm) of arsenic, thallium and lead and 0.2 ml of 1,000 mg/L (ppm) selenium into a 200 ml volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 2,000 ppb arsenic, thallium and lead, 1,000 ppb selenium.

In-house Continuting Calibration Verification Solution

Pipette 1.0 ml each 1,000 mg/L (ppm) arsenic, selenium, thallium and lead into a 200 ml volumentric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 5,000 ppb for all elements.

STOCK STANDARD SOLUTIONS

Solution	Reference	Elements	Stock ID	mls taken	Dilution volume	Acid	Date	Ck
CCV	SQL	As, Se, Tl, Pb	ICP-P-50 [illegible]	1.0	200	HNO_3 5%	[illegible]	[illegible]
5000 ppb	~~As~~ 10	As	ICP-P-0070	1.0	200	HNO_3 5%	[illegible]	[illegible]
5000 ppb	~~Tl~~ 10	Tl	ICP-P-0041	1.0				
5000 ppb	~~Se~~ 10	Se	ICP-P-0057	1.0				
5000 ppb	~~Pb~~ 10	Pb	ICP-P-0066	1.0				
CCV	~~As, Se, Tl, Pb~~ SQL	As, Se, Tl, Pb	ICP-P-0070, 41, 57, 66	1.0			[illegible]	
5000 ppb	~~As, Tl~~ 11	As, Tl	ICP-P-0070, 41	1.0			[illegible]	
2500 ppb	~~Se~~ 11	Se	ICP-P-57	0.5				
1500 ppb	~~Pb~~ 11	Pb	ICP-P-0066	0.3	✓	✓	✓	✓
5000 ppb	10	As	ICP-P-0070	1.0	200	HNO_3 2%	[illegible]	[illegible]
5000 ppb	10	Tl	ICP-P-0041	1.0				
2500 ppb	10	Se	ICP-P-0057	1.0				
1500 ppb	10	Pb	ICP-P-0066	1.0				
CCV	SQL	As, Se, Tl, Pb	ICP-P-0070, 41, 57, 66	1.0			[illegible]	
5000 ppb	11	As, Tl	ICP-P-0070, 41	1.0			[illegible]	
2500 ppb	11	Se	ICP-P-57	0.5				
1500 ppb	11	Pb	ICP-P-66	0.3	✓	✓	✓	✓
CCV	SQL	As, Se, Tl, Pb	ICP-P-0070, 41, 57, 66	1.0	200	HNO_3 2%	[illegible]	[illegible]
5000 ppb	10	As	ICP-P-0064	1.0	200	HNO_3 2%	[illegible]	[illegible]
5000 ppb	10	Tl	ICP-P-0065	1.0				
5000 ppb	10	Se	ICP-P-0063	1.0				
5000 ppb	10	Pb	ICP-P-0066	1.0				
CCV	SQL	As, Se, Tl, Pb	ICP-P-64, 65, 63, 66	1.0			[illegible]	
5000 ppb	11	As, Tl	ICP-P-64, 65	1.0			[illegible]	
2500 ppb	11	Se	ICP-P-63	0.5				
1500 ppb	11	Pb	ICP-P-66	0.3	✓	✓	✓	✓
CCV	SQL	As, Se, Tl, Pb	ICP-P-64, 65, 63, 66	1	200	HNO_3 2%	[illegible]	[illegible]
[illegible]	10	As	ICP-P-[illegible]	1			[illegible]	
[illegible]	10	Se	ICP-P-[illegible]	1				
[illegible]	10	Tl	ICP-P-[illegible]	1				
[illegible]	10	Pb	ICP-P-[illegible]	1				
[illegible]	11	As, Tl	ICP-P-[illegible]	1			[illegible]	
[illegible]	11	Se	ICP-P-[illegible]	0.5				
[illegible]	11	Pb	ICP-P-[illegible]	0.3	✓	✓	✓	✓

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APPENDIX B

Working Standard Solution

100 ppb standard - Pipette 4.0 ml of the stock standard solution into a 200 ml volumetric flask and bring to volume with 0.5 percent nitric acid.

50 ppb Standard - Pipette 2.0 ml of the stock standard solution into a 200 ml volumetric flask and bring to volume with 0.5 percent nitric acid.

Intermediate CRA Solution - Pipette 4.0 ml of the stock CRA solution into a 200 ml volumetric flask and bring to volume with 0.5 percent nitric acid.

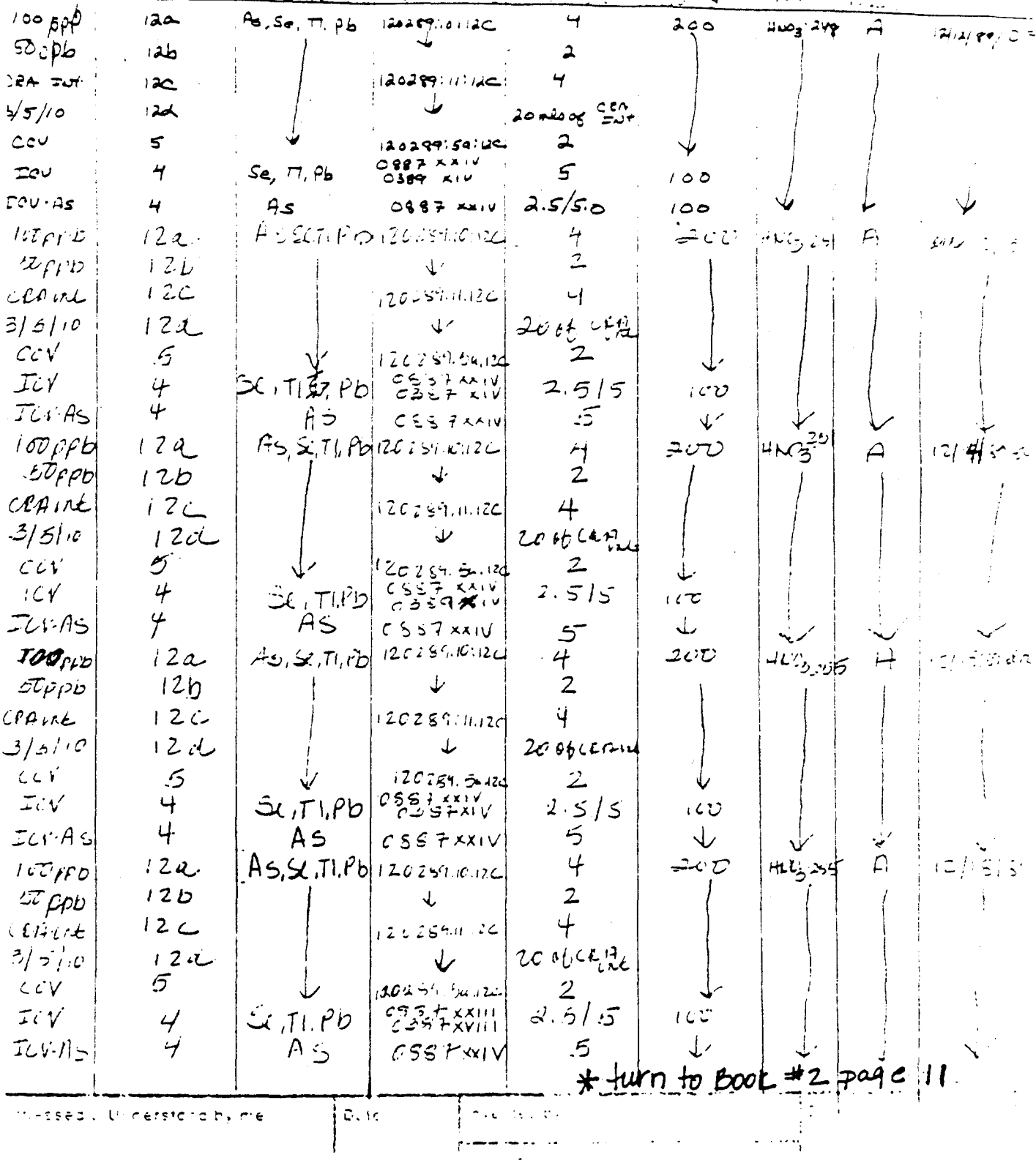
CRA Solution: 10.0 ppb As, Tl
5.0 ppb Se
3.0 ppb Pb

Pipette 20.0 ml of the intermediate CRA solution into a 200 ml volumetric flask and bring to volume with 0.5 percent nitric acid.

Blank Solution - Add 0.5 percent nitric acid to 100 ml nano pure water.

In-house Continuing Calibration Verification Solution (CCV) - Pipette 2.0 ml of the stock CCV solution into a 200 ml volumetric flask and bring to volume with 0.5 percent nitric acid. This solution contains 50.0 ppb all elements.

Working Standard Solutions



* turn to Book #2 page 11

APPENDIX C

TABLE XI
MULTI-RESPONSE COMPOUNDS

All PCB's and Toxaphene: Working Standards

Conc. Stock	ul Used	ul DBC* Stock Added	Final Volume	Final Conc.
5,000 ug/ml	2	10 ul	10 ml	1.0
5,000 ug/ml	4	10 ul	10 ml	2.0
5,000 ug/ml	10	10 ul	10 ml	5.0
5,000 ug/ml	20	10 ul	10 ml	10.0

Chlordane:

Conc. Stock	ul Used	Final Volume	Final Conc.
5,000 ug/ml	40	2 ml	100 ug/ml

Working Standards: Chlordane

Conc. Stock	ul Used	ul DBC* Stock Added	Final Volume	Final Conc.
100	10.0	10 ul	10 ml	0.1
100	20.0	10 ul	10 ml	0.2
100	50.0	10 ul	10 ml	0.5
100	100.0	10 ul	10 ml	1.0

*DBC stock at 100 ug/ml.

TABLE XII
PESTICIDE EVALUATION MIX STANDARDS

Stocks:

Compound	Stock Concentration	ul Used	Final Volume	Concentration (ug/ml)
Aldrin	5,000 ug/ml	20.0	10 ml	10.0
Endrin	5,000 ug/ml	40.0	10 ml	20.0
4,4'-DDT	5,000 ug/ml	80.0	10 ml	40.0
DBC	5,000 ug/ml	200.0	10 ml	100.0

Mixes:

MIX A

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
Aldrin	10 ug/ml		10 ml	0.025 ug/ml
Endrin	20 ug/ml	25 ul	10 ml	0.05 ug/ml
4,4'-DDT	40 ug/ml		10 ml	0.125 ug/ml
DBC	100 ug/ml	10 ul	10 ml	0.05 ug/ml

MIX B

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
Aldrin	10 ug/ml		25 ml	0.05 ug/ml
Endrin	20 ug/ml	125 ul	25 ml	0.10 ug/ml
4,4'-DDT	40 ug/ml		25 ml	0.25 ug/ml
DBC	100 ug/ml	25 ul	25 ml	0.10 ug/ml

MIX C

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
Aldrin	10 ug/ml		10 ml	0.10 ug/ml
Endrin	20 ug/ml	100 ul	10 ml	0.20 ug/ml
4,4'-DDT	40 ug/ml		10 ml	0.50 ug/ml
DBC	100 ug/ml	10 ul	10 ml	0.20 ug/ml

TABLE XIII
DDT SERIES MIX STANDARDS

Stock Standard:

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
4,4'-DDT	5,000 ug/ml	40.0	10 ml	20.0
4,4'-DDE	5,000 ug/ml	20.0	10 ml	10.0
4,4'-DDD	5,000 ug/ml	20.0	10 ml	10.0

Mix Standards:

MIX 1

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
4,4'-DDT	20 ug/ml	50 ul	10 ml	0.10 ug/ml
4,4'-DDD	10 ug/ml	50 ul	10 ml	0.05 ug/ml
4,4'-DDE	10 ug/ml	50 ul	10 ml	0.05 ug/ml
DBC	100 ug/ml	10 ul	10 ml	0.10 ug/ml

MIX 2

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
4,4'-DDT	20 ug/ml	100 ul	10 ml	0.20 ug/ml
4,4'-DDD	10 ug/ml	100 ul	10 ml	0.10 ug/ml
4,4'-DDE	10 ug/ml	100 ul	10 ml	0.10 ug/ml
DBC	100 ug/ml	10 ul	10 ml	0.10 ug/ml

MIX 3

Compound	Stock Concentration	ul Used	Final Volume	Final Concentration
4,4'-DDT	20 ug/ml	250 ul	10 ml	0.50 ug/ml
4,4'-DDD	10 ug/ml	250 ul	10 ml	0.25 ug/ml
4,4'-DDE	10 ug/ml	250 ul	10 ml	0.25 ug/ml
DBC	100 ug/ml	10 ul	10 ml	0.10 ug/ml

TABLE XIV

Compound	Standard Range (ug/ml)
Mirex	0.025 - 0.50
Dichloran	0.025 - 0.50
Isodrin	0.025 - 0.50
Kepone	0.025 - 0.50
Chlorobenzilate	0.05 - 0.60
Alachlor	0.10 - 2.0
Atrazine	2.0 - 32.0
Dicofor	0.01 - 0.18

IEA

RUNID :
ANALYSIS :
GC :

COL ID
INJ C

PHASE
OVEN C
DETECTOR TYPE
RUN TIME

FLOW
PROGRAM
C
ul INJ

FILE ID	VIAL POS.	CLIENT	LAB ID	INJ DATE	INJ TIME	SURR RT	SURR AREA	EX ID	ANAL	REPORT DATA PLT	AR RPT	MET RPT	RE-INT	COMMENTS

APPROVED BY

DATE /

FIGURE 1

GAS CHROMATOGRAPHY
QC BATCH APPROVAL FORM

Run ID _____

Analysis	Y/N	Initial/ Date	Comments
AR1221/AR1232 Required			
Do Extracts Require c/u or Dilution			
Analytical Sequence Checked			
First Injection OK			
Chain of Custodies Signed			
QC Checked and Reported in Inj. Log			
Chromatography Checked:			
Solvent Peak			
Baselines			
Surrogates			
Dilutions			
Matrix Problems			
MS Compounds OK			
Forms Reviewed			
Form 8:			
Dates/Times			
Breakdown			
% RSD in Criteria			
Form 8E:			
DBC Shift Criteria			
Dates/Times			
YWC Sample # Check to Inj. Log			
Form 9:			
All Compounds Present			
All Cf/RT Present			
Dates/Times of Standards Correct			
Windows (spot check)			
Correct SDG on All Forms			
Check Case # on Forms (to 8E)			
Sample Analysis			
Primary - Dilutions OK			
Target Compounds in RT Windows			
Confirmation List Written			
Confirmation Compounds of Interest in Windows			
Signed Out in Red Book			

Approved By: _____

FIGURE 3

QA 111088
Rev. 08/18/8

Reported By: _____
Group

Date: _____ Time: _____

CORRECTIVE ACTION REPORT/CASE NARRATIVE REPOR

Client: _____ Job/Case Number: _____ Sample Number(s): _____

Problem Reported By: _____ Deadline To Complete CA: _____

Person Who Is Requested To Provide Corrective Actions: _____

Category of Problem: _____
Poor Communication With ___. Sample Matrix; Instrument

Description of Problem: _____

Use back of CAR/CNR or attached sheet if more space is required

Recommended Corrective Action(s): _____

Actual Corrective Action(s) Taken: _____
Document Actual Corrective Actions On This

Form and Send a Copy of Completed CAR/CNR to QA Officer and Job Folder

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Help in solving problem is requested of: _____

Distribution: (circle) Lab Director, Lab manager, Marketing Manager, Branch Manager
Group/Section Leader, QA Officer: Organic[ ], Inorganic[ ]
Client Services, Job Folder, Other: _____

Document Problem and Corrective Action(s) In Case Narrative: [ ]
Attachments: Telephone Log, Services Change Request, Memo, Other:

301053

FIGURE 4

GC SAMPLE CONFIRMATION

INITIALS	
DATE OF ANALYSIS	
GC RUN ID	

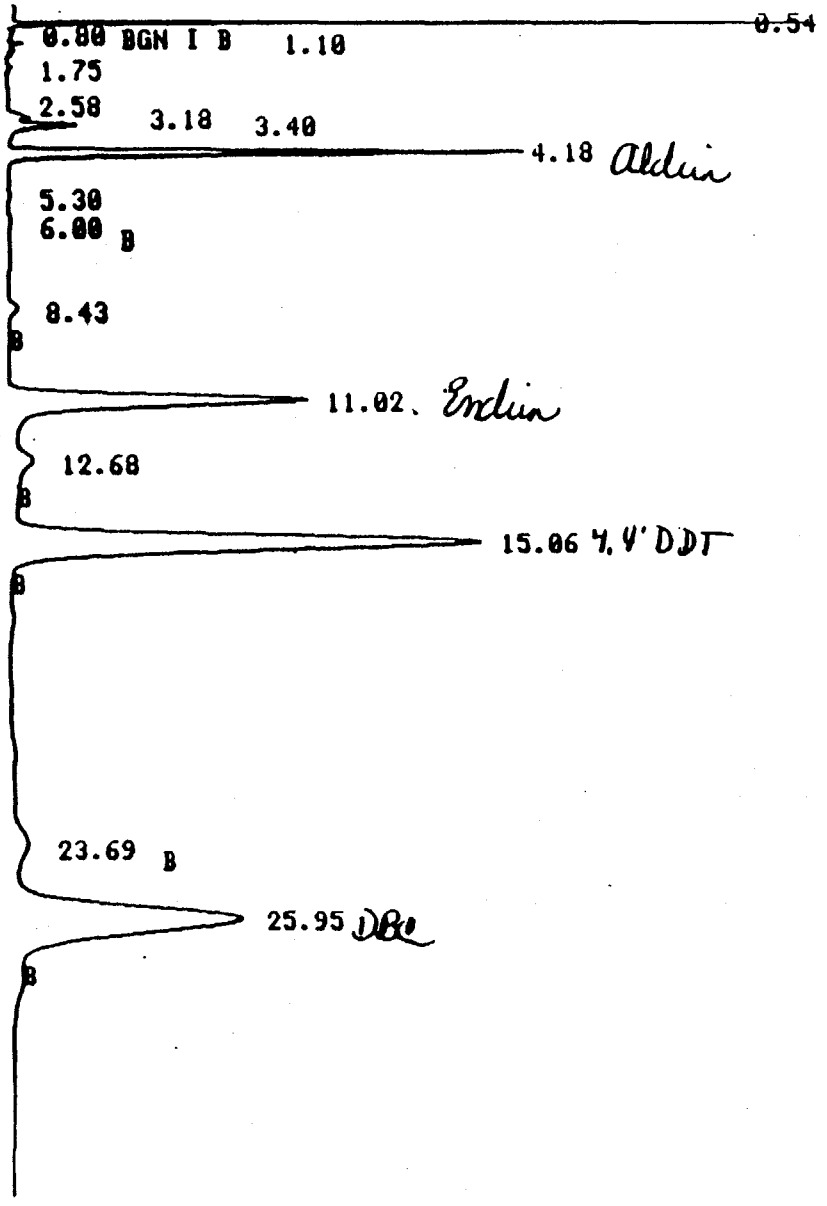
CLIENT	IEA SAMPLE	DILUTION	COMPOUND TO CONFIRM	COMMENTS

GC:1128-01

C...MAT...APH .. ..ETL on .., 28 :51 19(
FILE pest3.002a METHOD 106 Primary Pest

FILE 469 RUN 2 STARTED 14:54.5 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



Pesticide Evaluation Mix A
Col 1.5% SP 2250/1.95% SP2401
4 µl inj

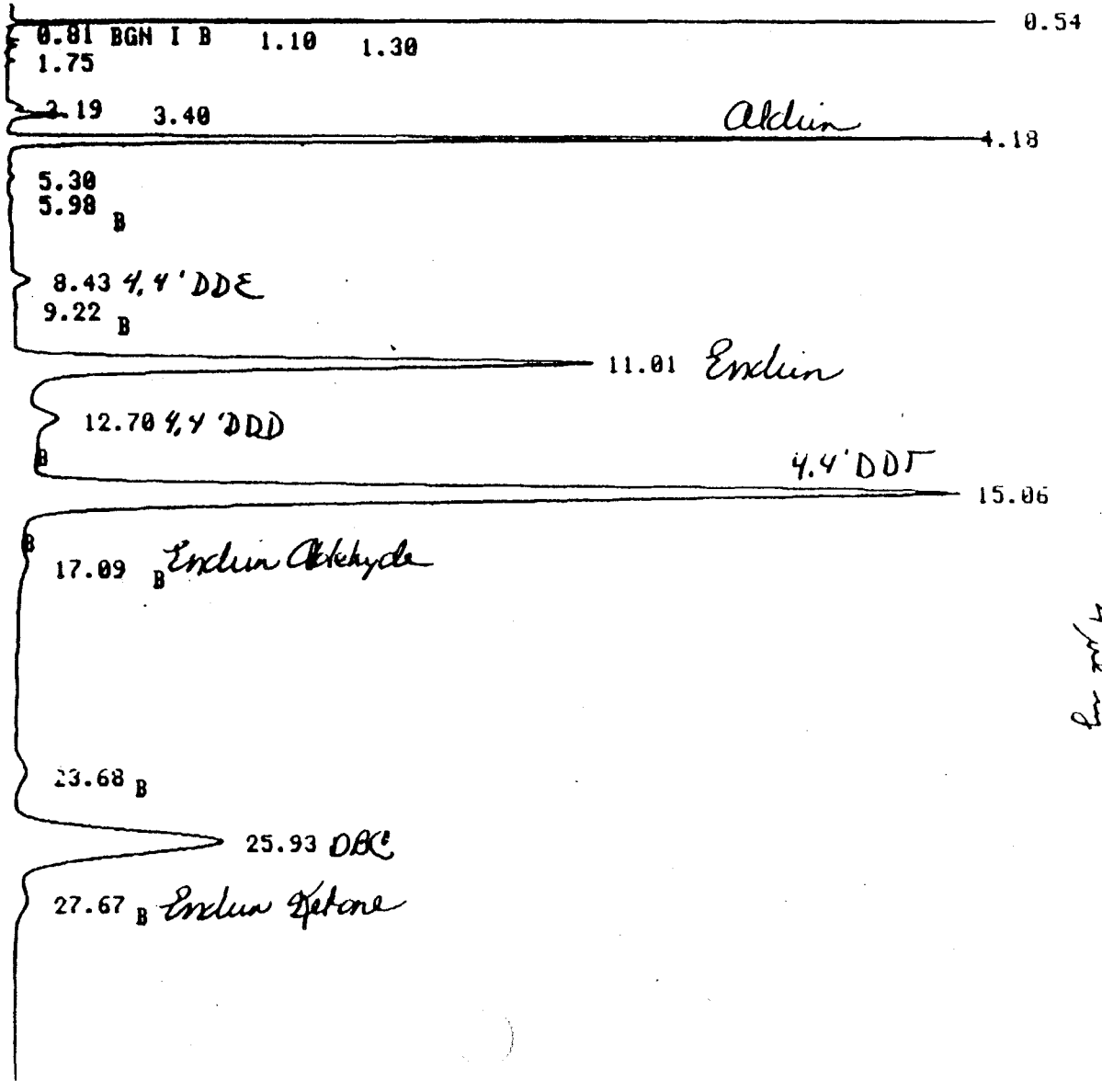
FIGURE 3

301056

CHROMATOGRAPH #2 SETUP Mon Sep 28 15:28:54 1987
FILE pest3.003a METHOD 106 Primary Pest

FILE 470 RUN 3 STARTED 15:31.7 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5

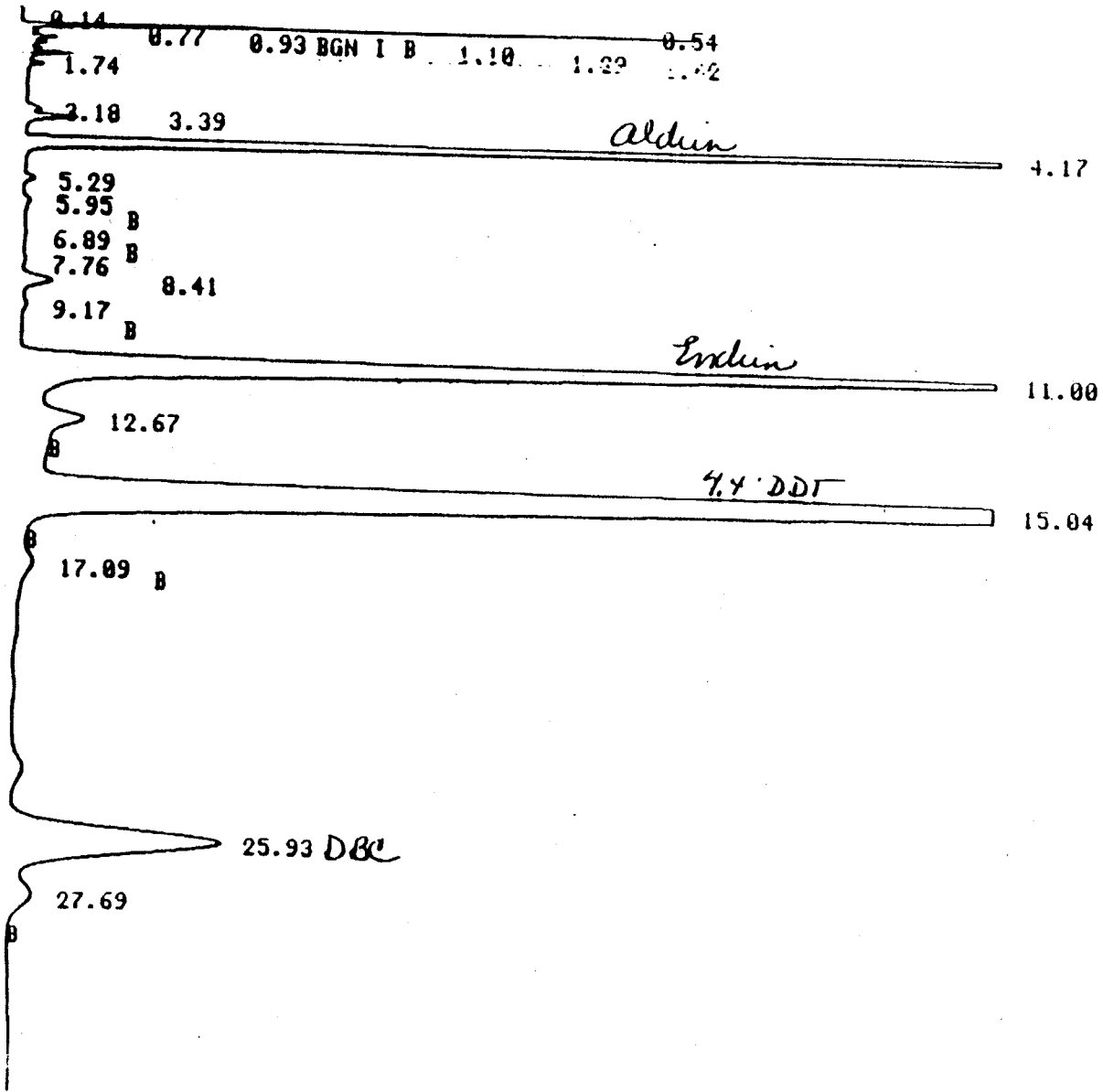


PESTICIDE EVALUATION MIX B
Cat 1,570 SP2250/1.95% SP2401
4 µl inj.

... DMA ...RAPI. ... SET... Mon Sep 28 16:0...7 19...
FILE pest3.004a METHOD 106 Primary Pest

FILE 471 RUN 4 STARTED 16:09.0 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



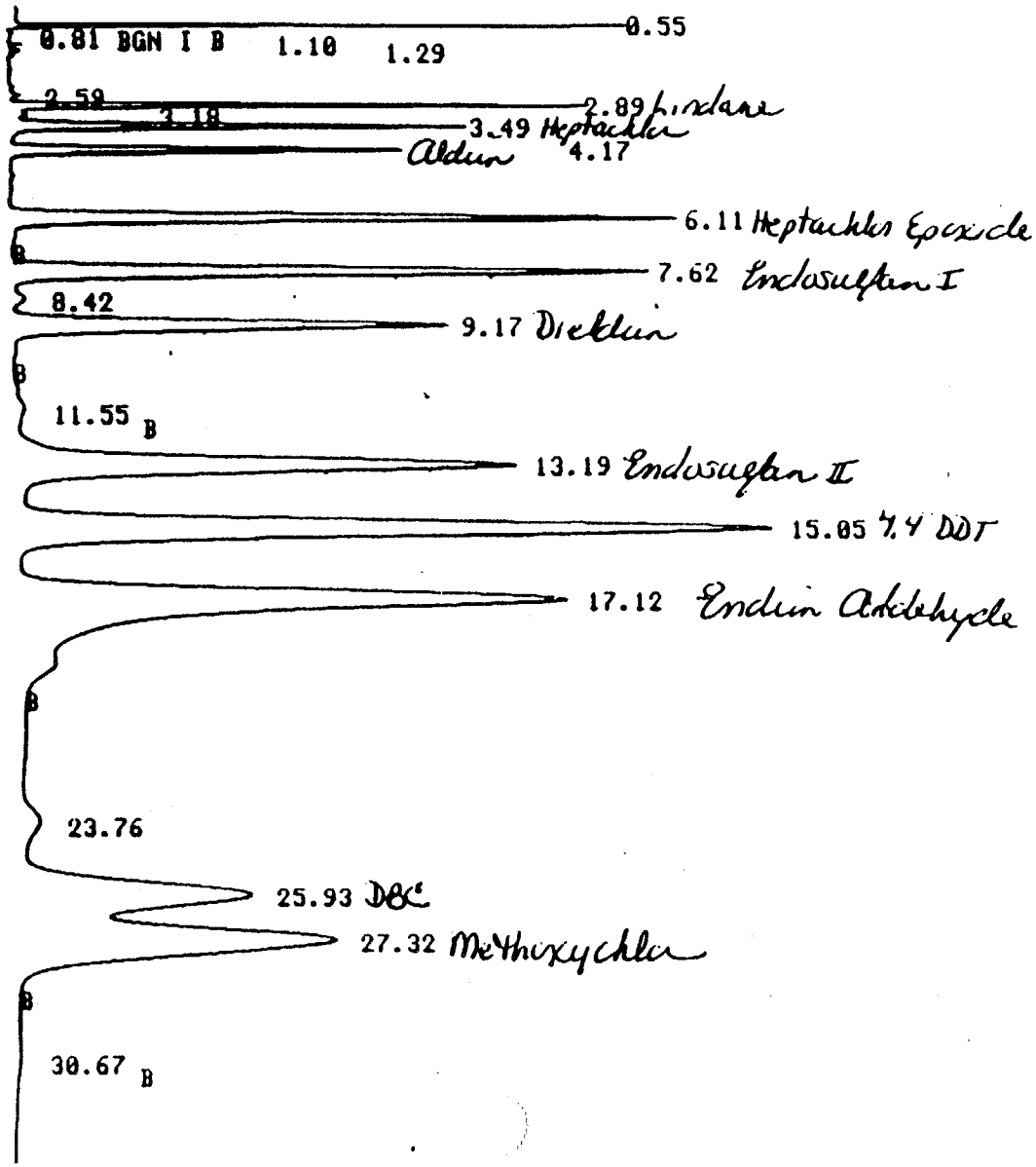
PESTICIDE EVALUATION MIX C
Col 15%SP2250/15%SP2401
4 ppm inj

301058

CHROMATOGRAM #2 SETUP Mon Sep 28 16:43:27 1987
FILE pest3.005a METHOD 106 Primary Pest

FILE 472 RUN 5 STARTED 16:46.2 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



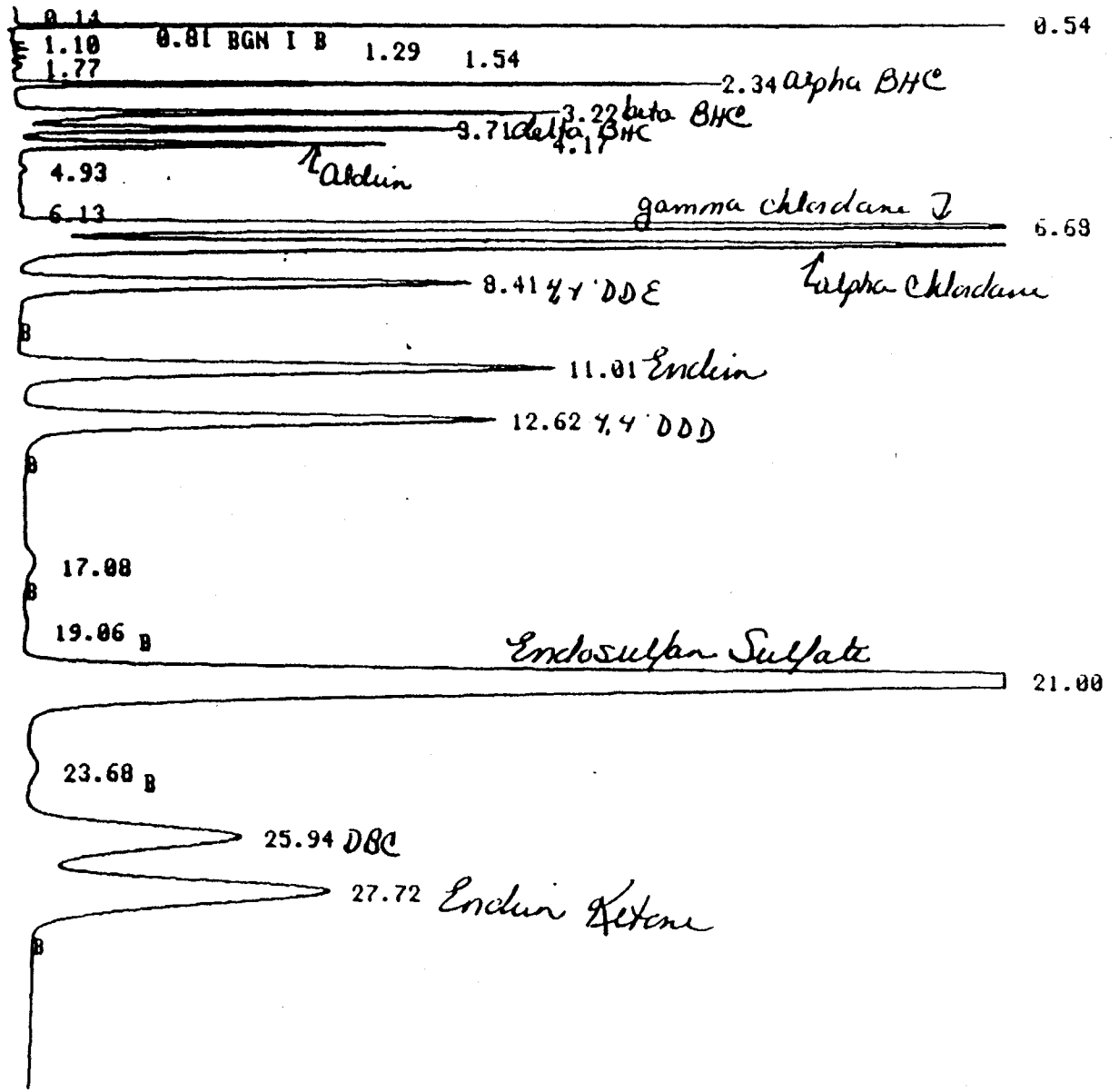
PESTICIDE INDIVIDUAL Mix A
Ca 1.5-50 225/1956 sp2401
4 µL inj

FIGURE 3

CHROMATOGRAPH #2 SETUP Mon Sep 28 17:20:38 1987
FILE pest3.006a METHOD 106 Primary Pest

FILE 473 RUN 6 STARTED 17:23.5 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



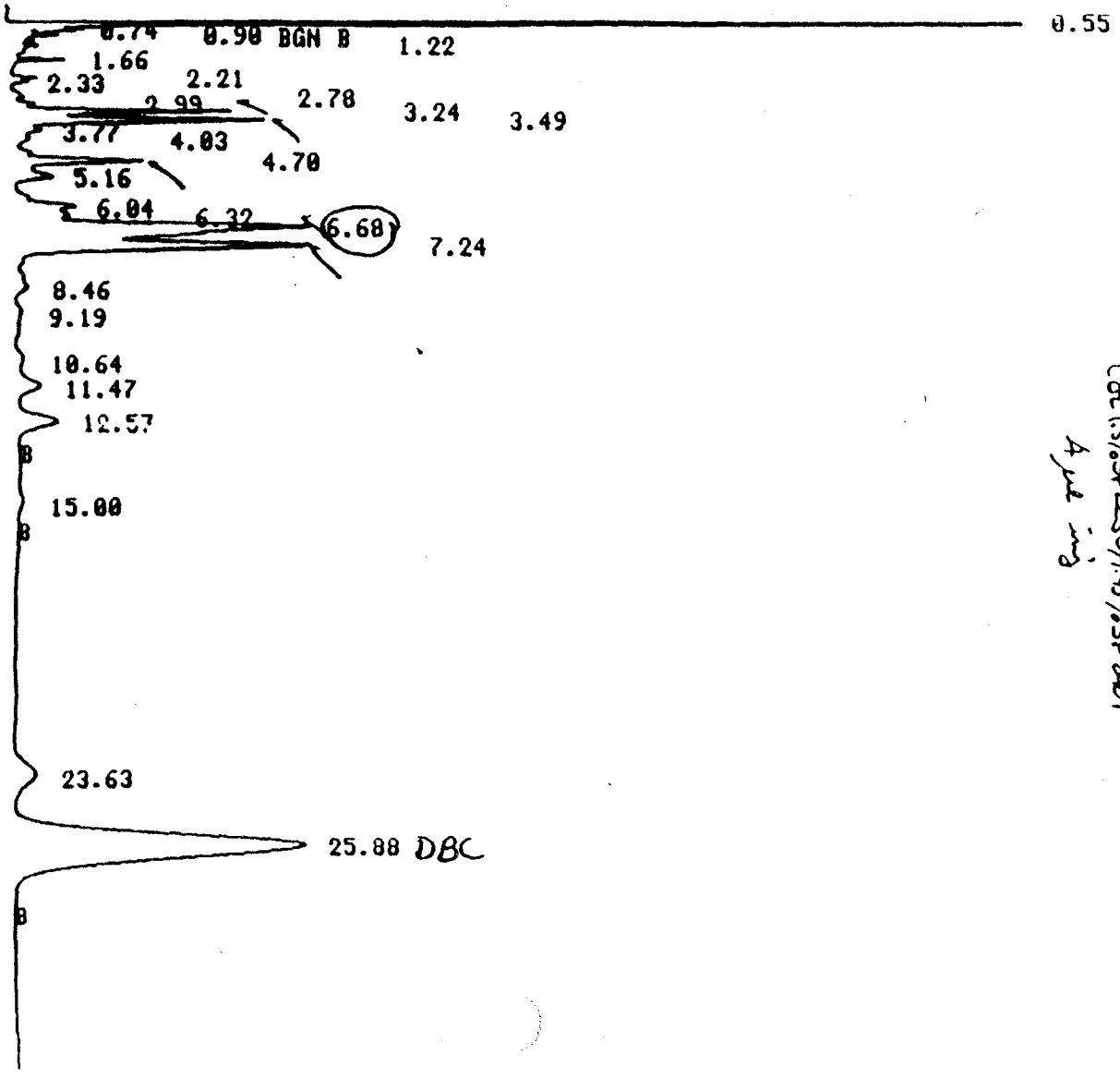
PESTICIDE INDIVIDUAL MIX B
Cat. # 5915220/4570SP2401
4 mo. std.

30059

CHROMATOGRAPH #2 SETUP Mon Sep 28 17:57:48 1987
FILE pest3.007a METHOD 106 Primary Pest

FILE 474 RUN 7 STARTED 18:00.7 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 0_5

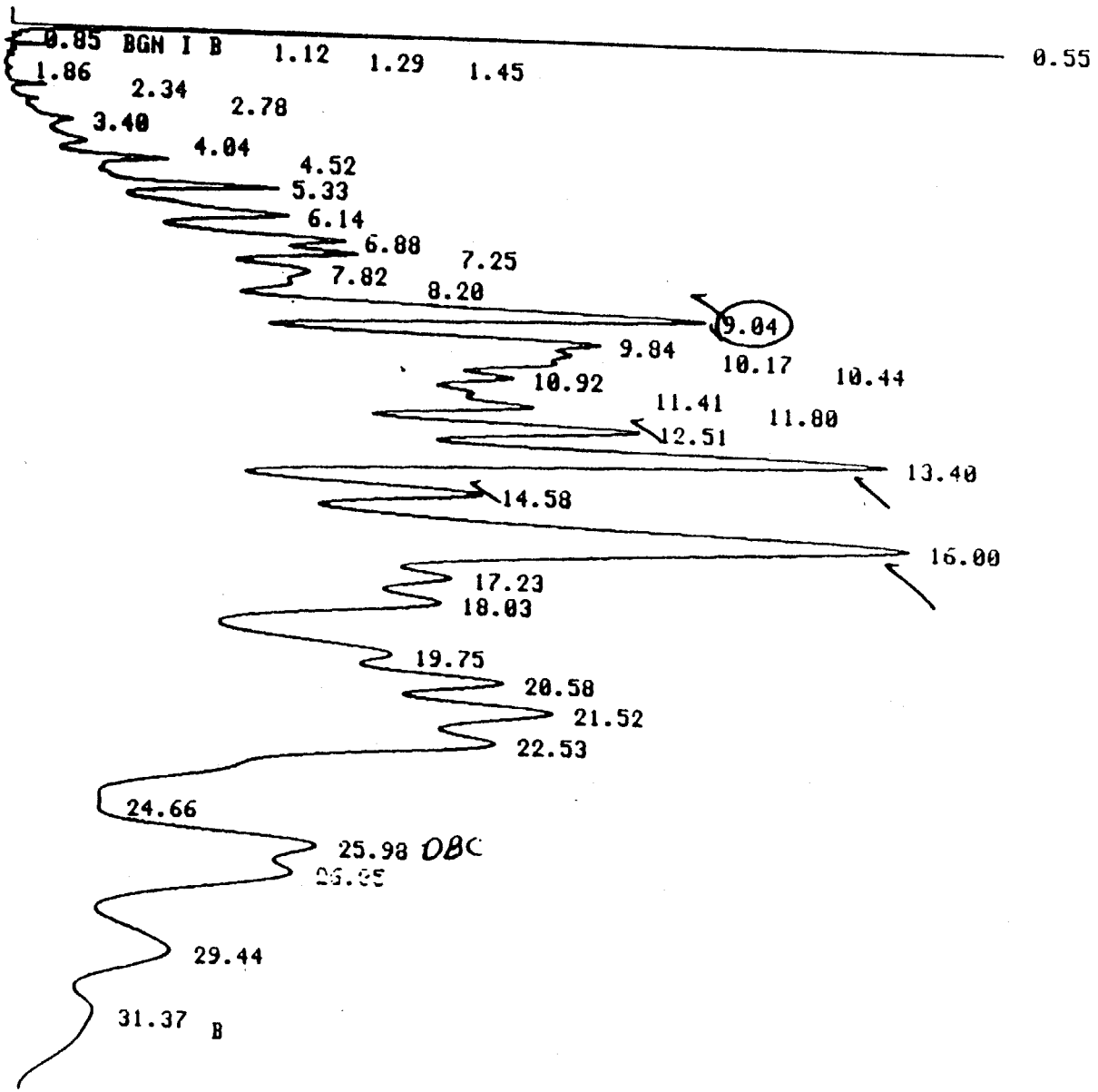


Chlordane .8 ng
Col 1 SP2250/1.5% SP2401
4 µl inj

CHROMATOGRAPH #2 SETUP Mon Sep 28 18:35:00 1987
FILE pest3.008a METHOD 106 Primary Pest

FILE 475 RUN 8 STARTED 18:38.0 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



TOXAPHENE 20.0 ng
(a mixture of 22 congeners)
A mix

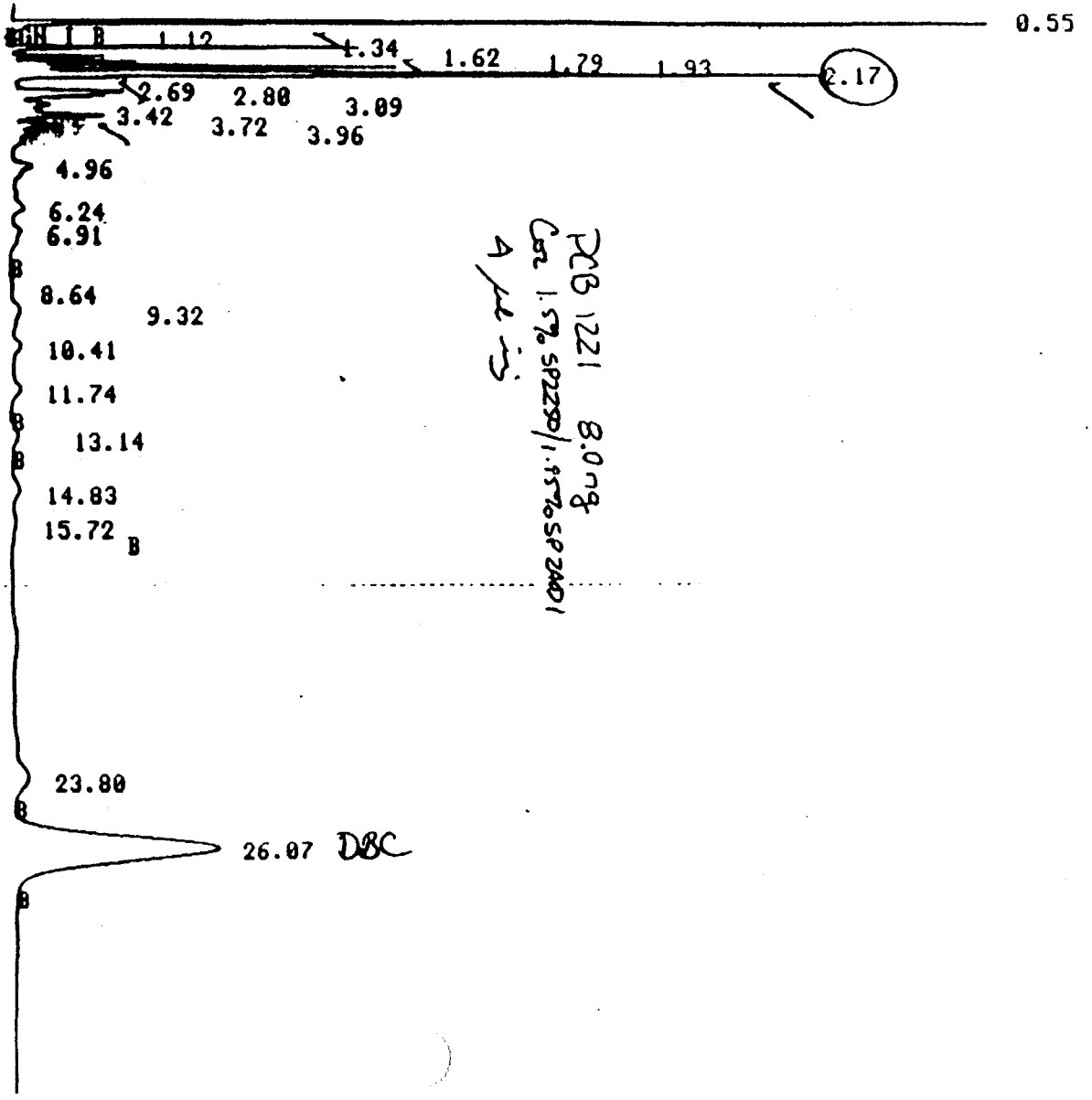
3010611

301062

FILE pest.009a METHOD 106 Primary Pest

FILE 311 RUN 43 STARTED 15:42.7 87/09/14 PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

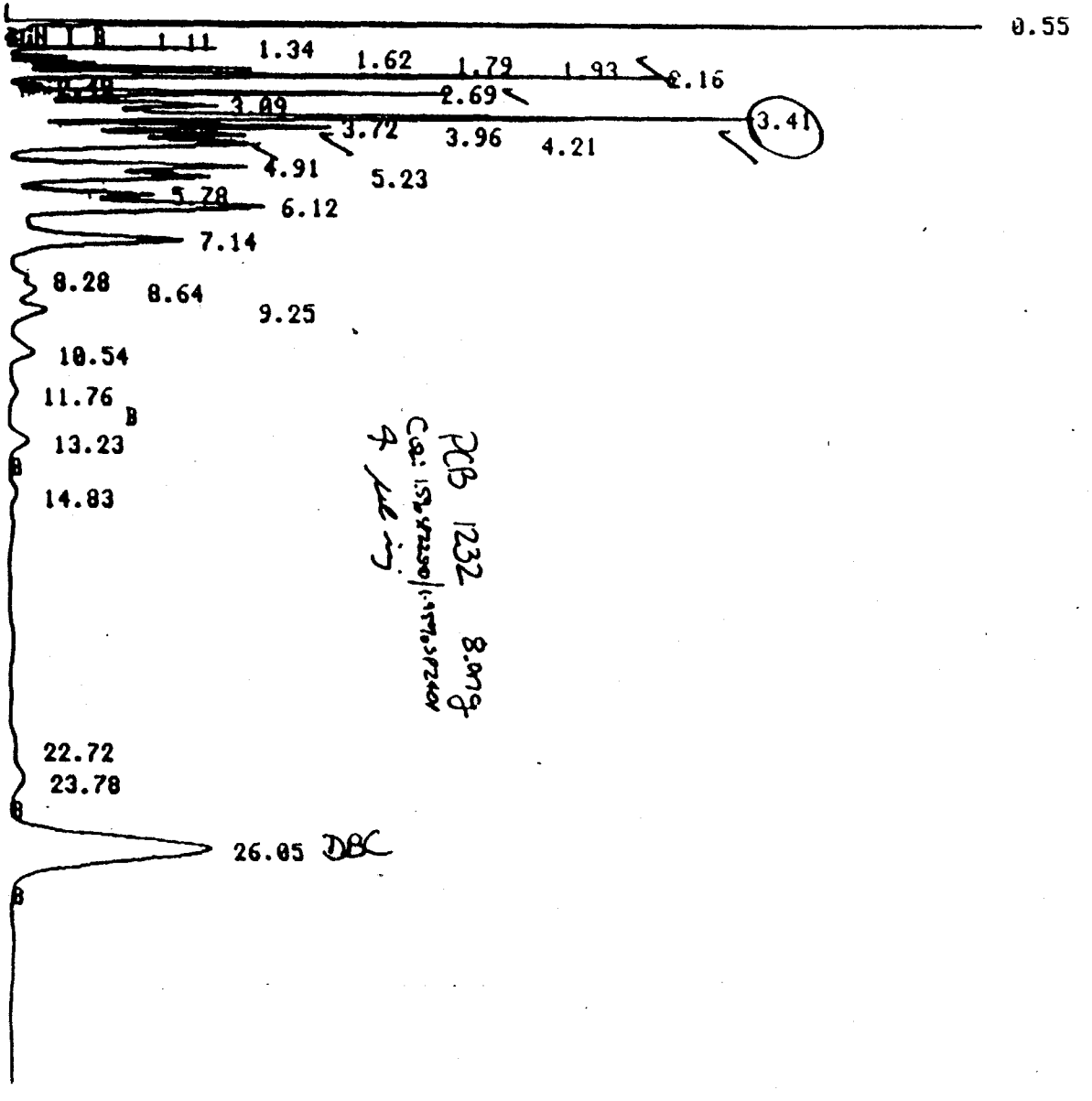
W_-6 A_4 C_5 O_5



CHROMATOGRAPH #2 SETUP Mon Sep 14 16:13:08 1987
FILE pest.010a METHOD 106 Primary Pest

FILE 312 RUN 44 STARTED 16:21.0 87/09/14 PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5

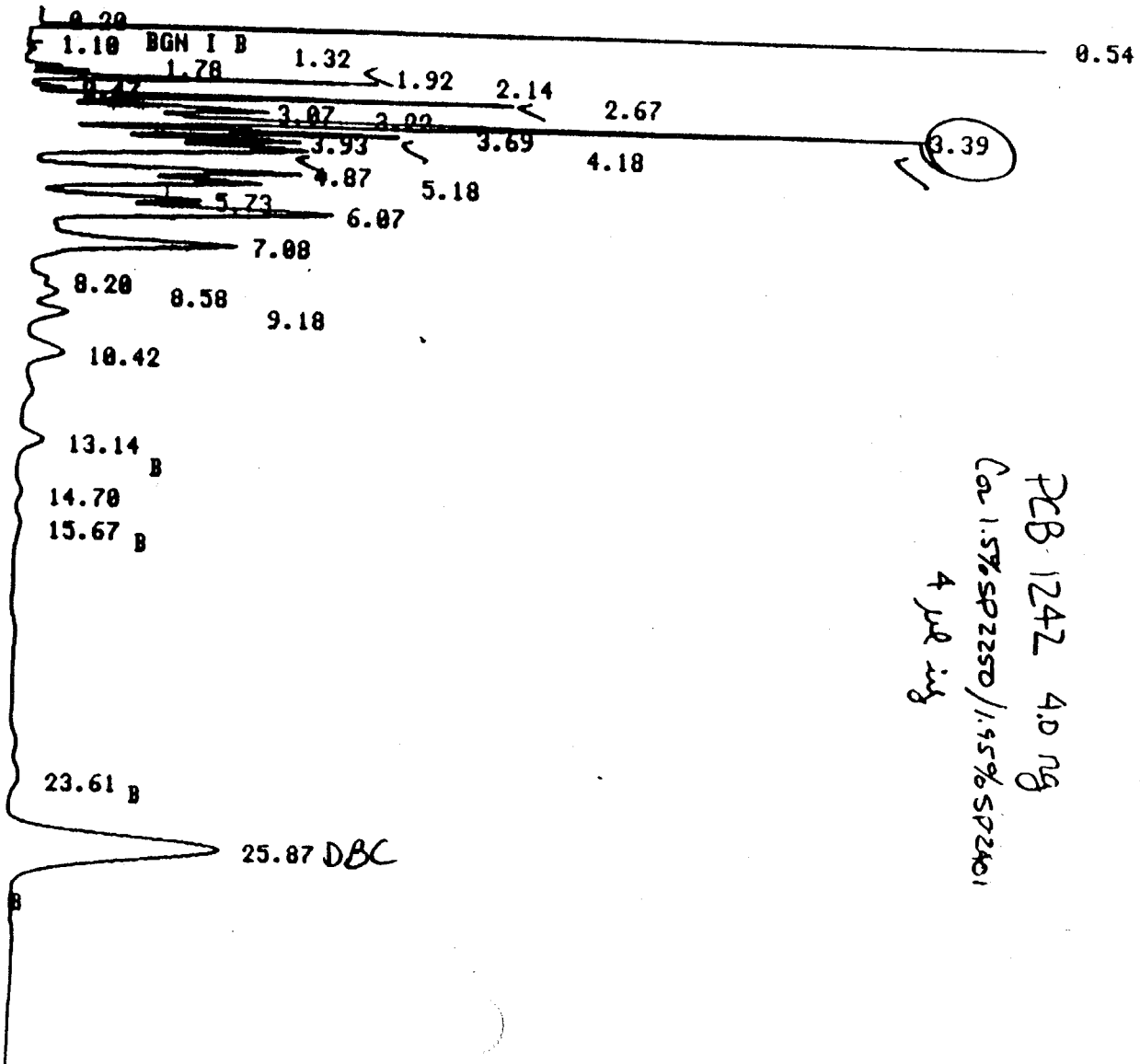


301064

CHROMATOGRAPH #2 SETUP Mon Sep 28 19:12:40 1987
FILE pest3.009a METHOD 106 Primary Pest

FILE 476 RUN 9 STARTED 19:15.2 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5

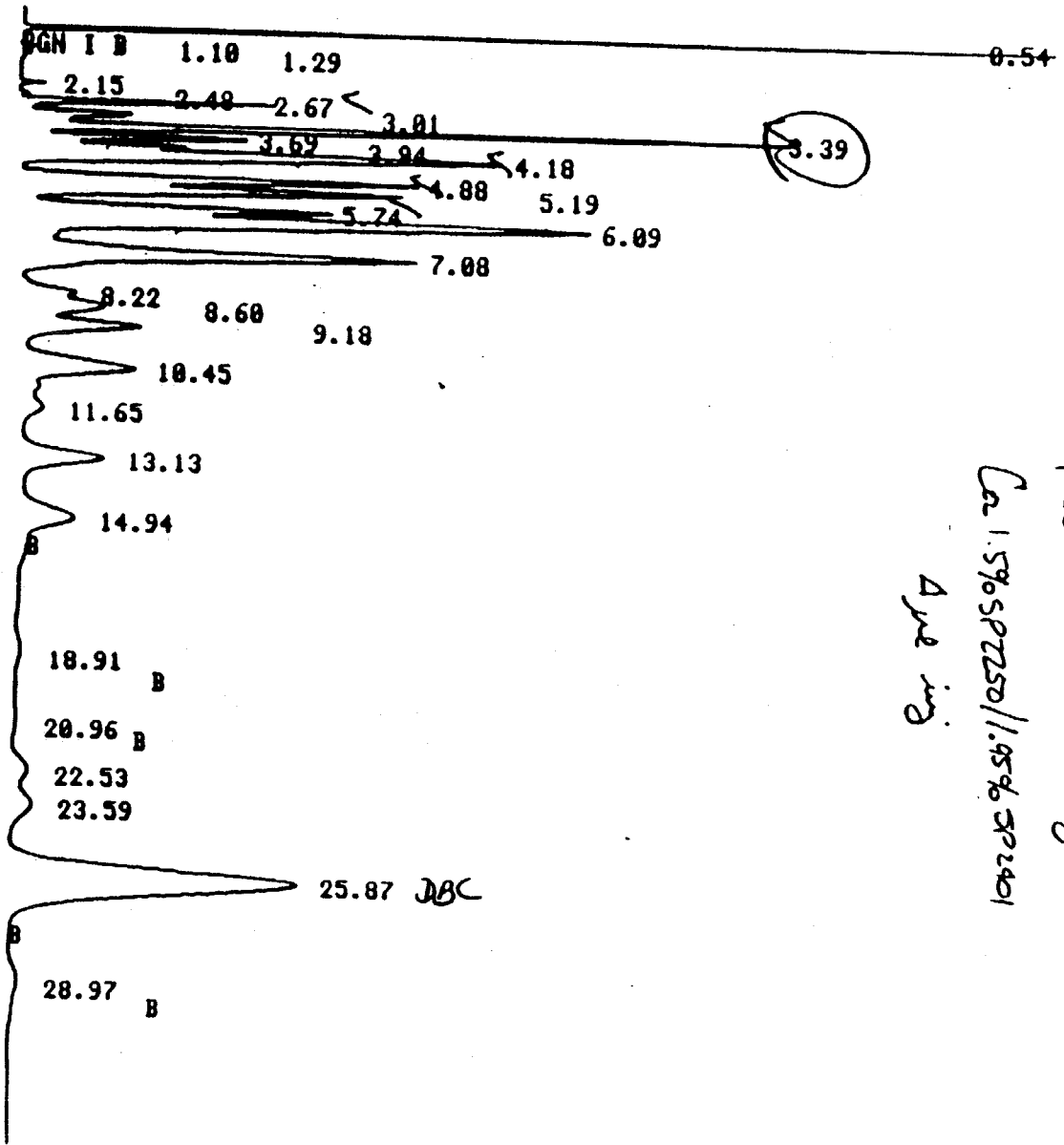


PCB 1242 4.0 ng
Ca 1.5% SP2250/1.95% SP2401
4 µL inj

CHROMATOGRAPH #2 SETUP Mon Sep 28 19:49:30 1987
FILE pest3.010a METHOD 106 Primary Pest

FILE 477 RUN 10 STARTED 19:52.5 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



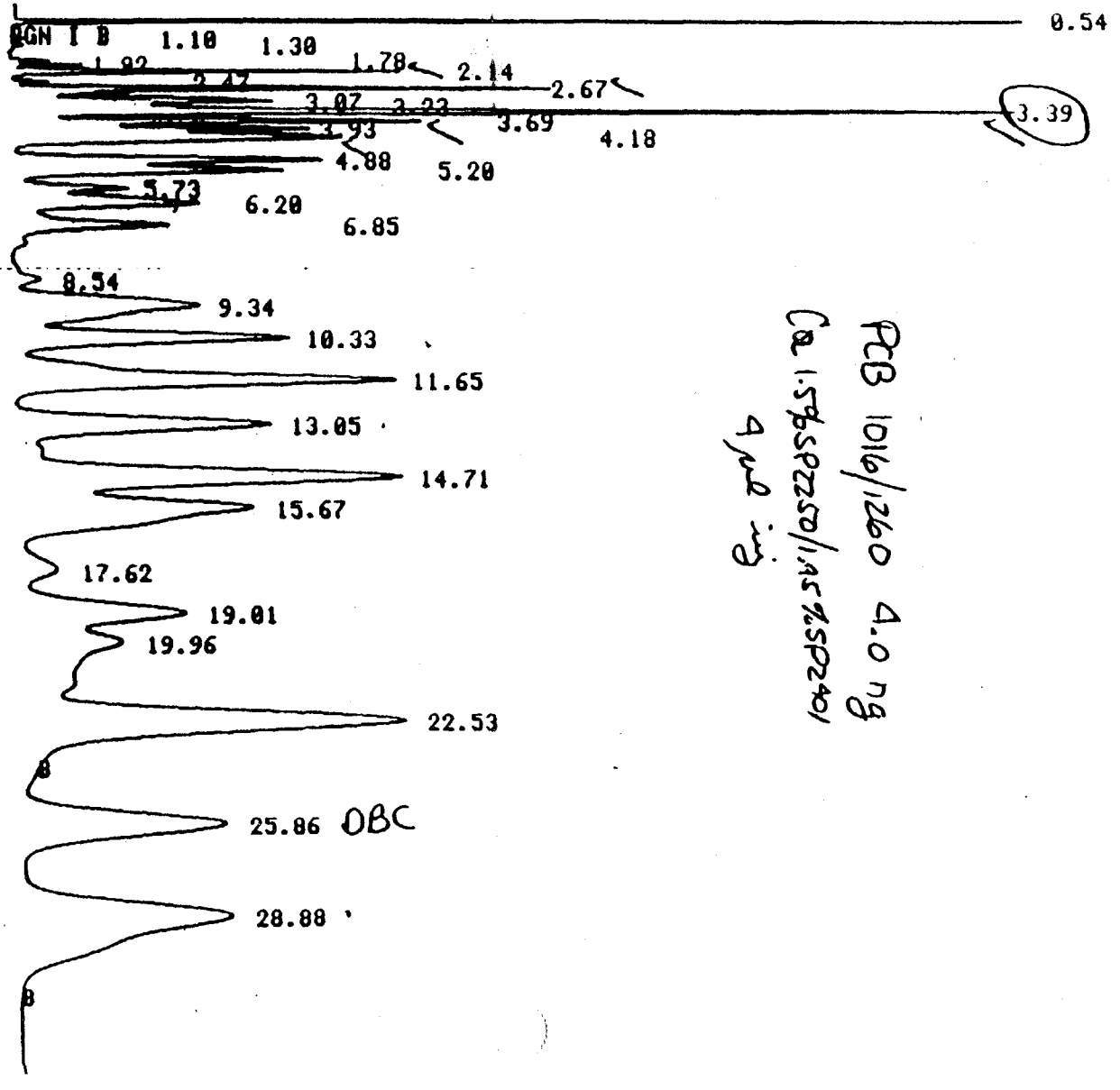
PCB 1248 4.0 ng
Col 1.5% SP2250/1.95% SP2401
4 ppm inj

30065

CHROMATOGRAPH #2 SETUP Mon Sep 28 20:26:42 1987
FILE pest3.011a METHOD 106 Primary Pest

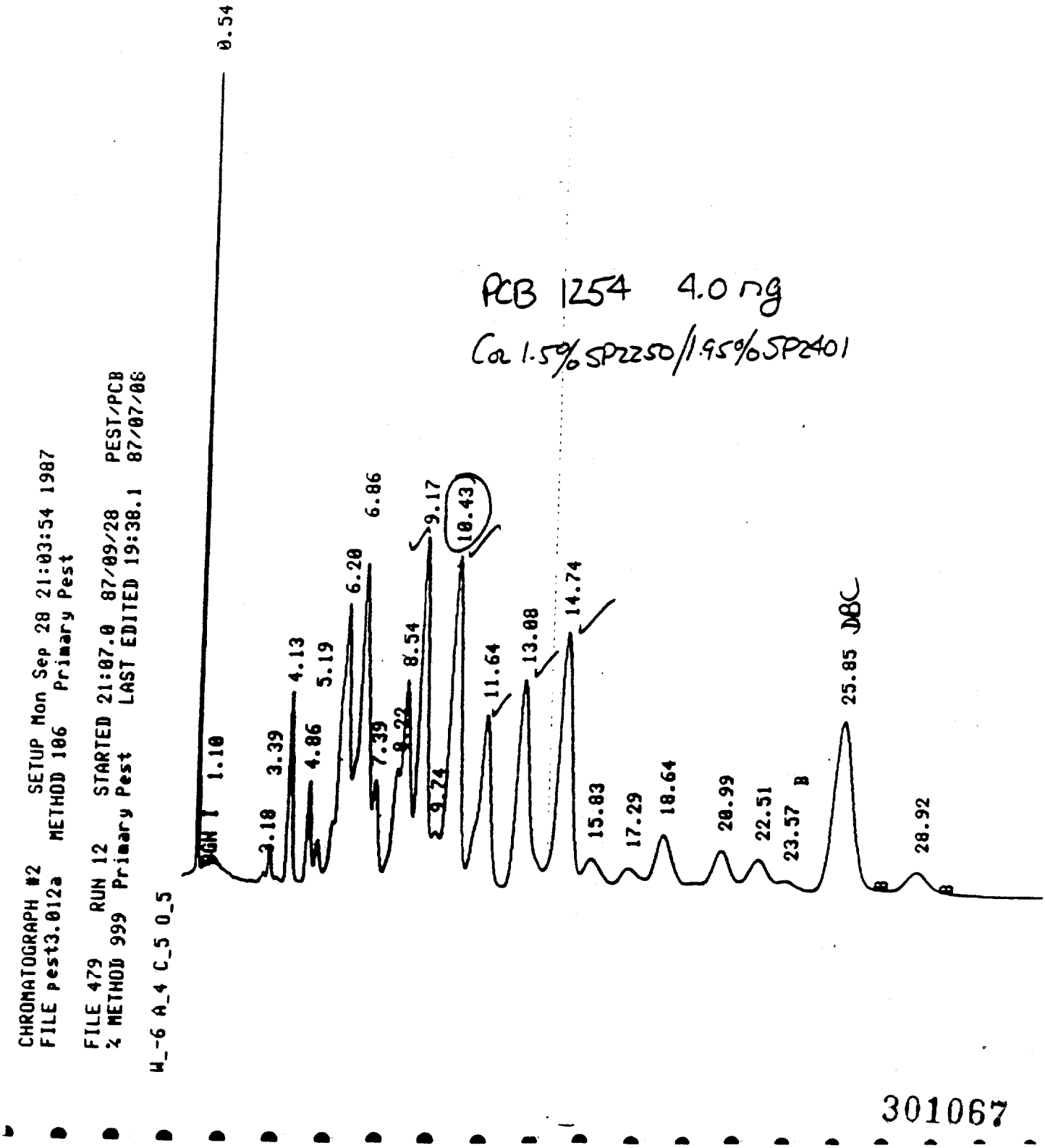
FILE 478 RUN 11 STARTED 20:29.7 87/09/28 PEST/PCB
% METHOD 999 Primary Pest LAST EDITED 19:38.1 87/07/08

W_-6 A_4 C_5 O_5



PCB 1016/1260 4.0 ng
(a 1.5% SP2250/1.95% SP2401
4 µL inj

FIGURE 17



301067

301068

ROP QRA #2 S. Tu 10: 15: 55
FILE pestconf3.003a METHOD 206 pestconf

FILE 37 RUN 3 STARTED 15:26.4 88/11/29
% METHOD 999 pestconf LAST EDITED 23:53.2 88/05/02

W_6 A_4 C_4 D_5

0.37
0.85 BGN B 1.16
1.95 B
3.54
4.77 Aldrin .20 ng 5.39
6.28
B W_7
9.90 4,4'DDE Endrin .40ng 10.38
12.64 4,4'DDD + Endrin Aldehyde
4,4'DDT 1.0ng 16.64
18.86 Endrin Ketone
B
I_8
34.75 DBC .40ng
B

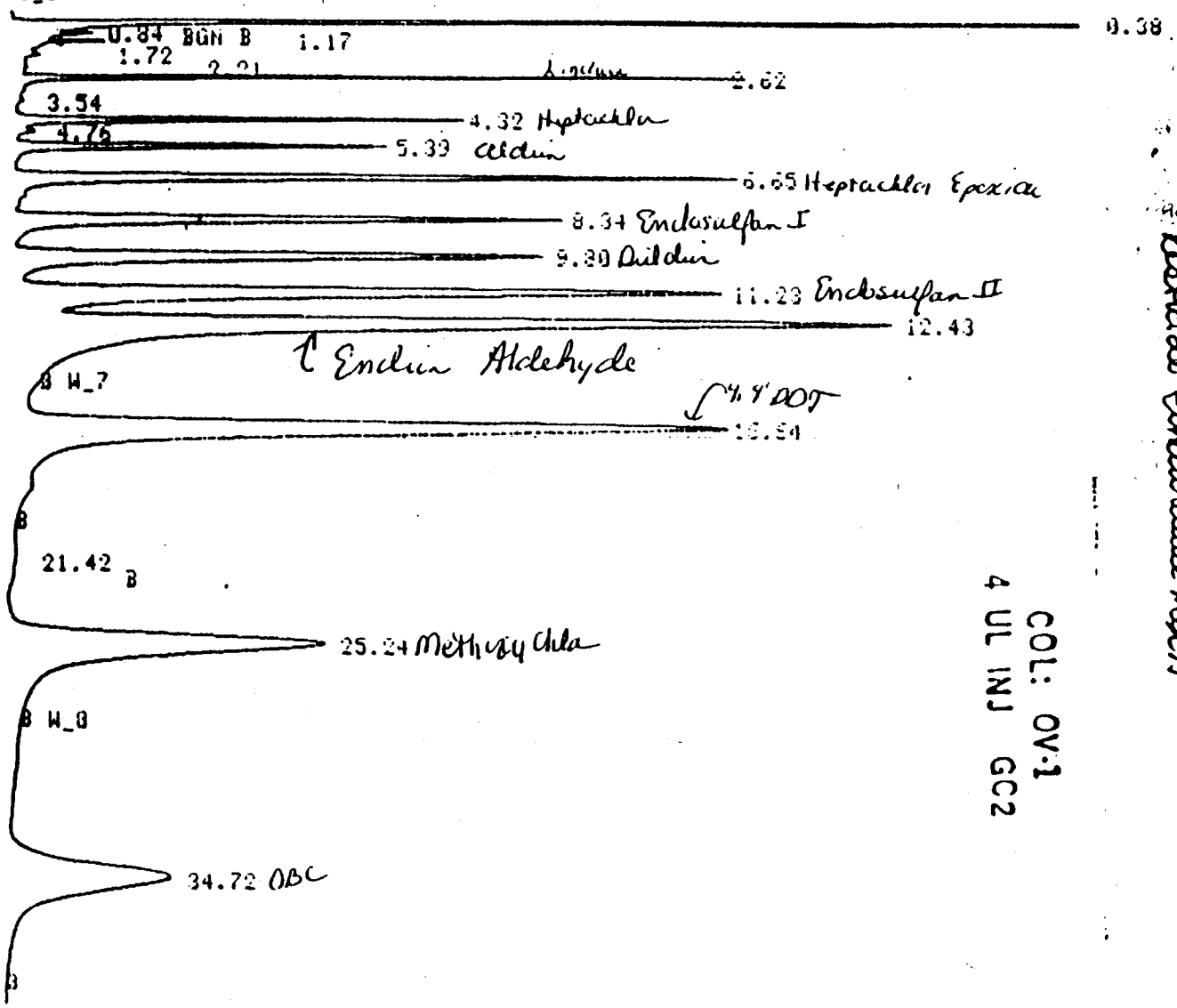
COL: OV-1
4 UL INJ GC2

Pesticide Evaluation Mix A

CHROMATOGRAPH #2 SETUP Tue Nov 29 16:50:45 1988
FILE pestconf9.005a METHOD 206 pestconf

FILE 39 RUN 5 STARTED 16:56.9 88/11/29
% METHOD 999 pestconf LAST EDITED 23:53.8 88/09/02

W_6 A_4 C_4 0_5



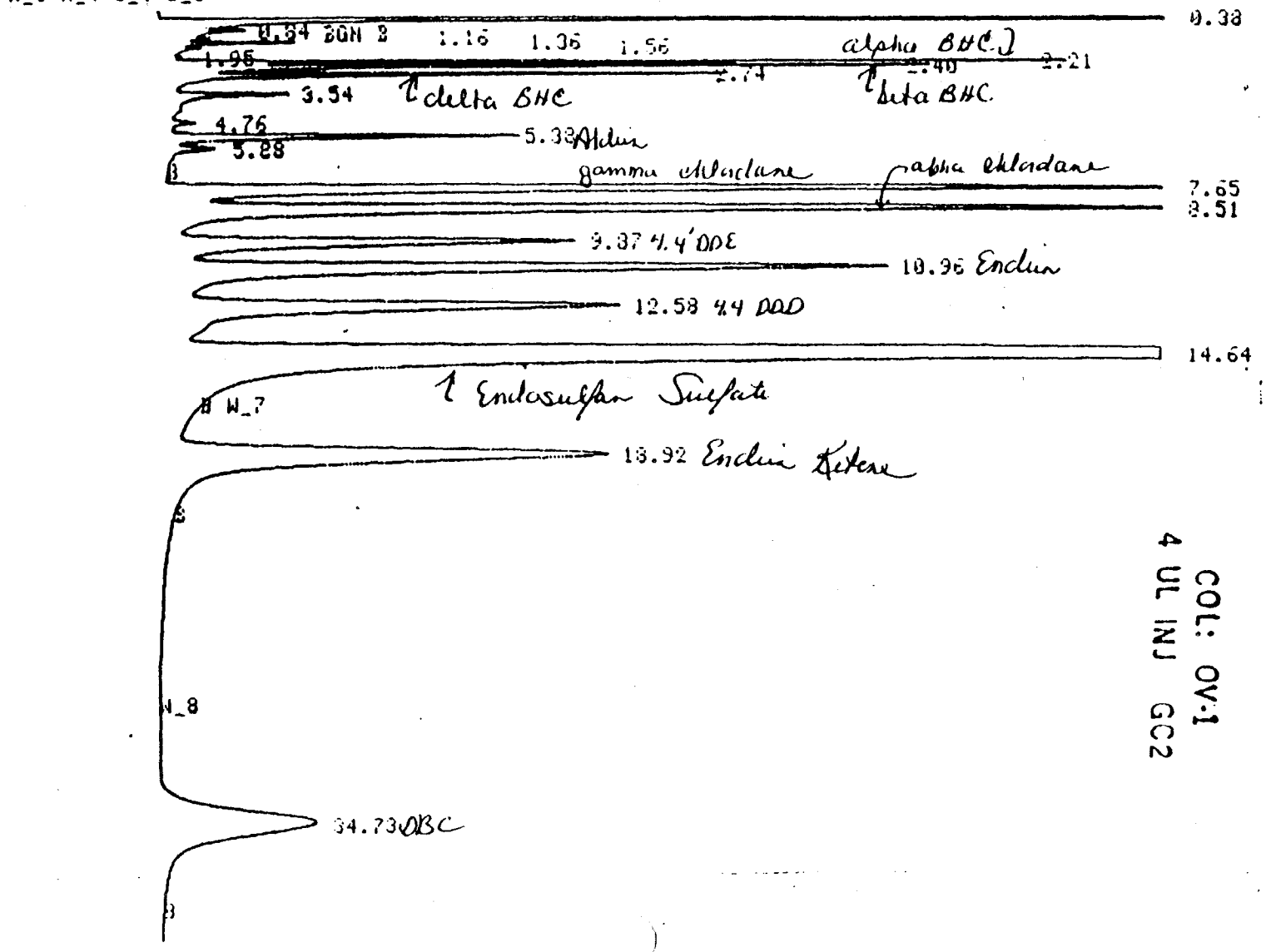
COL: OV-1
4 UL INJ GC2

Pesticide Individual Mix A

CHROMATOGRAPH #2 SETUP Tue Nov 29 17:35:33 1988
FILE pestconf9.006a METHOD 206 pestconf

FILE 40 RUN 6 STARTED 17:42.2 88/11/29
% METHOD 999 pestconf LAST EDITED 23:56.8 88/09/02

W_6 A_4 C_4 0_5



COL: OV-1
4 UL INJ GC2

Pesticide Individual Mixes

FIGURE 23

FIGURE 11

CALCULATION SUMMARY - QC

1) Calibration Factor:

$$C_f = \frac{\text{area}}{\text{mass injected (ng)}}$$

ng = ug/mL x uL inj.

2) Response Factor:

$$R_f = \frac{\text{conc std (ug/mL)}}{\text{total area}}$$

3) Percent RSD = $\frac{SD}{x} \times 100$

4) Percent Difference = $\frac{R_1 - R_2}{R_1} \times 100$

C_f: where R_1 = C_f from first analysis
R_2 = C_f from second analysis

DBC Shift: where R_1 = RT DBC initial injection
R_2 = RT DBC in subsequent samples

5) Breakdown

% for DDT $\frac{\text{area DDE + area DDD}}{\text{area DDT + area DDE + area DDD}}$

% for Endrin $\frac{\text{area Endrin Ketone + area Endrin Aldehyde}}{\text{area Endrin Ketone + area Endrin Aldehyde + Endrin area}}$

% Combined $\frac{\text{area DDE + area E.K. + area DDD/Endrin Aldehyde}}{\text{area DDE + area E.K. + area DDD/Endrin Aldehyde + area Endrin + area DDT}}$

6) Matrix Spike Percent Recovery: $\frac{SSR - SR}{SA} \times 100$

where: SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

7) Surrogate Percent Recovery: $\frac{Qd}{Qa} \times 100$

where: Qd = Quantity Determined
Qa = Quantity Added

SAMPLE CALCULATIONS

Pesticides, PCB's & Herbicides:

Low Level Soils: $\frac{\text{area sample (ng injected) final vol(uL)(20*)(DF)}}{\text{area std. (uL inj) (wt x dec. \% solids)}}$ = ug/Kg

Waters: $\frac{\text{area sample (ng injected) final vol (uL)}}{\text{area std. (uL inj) sample vol. extracted (mLs)}}$ = ug/L

Miscellaneous:

Wipe samples: (R_f std x area sample) final volume = total ug

Oils: $\frac{B \times V_t}{A \times W}$ = mg/Kg

where: $A = \frac{\text{area std}}{\text{ng std}}$ $B = \frac{\text{area sample}}{\text{uL inj}}$

V_t = volume sample (mL)
W = weight sample (gms)

Miscellaneous GC:

R_f std x area unknown = ug/mL

Impinger: ug/mL x initial vol sample = total ug

Note: all calculations are by external standard method

*for medium level soils use 10 as quantitation factor instead of 20. If GPC was done use DF of 2 times any other required dilution factor.

MATRIX SPIKE CALCS

$$\text{Percent RPD} = \frac{MS - MSD}{\frac{MS - MS2}{2}} \times 100$$

Theoretical Calc:

a) H_2O	spiking solution concentration		10 ml final volume final concentration
Lindane	.2 ug/mL	spike	.20 ug/L
Heptachlor	.2 ug/mL	1 ml	.20 ug/L
Aldrin	.2 ug/mL	to	.20 ug/L
Dieldrin	.5 ug/mL	1L	.50 ug/L
Endrin	.5 ug/mL	sample	.50 ug/L
4,4'DDT	.5 ug/mL		.50 ug/L

b) Soils	spiking solution concentration		total ug
Lindane	2.0 ug/mL	spike	.8
Heptachlor	2.0 ug/mL	with .4	.8
Aldrin	2.0 ug/mL	mL --	.8
Dieldrin	5.0 ug/mL	30g	2.0
Endrin	5.0 ug/mL	sample	2.0
4,4'DDT	5.0 ug/mL		2.0

ug/Kg calc. as follows:

$$\text{eg. Lindane} = \frac{.8 \text{ ug}}{\text{wt (gms)} \times \text{dec \% solids}} \times 1000 = \text{ug/Kg}$$

TARGET COMPOUND LIST (TCL) AND CONTRACT REQUIRED QUANTITATION LIMITS (CRQL)*

Pesticides/PCB's	Quantitation Limits** Water, ug/L	Low Soil/ Sediment*, ug/Kg
alpha-BHC	0.05	8.0
beta-BHC	0.05	8.0
delta-BHC	0.05	8.0
gamma-BHC (lindane)	0.05	8.0
Heptachlor	0.05	8.0
Aldrin	0.05	8.0
Heptachlor epoxide	0.05	8.0
Endosulfan I	0.05	8.0
Dieldrin	0.10	16.0
4,4'-DDE	0.10	16.0
Endrin	0.10	16.0
Endosulfan II	0.10	16.0
4,4'-DDD	0.10	16.0
Endosulfan sulfate	0.10	16.0
4,4'-DDT	0.10	16.0
Methoxychlor	0.5	80.0
Endrin ketone	0.10	16.0
alpha-Chlordane	0.5	80.0
gamma-Chlordane	0.5	80.0
Toxaphene	1.0	160.0
Aroclor-1016	0.5	80.0
Aroclor-1221	0.5	80.0
Aroclor-1232	0.5	80.0
Aroclor-1242	0.5	80.0
Aroclor-1248	0.5	80.0
Aroclor-1254	1.0	160.0
Aroclor-1260	1.0	160.0

* Medium soil/sediment contract required quantitation limits (CRQL) for pesticide/PCB TCL compounds are 15 times the individual low soil/sediment CRQL.

* Specific quantitation limits are highly matrix dependent. The quantitation limits listed herein are provided for guidance and may not always be achievable.

** Quantitation limits listed for soil/sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

Instrument ID: GC4A/GC4B

Laboratory: ILA, Inc.-CT - MDL

EPA PRIORITY POLLUTANT AND TCL PESTICIDES/PCB'S LIST AND MDL

All values are ug/L.

Compound	Replicate Analyses R_1	R_2	R_3	R_4	R_5	R_6	R_7	SD	MDL[1,2]
alpha-BHC	0.044	0.046	0.046	0.046	0.044	0.046	0.046	0.001	0.003
beta-BHC	0.048	0.049	0.050	0.050	0.049	0.051	0.049	0.001	0.004
delta-BHC	0.047	0.046	0.046	0.047	0.047	0.048	0.046	0.001	0.002
gamma-BHC	0.041	0.041	0.042	0.041	0.041	0.043	0.042	0.001	0.002
Heptachlor	0.057	0.058	0.057	0.058	0.056	0.058	0.056	0.001	0.003
Aldrin	0.044	0.046	0.046	0.047	0.045	0.046	0.045	0.001	0.003
Heptachlor Epoxide	0.048	0.052	0.053	0.049	0.052	0.051	0.052	0.002	0.005
Endosulfan I	0.025	0.025	0.030	0.025	0.029	0.026	0.024	0.002	0.007
Dieldrin	0.048	0.048	0.047	0.049	0.047	0.048	0.047	0.001	0.003
4,4'DDE	0.050	0.050	0.050	0.049	0.050	0.049	0.049	0.001	0.002
Endrin	0.231	0.241	0.234	0.229	0.231	0.224	0.226	0.005	0.017
Endosulfan II	0.493	0.502	0.497	0.503	0.518	0.509	0.497	0.085	0.027
4,4'DDD	0.333	0.343	0.343	0.355	0.341	0.359	0.361	0.011	0.034
Endosulfan Sulfate	0.322	0.335	0.374	0.419	0.377	0.401	0.365	0.034	0.107
4,4'DDT	0.265	0.271	0.272	0.282	0.271	0.288	0.282	0.008	0.026
Methoxchlor	0.256	0.256	0.256	0.256	0.256	0.192	0.321	0.037	0.116
Endrin Aldehyde	0.122	0.115	0.113	0.130	0.116	0.134	0.130	0.009	0.027
Toxaphene	0.784	0.8327	0.7776	0.8367	0.8236	0.7826	0.8226	0.0321	0.101
alpha-Chlordane	0.498	0.485	0.515	0.503	0.495	0.482	0.495	0.011	0.035
gamma-Chlordane	0.511	0.500	0.533	0.507	0.504	0.487	0.509	0.014	0.044
Endrin Ketone	0.479	0.470	0.491	0.485	0.478	0.469	0.0490	0.089	0.028
PCB-1016	1.558	1.383	1.492	1.463	1.520	1.697	1.452	0.0995	0.313
PCB-1221	1.655	1.307	1.342	1.618	1.411	1.311	1.326	0.1494	0.470
PCB-1232	1.451	1.553	1.548	1.437	1.406	1.384	1.325	0.0837	0.263
PCB-1242	1.483	1.514	1.440	1.467	1.552	1.520	1.440	0.0426	0.134
PCB-1248	1.442	1.465	1.619	1.508	1.656	1.544	1.514	0.0780	0.245
PCB-1254	1.287	1.054	1.244	1.368	1.332	1.665	1.260	0.1837	0.577
PCB-1260	1.280	1.411	1.694	1.358	1.628	1.413	1.256	0.1670	0.525

FIGURE 23b

[1] The reference to the MDL calculation is Federal Register 40 CFR Part 136 Appendix B, October 26, 1984.

The reference to the Precision and Accuracy is from SW 846 - 3rd Edition Chapter One Section 1.1.8

[2] Low and Medium soil/sediment MDL for Pesticides/PCB TCL compounds are 160 and 2400 times the individual low water MDL respectively.

Instrument ID: GC4A/GC4B **Laboratory: IEA, Inc.-CT - MDL**

EPA PRIORITY POLLUTANT AND TCL PESTICIDES/PCB'S

Compound	Precision	Accuracy
alpha-BHC	±10	107
beta-BHC	±17	109
gamma-BHC	±15	100
delta-BHC	±15	101
Heptachlor	±16	84
Aldrin	±38	70
4,4'DDE	±35	91
Dieldrin	±21	105
4,4'DDD	±41	104
Endrin Aldehyde	±5.5	92
4,4'DDT	±36	109
alpha-Chlordane	±14	108
gamma-Chlordane	±11	115
Endosulfan I	±28	103
Endosulfan II	±26	92
Endosulfan Sulfate	±16	115
Endrin	±15	103
Heptachlor Epoxide	±11	115
Methoxychlor	±23	94
Endrin Ketone	±1.7	96
Toxaphene	±4	81
PCB-1016	±6.6	151
PCB-1221	±11	142
PCB-1232	±5.8	144
PCB-1242	±22	117
PCB-1248	±5.1	154
PCB-1254	±14	132
PCB-1260	±18	114

Calibration Date:_____

Instrument ID:_____

Laboratory:_____

EPA PRIORITY POLLUTANTS PESTICIDES/PCB's

Initial Calibration Data

Calibration Level						
Compound	RF	RF	RF	RF	$\overline{RF}$	% RSD
alpha BHC						
beta BHC						
gamma BHC						
delta BHC						
Heptachlor						
Aldrin						
4,4' DDE						
Dieldrin						
4,4' DDD						
Endrin Aldehyde						
4,4' DDT						
Chlordane						
Endosulfan I						
Endosulfan II						
Endosulfan Sulfate						
Endrin						
Heptachlor Epoxide						
Toxaphene						
PCB - 1016						
PCB - 1221						
PCB - 1232						
PCB - 1242						
PCB - 1248						
PCB - 1254						
PCB - 1260						

RF - Response Factor
$\overline{RF}$ - Average Response Factor
% RSD - Percent Relative Standard Deviation

FIGURE 45

8D
PESTICIDE EVALUATION STANDARDS SUMMARY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID:_____ GC Column ID: _____

Dates of Analyses:_____ to _____

Evaluation Check for Linearity

PESTICIDE	CALIBRATION FACTOR EVAL MIX A	CALIBRATION FACTOR EVAL MIX B	CALIBRATION FACTOR EVAL MIX C	%RSD (</= 10.0%)	
ALDRIN					
ENDRIN					
4,4'-DDT					(1)
DBC					

(1) If > 10.0% RSD, plot a standard curve and determine the ng for each sample in that set from the curve.

Evaluation Check for 4,4'-DDT/Endrin Breakdown
(percent breakdown expressed as total degradation)

		DATE ANALYZED	TIME ANALYZED	ENDRIN	4,4'-DDT	COMBINED (2)
	INITIAL					
01	EVAL MIX B					
02	EVAL MIX B					
03	EVAL MIX B					
04	EVAL MIX B					
05	EVAL MIX B					
06	EVAL MIX B					
07	EVAL MIX B					
08	EVAL MIX B					
09	EVAL MIX B					
10	EVAL MIX B					
11	EVAL MIX B					
12	EVAL MIX B					
13	EVAL MIX B					
14	EVAL MIX B					

(2) See Form instructions.

FIGURE 26

8E

PESTICIDE EVALUATION STANDARDS SUMMARY
Evaluation of Retention Time Shift for Dibutylchlorendate

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID:_____ GC Column ID: _____

Dates of Analyses:_____ to _____

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED	% D	*
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						

* Values outside of QC limits (2.0% for packed columns, 0.3% for capillary columns)

PESTICIDE/PCB STANDARDS SUMMARY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Instrument ID:_____ GC Column ID: _____

DATE(S) OF ANALYSIS FROM:_____ TO:_____
TIME(S) OF ANALYSIS FROM:_____ TO:_____

DATE OF ANALYSIS _____
TIME OF ANALYSIS _____
EPA SAMPLE NO. (STANDARD) _____

COMPOUND	RT	RT WINDOW FROM	RT WINDOW TO	CALIBRATION FACTOR	RT	CALIBRATION FACTOR	QNT Y/N	%D
alpha-BHC								
beta-BHC								
delta-BHC								
gamma-BHC								
Heptaclor								
Aldrin								
Hept. epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endo.sulfate								
4,4'-DDT								
Methoxychlor								
Endrin ketone								
a. Chlordane								
g. Chlordane								
Toxaphene								
Aroclor-1016								
Aroclor-1221								
Aroclor-1232								
Aroclor-1242								
Aroclor-1248								
Aroclor-1254								
Aroclor-1260								

nder QNT Y/N: enter Y if quantitation was performed, N if not performed.
D must be less than or equal to 15.0% for quantitation, and less than or equal to 20.0% for confirmation.

ote: Determining that no compounds were found above the CRQL is a form of quantitation, and therefore at least one column must meet the 15.0% criteria.

or multicomponent analytes, the single largest peak that is characteristic of the component should be used to establish retention time and %D. Identification of such analytes is based primarily on pattern recognition

301080

FIGURE 13

NYSDEC Contract Laboratory Program
Bureau of Technical Services & Research
50 Wolf Road, Room 317
Albany, New York 12233-3502
(518) 457-7470

NYSDEC Sample Number _____

Method 608, 8080

ORGANICS ANALYSIS DATA SHEET

Pesticide/PCBs

Concentration: Low Medium (Circle One)

Date Extracted/Prepared: _____

Date Analyzed: _____

Conc/Dil Factor: _____

GPC Cleanup ☐ Yes ☐ No.

Separatory Funnel Extraction ☐ Yes ☐ No

Continuous Liquid-Liquid Extraction ☐ Yes ☐ No

CAS Number		µg/L or µg/kg (Circle One)
319-84-6	alpha-BHC	
319-85-7	beta-BHC	
319-86-8	delta-BHC	
58-89-9	gamma-BHC (Lindane)	
76-44-8	Heptachlor	
309-00-2	Aldrin	
1024-57-3	Heptachlor epoxide	
959-98-8	Endosulfan I	
60-57-1	Dieldrin	
72-55-9	4,4'-DDE	
72-20-8	Endrin	
33213-65-9	Endosulfan II	
72-54-8	4,4'-DDD	
1031-07-8	Endosulfan sulfate	
50-29-3	4,4'-DDT	
57-74-9	Chlordane	
8001-35-2	Toxaphene	
12674-11-2	AROCLOR-1016	
11104-28-2	AROCLOR-1221	
11141-16-5	AROCLOR-1232	
53469-21-9	AROCLOR-1242	
12672-29-6	AROCLOR-1248	
11097-69-1	AROCLOR-1254	
11096-82-5	AROCLOR-1260	

301081

1D

PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: (soil/water) _____ Lab Sample ID: _____

Sample wt/vol: _____(g/mL)____ Lab File ID: _____

Level: (low/med) _____ Date Received: _____

% Moisture: not dec._____ dec._____ Date Extracted: _____

Extraction: (SepF/Cont/Sonc) _____ Date Analyzed: _____

GPC Cleanup: (Y/N)___ pH:____ Dilution Factor: _____

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg)_____	Q
319-84-6	alpha-BHC		
319-85-7	beta-BHC		
319-86-8	delta-BHC		
58-89-9	gamma-BHC (Lindane)		
76-44-8	Heptachlor		
309-00-2	Aldrin		
1024-57-3	Heptachlor epoxide		
959-98-8	Endosulfan I		
60-57-1	Dieldrin		
72-55-9	4,4'-DDE		
72-20-8	Endrin		
33213-65-9	Endosulfan II		
72-54-8	4,4'-DDD		
1031-07-8	Endosulfan sulfate		
50-29-3	4,4'-DDT		
72-43-5	Methoxychlor		
53494-70-5	Endrin ketone		
5103-71-9	alpha-Chlordane		
5103-74-2	gamma-Chlordane		
8001-35-2	Toxaphene		
12674-11-2	Aroclor-1016		
11104-28-2	Aroclor-1221		
11141-16-5	Aroclor-1232		
53469-21-9	Aroclor-1242		
12672-29-6	Aroclor-1248		
11097-69-1	Aroclor-1254		
11096-82-5	Aroclor-1260		

FIGURE 10

PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

GC Column ID (1): _____ GC Column ID (2): _____

Instrument ID (1): _____ Instrument ID (2): _____

Lab Sample ID: _____

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME	RT WINDOW OF STANDARD FROM	RT WINDOW OF STANDARD TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 _____	Column 1 _____	_____	_____	-	-
02	Column 2 _____	_____	_____	-	-
03 _____	Column 1 _____	_____	_____	-	-
04	Column 2 _____	_____	_____	-	-
05 _____	Column 1 _____	_____	_____	-	-
06	Column 2 _____	_____	_____	-	-
07 _____	Column 1 _____	_____	_____	-	-
08	Column 2 _____	_____	_____	-	-
09 _____	Column 1 _____	_____	_____	-	-
10	Column 2 _____	_____	_____	-	-
11 _____	Column 1 _____	_____	_____	-	-
12	Column 2 _____	_____	_____	-	-

Comments: _____

Aq

EPA TCL PESTICIDES/PCB's

All results reported as ug/L.

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
alpha BHC						0.05
beta BHC						0.05
gamma BHC						0.05
delta BHC						0.05
Heptachlor						0.05
Aldrin						0.05
4,4' DDE						0.05
Dieldrin						0.10
4,4' DDD						0.10
Methoxychlor						0.50
Endrin Ketone						0.10
4,4' DDT						0.10
alpha Chlordane						0.50
gamma Chlordane						0.50
Endosulfan I						0.10
Endosulfan II						0.10
Endosulfan Sulfate						0.10
Endrin						0.10
Heptachlor Epoxide						0.10
Toxaphene						1.0
PCB - 1016						0.5
PCB - 1221						0.5
PCB - 1232						0.5
PCB - 1242						0.5
PCB - 1248						0.5
PCB - 1254						1.0
PCB - 1260						1.0

U, J, B - See Appendix for definition.

301084

Soil

EPA TCL PESTICIDES/PCB's

All results reported as ug/Kg.

Sample Identification

Dilution Factor					
Method Blank I.D.					
Compound					Lower Limits of Detection with no Dilution
alpha BHC					8.0
beta BHC					8.0
delta BHC					8.0
gamma BHC					8.0
Heptachlor					8.0
Aldrin					8.0
Heptachlor Epoxide					8.0
Endosulfan I					8.0
Dieldrin					16
4,4' DDE					16
Endrin					16
Endosulfan II					16
4,4' DDD					16
Endosulfan Sulfate					16
4,4' DDT					16
Methoxychlor					80
Endrin Ketone					16
alpha Chlordane					80
gamma Chlordane					80
Toxaphene					160
PCB - 1016					80
PCB - 1221					80
PCB - 1232					80
PCB - 1242					80
PCB - 1248					80
PCB - 1254					160
PCB - 1260					160

U, J, B - See Appendix for definition.

EPA TCL PESTICIDES/PCB's

All results reported as ug/Kg.

	Sample Identification				
Dilution Factor					
Method Blank I.D.					MEDIUM LEVE
Compound					Lower Limits Detection wi no Dilutio
alpha BHC					120
beta BHC					120
gamma BHC					120
delta BHC					120
Heptachlor					120
Aldrin					120
4,4' DDE					120
Dieldrin					240
4,4' DDD					240
Methoxychlor					1,200
Endrin Ketone					240
4,4' DDT					240
alpha Chlordane					1,2
gamma Chlordane					1,2
Endosulfan I					120
Endosulfan II					120
Endosulfan Sulfate					120
Endrin					120
Heptachlor Epoxide					120
Toxaphene					2,400
PCB - 1016					1,200
PCB - 1221					1,200
PCB - 1232					1,200
PCB - 1242					1,200
PCB - 1248					1,200
PCB - 1254					2,400
PCB - 1260					2,400

U, J, B - See Appendix for definition.

FIGURE --

TABLE _____
JOB # _____
CLIENT _____

Soil

EPA PRIORITY POLLUTANT PESTICIDES/PCB's

All results reported as ug/Kg.

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
alpha BHC						8.0
beta BHC						8.0
gamma BHC						8.0
delta BHC						8.0
Heptachlor						8.0
Aldrin						8.0
4,4' DDE						8.0
Dieldrin						16
4,4' DDD						16
Endrin Aldehyde						16
4,4' DDT						16
alpha Chlordane						80
gamma Chlordane						80
Endosulfan I						16
Endosulfan II						16
Endosulfan Sulfate						16
Endrin						16
Heptachlor Epoxide						16
Toxaphene						160
PCB - 1016						80
PCB - 1221						80
PCB - 1232						80
PCB - 1242						80
PCB - 1248						80
PCB - 1254						160
PCB - 1260						160

U - See Appendix for definition.
J - See Appendix for definition.
B - See Appendix for definition.
D - See Appendix for definition.

301087

FIGURE 1

TABLE _____
CLIENT _____
JOB # _____

EPA PRIORITY POLLUTANTS
PESTICIDES/PCB's

Aqueous

All Results Reported as ug/L.

	Sample Identification					
Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
alpha BHC						0.01
beta BHC						0.01
gamma BHC						0.01
delta BHC						0.01
Heptachlor						0.01
Aldrin						0.01
4,4' DDE						0.01
Dieldrin						0.01
4,4' DDD						0.05
Endrin Aldehyde						0.05
4,4' DDT						0.05
Chlordane						0.10
Endosulfan I						0.01
Endosulfan II						0.05
Endosulfan Sulfate						0.05
Endrin						0.05
Heptachlor Epoxide						0.01
Toxaphene						1.0
PCB - 1016						0.20
PCB - 1221						0.20
PCB - 1232						0.20
PCB - 1242						0.20
PCB - 1248						0.20
PCB - 1254						0.20
PCB - 1260						0.20

*U - See Appendix for definition.
*J - See Appendix for definition.
*B - See Appendix for definition.
*D - See Appendix for definition.

301088

Soil

EPA PRIORITY POLLUTANTS PESTICIDES/PCB's

All results reported as ug/Kg.

	Sample Identification				
Dilution Factor					
Method Blank I.D.					MEDIUM LEVEL
Compound					Lower Limits of Detection with no Dilution
alpha BHC					120
beta BHC					120
gamma BHC					120
delta BHC					120
Heptachlor					120
Aldrin					120
4,4' DDE					120
Dieldrin					240
4,4' DDD					240
Endrin Ketone					240
4,4' DDT					240
alpha Chlordane					1,200
gamma Chlordane					1,200
Endosulfan I					120
Endosulfan II					120
Endosulfan Sulfate					120
Endrin					120
Heptachlor Epoxide					120
Toxaphene					2,400
PCB - 1016					1,200
PCB - 1221					1,200
PCB - 1232					1,200
PCB - 1242					1,200
PCB - 1248					1,200
PCB - 1254					2,400
PCB - 1260					2,400

U, J, B - See Appendix for definition.

Aqueou

PESTICIDE RESULTS

All results reported as ug/L.

Sample Identification

Dilution Factor					
Method Blank I.D.					
Compound					Lower Limits of Detection with no Dilution
Lindane					0.01
Endrin					0.05
Methoxychlor					0.5
Toxaphene					1.0

Sample Identification

Dilution Factor					
Method Blank I.D.					
Compound					Lower Limits of Detection no Dilut.
Lindane					0.01
Endrin					0.05
Methoxychlor					0.5
Toxaphene					1.0

U, J, B - See Appendix for definition.

301090

FIGURE 38

Aqueous

E.P. TOXICITY PESTICIDE RESULTS

All results reported as mg/L.

Sample Identification

Dilution Factor					
Method Blank I.D.					
Compound					Lower Limits of Detection with no Dilution
Lindane					<0.01
Endrin					<0.001
Methoxychlor					<0.5
Toxaphene					<0.1

Sample Identification

Dilution Factor					
Method Blank I.D.					
Compound					Lower Limits of Detection with no Dilution
Lindane					<0.01
Endrin					<0.001
Methoxychlor					<0.5
Toxaphene					<0.1

T, J, B - See Appendix for definition.

301091

FIGURE 39

TABLE_____
JOB #_____
CLIENT_____

Aque

PCB's ANALYSIS

All results reported as ug/L.

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
PCB - 1016						0.50
PCB - 1221						0.50
PCB - 1232						0.50
PCB - 1242						0.50
PCB - 1248						0.50
PCB - 1254						0.50
PCB - 1260						0.50

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
PCB - 1016						0.50
PCB - 1221						0.50
PCB - 1232						0.50
PCB - 1242						0.50
PCB - 1248						0.50
PCB - 1254						0.50
PCB - 1260						0.50

U - See Appendix for definition.
J - See Appendix for definition.
B - See Appendix for definition.
D - See Appendix for definition.

301092

FIGURE 10

TABLE_____
JOB #_____
CLIENT_____

Soil

PCB's ANALYSIS

All results reported as mg/Kg.

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
PCB - 1016						0.20
PCB - 1221						0.20
PCB - 1232						0.20
PCB - 1242						0.20
PCB - 1248						0.20
PCB - 1254						0.20
PCB - 1260						0.20

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
PCB - 1016						0.20
PCB - 1221						0.20
PCB - 1232						0.20
PCB - 1242						0.20
PCB - 1248						0.20
PCB - 1254						0.20
PCB - 1260						0.20

U - See Appendix for definition.
J - See Appendix for definition.
B - See Appendix for definition.
D - See Appendix for definition.

301093

FIGURE -1

PCB'S ANALYSIS

All results reported as mg/Kg.

Sample Identification

Dilution Factor					
Method Blank I.D.					MEDIUM LEVEL
Compound					Lower Limits Detection wit no Dilution
PCB - 1016					3.0
PCB - 1221					3.0
PCB - 1232					3.0
PCB - 1242					3.0
PCB - 1248					3.0
PCB - 1254					3.0
PCB - 1260					3.0

Dilution Factor					
Method Blank I.D.					
Compound					Lower Limits Detection no Dilu.
PCB - 1016					3.0
PCB - 1221					3.0
PCB - 1232					3.0
PCB - 1242					3.0
PCB - 1248					3.0
PCB - 1254					3.0
PCB - 1260					3.0

U, J, B - See Appendix for definition.

301094

FIGURE -2

TABLE _____
JOB # _____
CLIENT _____

Soil

PCB's ANALYSIS

All results reported as ug/Kg.

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
PCB - 1016						10
PCB - 1221						10
PCB - 1232						10
PCB - 1242						10
PCB - 1248						10
PCB - 1254						10
PCB - 1260						10

Sample Identification

Dilution Factor						
Method Blank I.D.						
Compound						Lower Limits of Detection with no Dilution
PCB - 1016						10
PCB - 1221						10
PCB - 1232						10
PCB - 1242						10
PCB - 1248						10
PCB - 1254						10
PCB - 1260						10

U - See Appendix for definition.
J - See Appendix for definition.
B - See Appendix for definition.
D - See Appendix for definition.

2E
WATER PESTICIDE SURROGATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.:_____

	EPA SAMPLE NO.	S1 (DBC) #	OTHER
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			

ADVISORY QC LIMITS (24-154)

S1 (DBC) = Dibutylchlorendate

Column used to flag recovery values

* Values outside of QC limits

D Surrogates diluted out

FIGURE --

2F

SOIL PESTICIDE SURROGATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Level: (low/med)_____

	EPA SAMPLE NO.	S1 (DBC) #	OTHER
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			

ADVISORY QC LIMITS (20-150)

S1 (DBC) = Dibutylchlorendate

Column to be used to flag recovery values

* Values outside of QC limits

D Surrogates diluted out

FIGURE 3E

WATER PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMIT REC.
Lindane					56-12
Heptachlor					40-13
Aldrin					40-12
Dieldrin					52-12
Endrin					56-12
4,4' DDT					38-12

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LI RPD	QC LI REC.
Lindane					15	56-1
Heptachlor					20	40-1
Aldrin					22	40-1
Dieldrin					18	52-1
Endrin					21	56-1
4,4' DDT					27	38-1

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

FIGURE 4.6

3F

SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name:_____ Contract:_____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix Spike - EPA Sample No.: _____ Level:(low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
Lindane					46-127
Heptachlor					35-130
Aldrin					34-132
Dieldrin					31-134
Endrin					42-139
4,4' DDT					23-134

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
Lindane					50	46-127
Heptachlor					31	35-130
Aldrin					43	34-132
Dieldrin					38	31-134
Endrin					45	42-139
4,4' DDT					50	23-134

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

RPD:_____ out of _____ outside limits
Spike Recovery:_____ out of _____ outside limits

COMMENTS: _____

_____

Client_____ Date Extracted_____

Job_____ Date Analyzed_____

PCB SPIKE RESULTS

Results Reported as ug/L mg/Kg.

PCB Spiked	Amount Spiked	Sample Results	Conc. MS	% Recovery	Conc. MSD	% Recovery	RPD

FIGURE 4B

4C
PESTICIDE METHOD BLANK SUMMARY

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Lab Sample ID: _____ Lab File ID: _____

Matrix: (soil/water) _____ Level: (low/med) _____

Date Extracted: _____ Extraction: (SepF/Cont/Sonc) _____

Date Analyzed (1): _____ Date Analyzed (2): _____

Time Analyzed (1): _____ Time Analyzed (2): _____

Instrument ID (1): _____ Instrument ID (2): _____

GC Column ID (1): _____ GC Column ID (2): _____

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				

COMMENTS: _____

METHOD BLANK SUMMARY

Case No. _____ Region _____ Contractor _____ Contract No. _____

FILE ID	DATE OF ANALYSIS	FRACTION	MATRIX	CONC. LEVEL	INST. ID	CAS NUMBER	COMPOUND (HSL, TIC OR UNKNOWN)	CONC.	UNITS	CRDL

Comments: _____

FIGURE D1

GAS CHROMATOGRAPHY
PACKAGING REVIEW FORM

Job #_____

Corresponding Run IDs_____

_____

	Initial/ Date	Comments
All Samples Analyzed		
All Samples Calculated		
Correct Deliverables		
Signed Off Red Book and LIMS		

Forms	Y/N	Comments
Form II - All Samples/Blanks Present		
Form III		
Form IV		
Dates Correct		
Corresponding Samples Correct		
Form I		
Ext. Data Correct		
Dates Correct		
CRQL Correct		
Flags Correct		
Sample Scans/Report Present		
QC Forms Present/In Correct Order		
Form X		
Header Data Correct		
Required Compounds Present		
Standards		
Labeled Correctly		
Order Correct		
Tabular Data		
IDs Correct		
Blank IDs Correct		
DF Correct		
Case Narrative		

Comments:_____

_____

_____

Prepared By_____

Approved By_____

301103

FIGURE 11

GAS CHROMATOGRAPHY

QUALITY CONTROL APPROVAL REPORT

Job #_____ Corresponding Run IDs_____

Client_____ _____

Level of Reporting_____ _____

	Approved By
QC Batch Approval Sheets Completed and Authorized	
Job Packaging Review Form Completed and Authorized	
All Required Forms Present	
Required Raw Data Present	
Case Narrative Complete with Deviations from Protocols/SOP Noted	
Result Transcriptions and Calculations Checked	
Logbook Copies Present	

Approval to Release Data_____

Date _____

Comments:_____

_____

_____

_____

FIGURE 10

4,4'-DDT — 5000 ug/ml (methanol)

Ampule #	Lot #	Date Rec'd	Bot #	Rec'd By	Date Used	Used
1	124-02-03	5/1/86	3	MKL	4/10/87	KPC
2	124-07-01	4/7/87	19	KPC	7/9/87	KPC
3	124-04-01	4/7/87	19	KPC		
4	124-04-01	4/7/87	19	KPC		

401120
702 [illegible]

EPA ICV Solution

Rec'd 3/12/90

ICF TECHNOLOGY INCORPORATED

October 13, 1989

INITIAL CALIBRATION VERIFICATION SOLUTIONS FOR THE
CONTRACT LABORATORY PROGRAM
EPA-EMSL AND ICF TECHNOLOGY, INC.

The Initial Calibration Verification Solutions (ICVs) are to be used to evaluate the accuracy of the initial calibrations of ICP and AA instruments. These solutions may also be used as aqueous Laboratory Control Solutions.

The values for each element in the ICVs are listed below in ug/L (ppb) for the solutions that result after the concentrates have been diluted according to the following instructions:

1. ICV-1 (0489) -- For ICP use, dilute the ICV-1 concentrate 10-fold with 2% (v/v) nitric acid; pipet 10 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

2. ICV-2 (0989) -- For furnace AA use, dilute the ICV-2 concentrate 20-fold with 2% (v/v) nitric acid; pipet 5 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

3. ICV-3 (0189) -- For ICP use, dilute the ICV-3 concentrate 10-fold with 2% (v/v) nitric acid; pipet 10 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid. For furnace AA use, dilute the ICV-3 concentrate 100-fold with 2% (v/v) nitric acid; pipet 1 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

4. ICV-4 (0389) -- For the furnace AA determination of lead and thallium, dilute the ICV-4 concentrate 10-fold with 2% (v/v) nitric acid; pipet 10 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid. For the furnace AA determination of silver and cadmium, dilute the ICV-4 concentrate 100-fold with 2% (v/v) nitric acid; pipet 1 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

EPA ICV Solution

ICV-1 (0489):

(after 10-fold dilution)

Element	Value (ug/L)
Al	2010
Ba	1960
Be	483
Cd	502
Ca	48700
Cr	510
Co	502
Cu	515
Fe	2040
Pb	4800
Mg	24600
Mn	504
Ni	480
K	49000
Ag	500
Na	49600
V	488
Zn	3100

ICV-2 (0989):

(after 20-fold dilution)

Element	Value (ug/L)
As	52.6
Se	52.6

ICV-3 (0189):

(after 10-fold dilution)

Element	Value (ug/L)
Sb	978

ICV-4 (0389):

(after 10-fold dilution)

Element	Value (ug/L)
Cd	95.8
Pb	97.5
Ag	98.6
Tl	97.0

APPENDIX D

Modifier Solutions

1) Nickel Nitrate for arsenic and selenium determinations. Dissolve 4.95 gm of ACS reagent grade nickel nitrate, $Ni(NO_3)_2 \cdot 6H_2O$ in 100 ml ml nano pure water.

2) Ammonium monobasic phosphate, magnesium nitrate for lead determinations. Dissolve 4.0 gm ACS reagent grade $(NH_4)_2H_2PO_4$ and 0.2 gm $Mg(NO_3)_2 \cdot 6H_2O$ in 100 ml nano pure water.

3) One percent sulfuric acid for thallium determinations. Add 1.0 ml concentrated sulfuric acid to 99.0 ml nano pure water.

MODIFIERS SOLUTIONS

Compound	Reference	Vial Stock #	Amount Weighed	Final Volume	Code	Date
[illegible]	5	[illegible]	[illegible]	100	[illegible]	[illegible]
[illegible]	5	Fisher 724199	0.2 gm	100	[illegible]	[illegible]
$Ni(NO_3)_2 \cdot 6H_2O$	7	N04999	4.9 g	100	[illegible]	[illegible]
[illegible]	8	[illegible]	[illegible]	200	[illegible]	[illegible]
[illegible]	7	[illegible]	[illegible]	200	[illegible]	[illegible]
$Ni(NO_3)_2 \cdot 6H_2O$	7	mx 6384 KxxB	4.9 gm	100 ml	[illegible]	[illegible]
H_2SO_4	9	H_2SO_4 #3	5 ml	50 ml	[illegible]	[illegible]
$Ni(NO_3)_2 \cdot 6H_2O$	7	N04999	4.9 gm	100	041889M	041889/M
$(NH_4)H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4 gm, 0.2 gm	100	050189M	050189/M
[illegible]	7	Fisher 724199	4.97 gm	100	[illegible]	[illegible]
$(NH_4)H_2PO_4 \cdot Mg(NO_3)_2$	8	Fisher 72199	4 gm, 0.2 gm	100	058089M	053089/M
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.92 gm	100	060589M	060589/M
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4 gm, 0.2 gm	100	060889	060889/M
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4 gm / 0.2 gm	100	061989	061989/M
H_2SO_4	9	H_2SO_4 #	~~5 ml~~ 2 ml	~~5~~ 200	061989	061989/M
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.90 gm	100	061989	061989/M
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.90 gm	100	032690	032690/M
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	(4 gm/0.2 gm) x 2	200	032690	032690/M
H_2SO_4	9	H_2SO_4 #37	5 ml	500	041090	041090/M
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.90	100	041090	41090/M
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	(4 gm/0.2 gm) x 4	400	041090	41090/M

301110

APPENDIX E

USER METHOD INDEX PAGE 1 OF 3 PAGES DATE: 81 12 11

NO.	ELE-MENT	WAVE-LENGTH	CONC RANGE	TIME	TECH-NIQUE	CALI - BRATION	HOA FILE	AB FILE
1	TL	276.8	100.0	5.0	ZEEMAN	AUTO SEL.	*	*
2	BB	217.6	100.0	5.0	ZEEMAN	AUTO SEL.	*	*
3	PB	283.3	100.0	5.0	ZEEMAN	AUTO SEL.	*	*
4	MG	183.7	100.0	5.0	ZEEMAN	LINEAR	*	*
5	SE	196.0	75.0	5.0	ZEEMAN	LINEAR	*	*
6	PB	283.3	100.0	5.0	ZEEMAN	AUTO SEL.	*	*
7	TL	276.8	100.0	5.0	ZEEMAN	AUTO SEL.	*	*
8	TL	276.8	100.0	5.0	ZEEMAN	AUTO SEL.	*	*
9								
10								
11								
12								
13								
14								

TIME: 13:08

301112

PROGRAMMING MODE INSTRUMENT USER METH # 06 - PB DATE: 90/05/1

ELEMENT: PB WAVELENGTH (NM): 283.3 SLIT (NM): 0.7
PYRO COATED TUBE WITH PLATFORM - MAX POWER HEATING - GAS STOP - MATRIX MOD.
PRETREAT TEMP: 850 ATOMIZE TEMP: 1900 CHARACT. MASS (PG) 12.0

1. TECHNIQUE: ZEEMAN	2. LAMP CURRENT (MA): 10
3. SIGNAL PROCESSING: PEAK AREA	4. CALIBRATION: AUTO SELECT
5. TIME (SECONDS): 5.0	6. READ DELAY (SECONDS): 0.0
7. SCREEN FORMAT: 1.0 GRAPHICS	8. PRINTER: MAIN SUPPL
9. RECORDER SIGNAL: 0.2 CONT ABS	10. RECORDER EXP: 1000
11. STATISTICS: 2 AVG. & SD & CV	12. NOMINAL WEIGHT 1.0
13. FOLLOVER(ABS): 1.500	14. BG SCALE: 1.0

15. S1: 3.0	16. S2: 50.0	17. S3: 100.0
18. S4:	19. S5:	20. S6:
21. S7:	22. S8:	23. RSLP:

TIME: 13:1

301113

PROGRAMMING MODE HGA 600 USER METH # 06 - PB DATE: 90/05/11

ELEMENT: PB WAVELENGTH (NM): 283.3 SLIT (NM): 0.7
PYRO COATED TUBE WITH PLATFORM - MAX POWER HEATING - GAS STOP - MATRIX MOD.
PRETREAT TEMP: 850 ATOMIZE TEMP: 1900 CHARACT. MASS (PG) 12.0

STEP NUMBER	FURNACE TEMPERATURE	TIME RAMP	TIME HOLD	INTERNAL GASFLOW	READ	RECORDER
1	130	5	55	300	-	-
2	200	5	25	300	-	-
3	850	7	20	300	-	-
4	1600	0	5	0	*	*
5	2650	1	2	300	-	-
6	----	1	--	300	-	-
7	----	1	--	300	-	-
8	----	1	--	300	-	-
9	----	1	--	300	-	-

TIME: 13:09

301114

PROGRAMMING MODE AUTOSAMPLER JEEP METH # 05 - PB DATE: 01 1[illegible]

SOLUTIONS	VIAL TRAY	VOLUME	BLANK VOLUME
BLANK	01		20
STANDARD 1	02	20	--
STANDARD 2	03	20	--
STANDARD 3	04	20	--
STANDARD 4	--	20	--
STANDARD 5	--	20	--
STANDARD 6	--	20	--
STANDARD 7	--	20	--
STANDARD 8	--	20	--
PIPELINE	--	20	--
MATRIX MODIFIER 1	00	05	--
MATRIX MODIFIER 2	--	10	--
SAMPLE 05 TO 38 WITH MODIFIER : + -		20	--
SAMPLE -- TO -- WITH MODIFIER : + -		20	--

RECAL LOCATIONS: -- : -- : -- : -- : -- NUMBER OF INJECTIONS:

MODIFIER: 0 OR 1

TIME: 13:10

301115

APPENDIX F

Instrument: 3030

Date	Initials	Hours Run	Routine Maint.	NonRoutine Maint.	QC Failure	Elements an Samples Run

301117

APPENDIX G

Std Prepped Date : 05 10 99

Start Run Time : _____

CERK LABORATORIES
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458

Date : 05

FURNACE DATA SHEET

ELEMENT : Pb I.D. wt. File : _____ Instrument : 3030 Analyst : DMA

SUP	ID	SAMPLE ID	% SOLIDS	WT./VOL/VOL	DIL FACTOR	SAMPLE RECOVERY	% RECOVERY	FLAGS	VALUE ug/L
5		ICV							
6		ICB							
7		CRA							
8		CCV1 (50)							
9		CCB1							
10		PBW J100		100/100					
11		PBRNA							
12		LCSW J100							
13		LCSWA							
14		J100001							
15		001A							
16		J100001D							
17		001DA							
18		J100001S							
19		CCV2 (50)							
20		CCB2							
21		100002							
22		002A							
23		100003							
24		003A							
25		100004							
26		004A							
27		100005							
28		005A							
29		100006							
30		006A							
31		CCV3 (50)							
32		CCB3							
33		100007							
34		007A							
35		100008							
36		008A							
37		CCV4 (50)							
38		CCB4							
39									
40									

301119

APPENDIX H

QA [illegible]
Revised 01/11/[illegible]

YORK LABS/CT

Reported By: _____
Group
Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE REPORT

Client: _____ Job Number: _____ Sample Number(s): _____

Problem Reported By: _____

Category of Problem: _____
Poor Communication With ___. Sample Matrix, Instrument

Description of Problem: _____

_____
Use back of CAR/CNR or attached sheet if more space is required

_____

_____

_____

_____

_____

_____

Recommended Corrective Action(s): _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline For CAR: _____ Reply to Originator Required? [ ]

Actual Corrective Action(s) Taken: _____
Document Actual Corrective Actions On This

_____
Form and Send a Copy of Completed CAR/CNR to QA Officer and Job folder

_____

_____

_____

_____

_____
Signature and Date

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Initial Distribution: Lab manager, Marketing Manager, Branch Manager, QA Officer, Client Services, Job Folder, Other: _____
SECONDARY DISTRIBUTION: ORIGINATOR, QA OFFICER, OTHER: _____

Document Problem and Corrective Action(s) In Case Narrative: [ ]
Attachments: Telephone Log, Services Change Request, Memo, Other:

301121

APPENDIX I

Mg = 12.0
Sens: 25 ppb = 0.183

Element: Pb
Instrument: 3030

Date	Lamp	Energy	Blank	~~6.0ppb~~ 30ppb	~~25.0ppb~~ NA	50.0ppb	~~75.0ppb~~ 100ppb

301123

S.O.P. APPROVAL SHEET

REVISION CODE:

_____

SIGNATURE AND DATE:

_____

_____

RETURN TO JEFFREY C. CURRAN FOR FINAL APPROVAL

_____
JEFFREY C. CURRAN
LABORATORY MANAGER

an environmental testing company
300 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX: (203) 268-5346

STANDARD OPERATING PROCEDURE

PERKIN-ELMER 5100PC ATOMIC ABSORPTION SPECTROMETER (GRAPHITE FURNACE) 5100N

AS:092089:2

301125

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

STANDARD OPERATING PROCEDURE
PERKIN-ELMER 5100PC ATOMIC ABSORPTION SPECTROMETER
(GRAPHITE FURNACE) 5100N

INTRODUCTION

This method outlines the operation of the Perkin-Elmer 5100 PC for graphite furnace analysis of environmental samples.

It is the intent of this procedure to comply with the IEA business ethics policy.

1.0 SCOPE AND APPLICATION

1.1 This procedure describes the analysis of water and soil samples utilizing the Perkin-Elmer Model 5100PC Atomic Absorption Spectrometer.

1.2 The analytes determined using this procedure are:

Arsenic	Thallium
Selenium	Lead

2.0 SUMMARY OF METHOD

2.1 The sample is digested with acid and peroxide according to the sample preparation standard operating procedure for furnace.

2.2 The 5100PC graphite furnace is standardized using a blank and 3 working standards.

2.3 Appropriate QC check standards are measured according to EPA or routine protocols at the beginning, throughout the duration, and at the end of the analysis to assure measurement is accurate.

3.0 INTERFERENCES

Please refer to manufacturer's instruction regarding specific elements

4.0 APPARATUS AND MATERIALS

4.1 Perkin-Elmer Model 5100PC Atomic Absorption Spectrometer

4.2 Perkin-Elmer Autosampler Model AS-60

4.3 Epson Equity III+ Personal Computer

4.4 1,000 ul variable Eppendorf pipette

4.5 2.0 ml autosampler cups

4.6 20 ul fixed Eppendorf pipette

4.7 Furnace working standards as described in the furnace standards and solutions preparation logbook - See Appendix B

4.8 EPA QC standard solutions as described per EPA in the atomic spectroscopy reagents logbook as shown in Figure 1.0 - See Appendix C

4.9 Modifier solutions as described in furnace standards and solutions preparation logbook as shown in Figure 1.0 - See Appendix D

4.10 Perkin-Elmer users manual and reference manual - Model 5100PC

5.0 REAGENTS

5.1 Nitric acid

5.2 1,000 ppm standard solutions of arsenic, selenium, thallium, and lead

5.3 Nickel nitrate $Ni(NO_3)_2 \times 6H_2O$ ACS reagent grade

5.4 Ammonia monobasic phosphate $(NH_4)_2H_2PO_4$ ACS reagent grade

5.5 Magnesium nitrate $Mg(NO_3)_2 \times 6H_2O$ ACS reagent grade

5.6 Sulfuric acid

5.7 Nanopure water

Stock standard solutions, CRA solution/analytical spike solution and in-house continuing calibration solutions are prepared from the 1,000 ppm standard solutions. See Appendix A for procedure. Stock standards are dated, initialed and coded to allow for traceability of the 1,000 ppm solution, lot and batch of nitric acid used. See Appendix E for example new stock stolutions are prepared monthly.

Working standard solutions, CRA and continuing calibration solutions are prepared from the stock solutions. These solutions are also dated, initialed and coded for traceability. See Appendix B for example.

Modifier solutions are prepared on an as needed basis, date coded, and initialed. See Appendix D for example.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 Samples should be preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection.

6.2 Holding times for furnace analysis are 180 days for both the current EPA SOW and SW846 protocols.

7.0 PROCEDURE

7.1 Startup of the 5100PC

WARNING: Not to be operated in proximity to heart patients wearing electronic pacemakers. This instrument contains powerful magnets which may affect pacemakers, watches and magnetic storage media, such as recording tape and computer disks.

A) Prior to startup the operator should be familiar with the user's manual and reference manual supplied by Perkin-Elmer.

B) Before starting the instrument, check the exhaust vents to be sure they are operating. The switch is located on the wall by the telephone, it should always be on. Also check to make sure the cooling water is on and draining at a rate of approximately 1.0 liter/minute.

C) Check that the pressure of the argon supply gas is at 50 psi and that there is ample gas in the tank.

D) Check that the rinse water bottle is full (0.5 percent HNO_3 solution) and that the reservoir is empty.

E) Check that there is plenty of paper feeding to the printer and that it is aligned at the top of the page.

F) Turn on the instrument in this order only:

1) Power switch to HGA-600
2) Power switch to Zeeman 5100
3) Power switch on far left of instrument
4) Power switch on far right side of computer
5) Power switch on far right side of computer monitor
6) Power switch on far left side of printer
7) Turn on monitor, bottom knob on right hand side of monitor

G) The screen on the monitor will show two large "windows" the top one will be labeled "C:\5100_PC\", the bottom window will be labeled "C:\AA_USER\5100\". Both of these windows will have many icons in them. If you do not see these windows you will see the prompt "C>". Type: "Gem" and hit enter key and these windows will appear. Using the mouse, move the arrow to the top window and position arrow on the icon that says "5100_PC.EXE". Hit the left hand button on the Ligitech mouse twice (double click) in a rapid motion. This

will change the screen on the monitor to the Perkin-Elmer Atomic Absorption Laboratory Benchtop Software. Icons will appear in the right hand side of this screen. The icon labeled 5100 will be highlighted, double click with the mouse on the icon labeled "multi". The screen will change to the 4 windows used while analyzing samples, "Display Peaks, Display Data, HGA Control and Multi-Element Control".

Electrodeless Discharge Lamps (EDL) can now be lit. Turn toggle switch on EDL power supply to on. Turn Micronta timer on. Turn appropriate lamp number knob fully clockwise and turn lamp igniter on. Place igniter in front of EDL and watch power supply wattage increase, turn knob counterclockwise to the desired wattage, (8 watts for arsenic, 4.5 watts for selenium). Allow at least ½ hour for lamps to warm up, check wattage occasionally because lamp wattage increases as they warm up.

Clean <u>all</u> windows on the furnace with Kimwipes. Take out the windows on both sides of the furnace compartment and clean, unscrew the left hand side of the furnace housing and remove the quartz window to clean. Turn (either way) the small window on the right side of furnace housing and pull out. Clean both sides of this window with a cotton swab and Kimwipe.

Open the furnace housing by pressing the black button labeled "furnace" on the HGA-600 module. Remove the graphite tube and clean the inner and outer portions of the contact rings with a cotton swab and a Kimwipe. Tubes and platforms last approximately 16-20 hours (300 burns). Check the daily run and maintenance logbook to determine whether the tube is still good or needs to be replaced. Also fill out the maintenance logbook at this time for the run you are setting up. To put a tube back in the furnace make sure the lengthwise grooves in the tube are facing the left side of the furnace housing and use the alignment tool to get the proper location of the entrance hole of the tube in the contact ring. To close furnace hit the black furnace button on the HGA-600 module and replace all windows.

Put the autosampler in standby placing arrow on the diamond shape in the top right hand corner of the multi-element control window and click once. This will enlarge the window and bring it to the center of the screen. Click once on "sampler standby" and autosampler arm can now be removed manually. Pull down mirror behind furnace so you can see inside the graphite tube. Turn the gray knob on the left hand side of autosampler arm counterclockwise to bring arm into sample deposit position. Watch arm as it enters the

small hole in the graphite tube making sure it does not hit the side of the tube. If the arm needs adjustment turn the lock knob on front of AS-60 counterclockwise to unlock. The gray knob on the left of autosampler table is the lateral control, it will move the table left or right. The gray knob on the front of the table is the horizontal control, it will move the table forward or backward. Make the necessary adjustment to allow the autosampler capillary to enter the sample introduction hole without striking the edges. When properly aligned lock the tray into place. Check the penetration depth of the capillary by lowering the capillary tip into the graphite until it is about 2-3 mm above the L'vov platform. If the sampling arm contacts the stop before this position is reached, turn penetration depth control counterclockwise to lower the stop. This is the tan knob closest to you on the right hand side of the autosampler arm. The immersion depth of the capillary tip into the sample cups can also be adjusted using the immersion depth control, the tan knob in front of penetration depth control. The immersion depth should be adjusted so that the capillary tip is only immersed in the sample solution as deep as necessary. The deeper the tip is immersed, the greater the risk that liquid will hang up on the capillary.

After all adjustments are complete click on sampler standby again to take autosampler out of standby mode. Click on the diamond shape again to put window back in original position. Be sure to move mirror back out of the path of the detector.

The graphite tube must now be conditioned to remove any contamination. Place arrow on "windows menus" and select "element parameter" from the dropdown menus. If the instrument was already on, the last element file run will appear. Place arrow on "file window" and click on "open" dropdown menu. The element files will appear in the center of the screen. You are now looking for the file labeled "COND___ HGA". If this is not in the window, move arrow to the bottom right hand side of window and click on the down arrow symbol. This will move files up one to see additional files. Continue doing this until you see the "COND_HGA" file. Place arrow on this file and click once. Click once on "OK" box and instrument will set up this file. Click once on "bow tie" in left hand corner to remove element parameter window. If the arsenic lamp is not lit, you will get an error message stating that there is not enough energy, click on "OK" box. Click once on HGA control window to bring it to the front. Click twice on "HGA ON/OFF" icon to start the conditioning program. The instrument will go through a series of heat up and cool down steps to burn off contamination. Do this twice to make sure tube and platform are fully conditioned.

Check lamp alignment of all lamps before starting run. Using the mouse move the arrow to the windows menu and select "align lamp" from the dropdown menus. Place arrow on desired element lamp number and click once. Align lamps in the same order as you intend to run them. The turret will move into position and the wavelength and slit will adjust itself automatically. When the instrument is ready you will see an energy bar graph on the screen and an energy valve. Hit the F2 key (AGC/AIC) to bring the bar to the middle position. To align the lamp, turn horizontal and vertical alignment screws holding the lamp in place, watching for maximum deflection (to the right) of the bar graph. If bar goes off scale, hit the F2 key again to bring back to center position. Slide lamp back and forth until the graph can no longer be increased. Repeat this procedure for each lamp. Check to make sure power cords to the EDL's will not get tangled in the turret as it moves to next lamp position. To exit out of this menu, place arrow in upper left hand corner "bow tie" and click once.

7.2 Sample Evaluation

A) Locate the samples to be analyzed and compare them to the applicable prep log and traveler in order to determine if they require CLP or routine analysis protocols, or New Jersey DEP procedures which will require signing their internal chain of custody and for completeness. To determine run sequence, look at samples, paying attention to highly colored samples and sample identification (from traveler). Highly colored samples are usually an indication of high analyte concentration and high background. They should be put at the end of the run to avoid carryover and contamination of cleaner samples. Sometimes the sample identification supplied by the client, located on the traveler, can give you an idea of where the sample came from and also possible concentration of analytes.

B) You can now set up your furnace run log. The first 4 cup positions are reserved for blank and standards. The run log starts at cup 5 and goes to cup 40. The Initial Calibration Verification (ICV) standard goes in cup 5 followed by the Initial Calibration Blank (ICB). The CRA solution is next. then, depending on the protocol either a Continuing Calibration Verification (CCV) and continuing calibration blank (CCB) is run if it is CLP protocol, or samples are run if it is regular protocol. Two cup positions are used for every sample, the first position is the sample run as prepared, the second position is the sample spiked with a known spike solution. Calibration checks (CCV and CCB) are run after every 10 sample cups, the CRA solution is counted as a sample

but does not need to be spiked. The pre-digested spike sample also does not need to be analytically spiked. The sample is followed immediately by its analytical spike. Cup position 39 is reserved for the thallium matrix modifier and cup 40 is for the lead modifier. Cup position 00 is located in the white part of the AS-60 just to the left of the rinse position, this is the location of the arsenic and selenium matrix modifier.

Once the run log is filled out completely the autosampler tray can be set up. Standards must be made daily, to make, see "standards and solutions preparation logbook" for directions. Instructions are given in the preparation section of the book. Working standards must be dated and initialed every day and the "working solution" section of the logbook must be filled in every time standards are made. It is easier to pour out your blanks, standards, modifiers and QC first, then concentrate on your samples and analytical spikes. Using the 1,000 ul pipette add exactly 2,000 ul of sample to your autosampler cup and 20 ul of the spike solution. Stir this solution with the 20 ul pipette tip to assure proper mixing of the spike into the sample. This is your analytical spike and should immediately follow the cup with approximately 2,000 ul of sample. This spiked sample is labeled on your run log as the sample number followed by the capital letter "A", i.e. 1234001A.

After the autosampler tray is set up, carefully place it in the autosampler. The tray fits over the white piece in the center of the sampler. Line up the small cutout piece in the center of the sampler with position #36 on the tray and press down slowly.

7.3 Software - You are now ready to set up your ID/weight parameter table on the computer. Using the mouse, move arrow to the window menu bar, menus will drop down. Move arrow down to "ID/weight parameter" and click once. The ID/weight parameter window will appear over the other windows. If the instrument was already on, the last ID/weight table will appear. Place arrow on file menu bar and click on "new" dropdown menu. This will change ID/weight parameter window to a blank window. Hit the caps lock key, then type in your initials, hit enter. Move arrow to the "ug" box and click once, move arrow to "L" box and click once. Move arrow to the first long box under the position column and click once, it will then be highlighted. Move arrow to the edit menu bar and click on the global entry dropdown menu. Fill in your starting cup position number (usually 5), hit enter, then type in last sample position, hit enter again. Move arrow to the "OK" box and click once. This automatically fills in your sample position locations which corresponds to the cup positions on your autosampler tray and your run

log. Using the arrow keys, move the highlighted bar one space to the right, to "Sample ID". Type in the sample ID of your first sample (usually ICV). Move arrow down one position and type in next sample ID. Continue doing this until you have filled in all sample ID's in this window. Hit the "Page Down" key to bring up the next window and place arrow key at the first sample ID bar and click mouse to highlight. Continue this procedure until you have typed in all your samples. Move arrow to the file menu and click on "Save As" menu. Type in the name selected for this ID weight file and hit enter. This name should also be written on your furnace run log. To close out ID/weight parameter window, move arrow to the "bow tie" in the left hand corner of the window and click once.

You are now ready to program the instrument as to which elements and sample locations you want to run. Under the "windows" menu bar select the "multi-element parameter" menu. If the instrument was already on, the last multi-element run will appear. Place arrow on file menu bar and click on "new" dropdown menu. Type in your initials and hit enter. Under data storage file type in "raw", hit enter. The ID/weight file you just entered above will be listed next with the extension .A60 after its name. If it is not listed, hit the ESC key and type it in, then hit enter. Under sample positions type in your first sample cup (usually 5), dash (-) then last sample location, then hit enter. The first element file will now be highlighted. Always run elements in this order: arsenic, selenium, thallium and lead. The arsenic file is ASCLP, type this in and hit enter, the program automatically assigns the extension .GEL after your file. Delay should be between 0-5 minutes to allow lamps to warm up. Arsenic and selenium lamps are EDL's so they must be given at least ½ hour to warm up, that is why they are lit up at the beginning of the set up. Thallium and lead are hollow cathode lamps which are given a 5 minute warmup time. If you only want to run selected sample positions, move arrow to the "samples to analyze" bar and click once. Type in sample positions, consecutive positions are listed as first cup location - last cup position (20-25), to run selected locations, separate cups with a comma (20-25, 30-35). You are only allowed to separate 3 times so you must group samples with the same sample analysis together as best as possible when setting up your run log.

Continue filling in the element files, delay and samples to analyze for the rest of the elements. Element files are: ASCLP, SECLP, TLCLP and PBCLP. These element files contain the proper standard concentrations and sample replicates to meet EPA requirements. See attached printed of element parameter.

When the multi-element window is complete, add to the element file OS. This is a dummy file that will turn the last hollow cathode lamp off when it is through. Close out this window by placing arrow in the upper left hand corner "bow tie" and click the mouse once.

Put arrow anywhere in the multi-element control window (bottom right hand window), and click once, this will bring this window to the front. Place arrow in printer on/off box and click once, this box will then be highlighted. Put arrow in start/stop auto run box and click once. This will start the run. Each standard and sample will require double injections, with the result being the average of the two burns.

A message will appear over the windows to ask you if you want to overwrite data in data file or append to file. Place arrow on "OK" box and click to overwrite data file. The run will now start. Each standard and sample will be injected into the furnace twice, thereby giving two burns per sample as prescribed in the current EPA contract.

Keep a close watch while the instrument is standardizing to assure conditions are optimal. Watch how the autosampler is picking up sample and how it deposits it on the platform. The blank should have a peak area (A-S) between -0.005 and +0.005. Peak area (A-S) for standards should not deviate more than ±20 percent from the sensitivity check values listed in the Perkin-Elmer literature. If any of these criteria are out, stop the run and fix the problem before you begin again. Without proper standardization the run will fail so it is to your benefit to watch the beginning of each run as closely as possible. Monitor the run frequently during the analysis. Check the precision to assure that the replicates are within acceptable limits (RSD ≤20 percent) for those samples greater than the CRDL. Check that initial and continuing QC passes and analytical spike recoveries are acceptable. Again, if any of these things fail, it is an indication that there is a problem somewhere. Stop the run, identify the problem and make the appropriate corrective action before proceeding.

7.4 Instrument Shutdown - To shut down the instrument reverse steps of startup (4.1.6), computer first, 3 power switches on the instrument, then EDL's.

7.5 The 5100 has a bound maintenance logbook that is filled out every time an analysis is to be performed. The book is kept underneath the furnace on top of the HGA-600 power unit. The first column is for the date of analysis. The second column is for the analyst's initials. The next column is for recording maintenance performed, such as changing the graphite tube and platform, contact rings, or sampling capillary. The next column is for how many hours the instrument was operating and the last column is for the element and the job/case number that was analyzed (Appendix E).

7.6 A separate folder in the main filing cabinet is maintained for repair work which required a service call from a certified Perkin-Elmer technician. There is a folder labeled "5100" with the completed service tags, describing the problem, along with the corrective action.

7.7 A furnace autosample logbook is also kept under the 5100 PC desk. Every time analysis is performed - a copy of the furnace data sheet goes in the book, the elements that were analyzed for are written in the "element" space on the upper left hand corner.

8.0 QUALITY CONTROL

When the run is complete, check that all quality control (ICV's, prep blanks and laboratory control samples) meets acceptable criteria. If contamination is evident on prep blanks or LCS's fail to recover, refer to protocol to determine corrective action. If necessary submit a "corrective action report" with a copy of the data and prep sheet attached explaining the problem, to the supervisor for immediate action (Appendix I).

9.0 METHOD PERFORMANCE

9.1 Instrument detection limits are determined quarterly per the current EPA contract as stated in Exhibit E, Section II.

9.2 Control charts are kept for tracking of accuracy (LCS and spike recoveries), and precision (RPD on the duplicate analysis).

10.0 DATA REDUCTION AND DATA HANDLING

If data meets all criteria, calculate results and keep track of any reruns or dilutions needed. Once a job has been fully completed, sign and date all relevant chain of custody sheets. At the end of each completed analysis, report all sensitivities for analyses that were run, in the appropriate logbook for standards (Appendix J). For non-CLP work, report results and quality control on final report sheets. CLP work should go into the appropriate job folder.

11.0 SPECIAL PROCEDURES - N/A

12.0 USE OF OTHER REQUIRED SOP'S

Glassware cleaning SOP, standards and sample preparation SOP, standards and sample prep log SOP and corrective action SOP.

13.0 DOCUMENTATION OF PROBLEMS

13.1 Corrective Action Reports

If any discrepancy occurs during analysis that requires attention such as standard or instrument problem, sample prep problem, etc., a corrective action report is warranted. This report should be prepared per the atomic spectroscopy corrective action report SOP.

13.2 All CLP-type reports and regular reports require a case narrative that specifically addresses any anomolies and their possible causes that may have occurred during analysis.

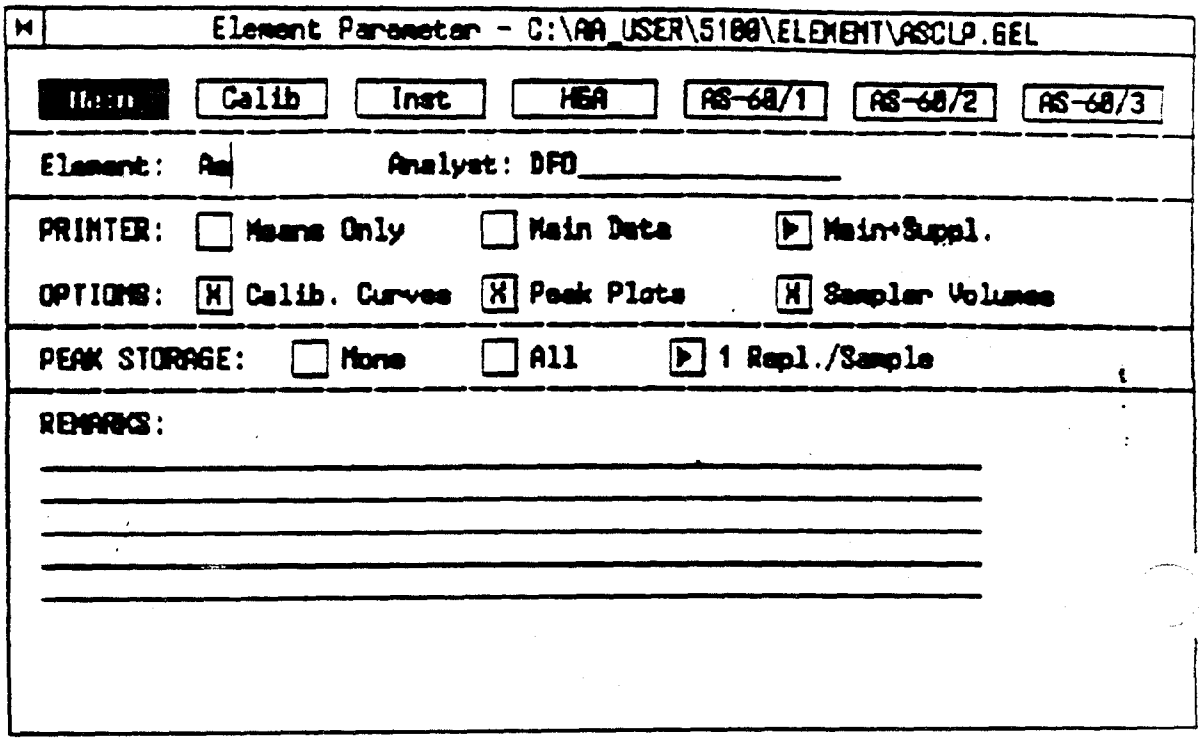
14.0 REFERENCES

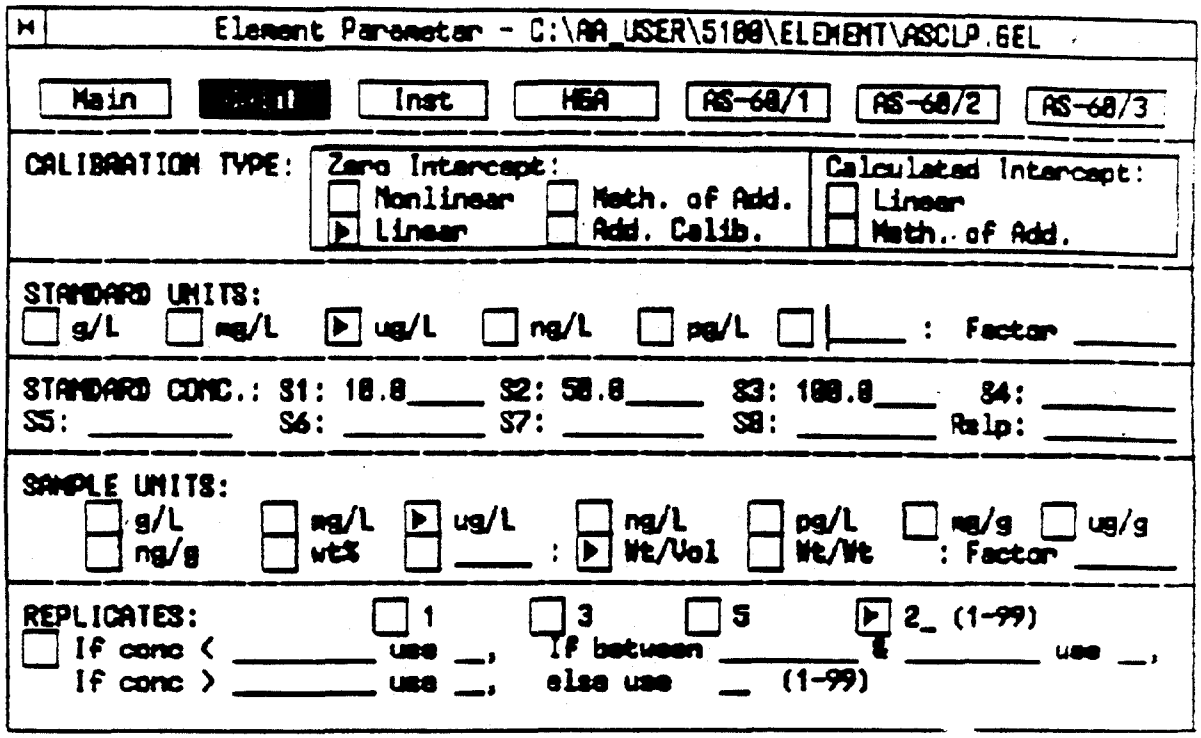
14.1 Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December, 1982.

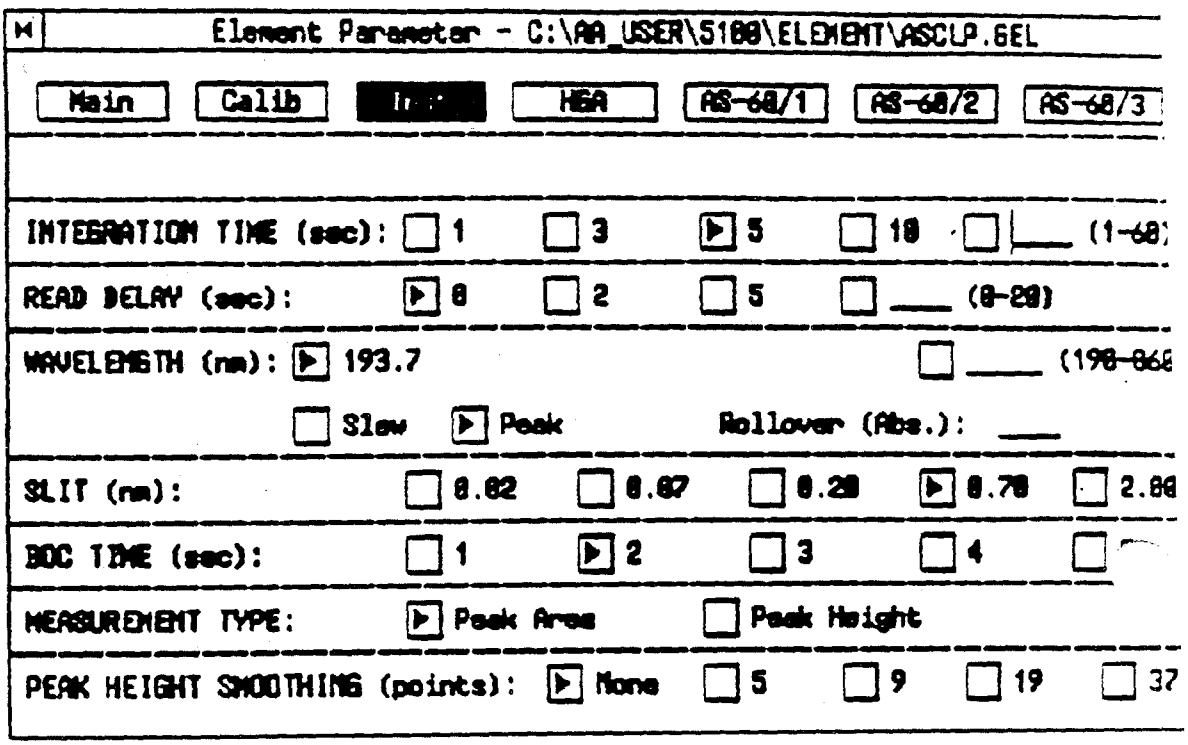
14.2 USEPA Contract Laboratory Program, SOW 7/88.

14.3 Test Methods for Evaluating Solid Waste, Volume 1A, USEPA SW846, 3rd Edition, November, 1986.

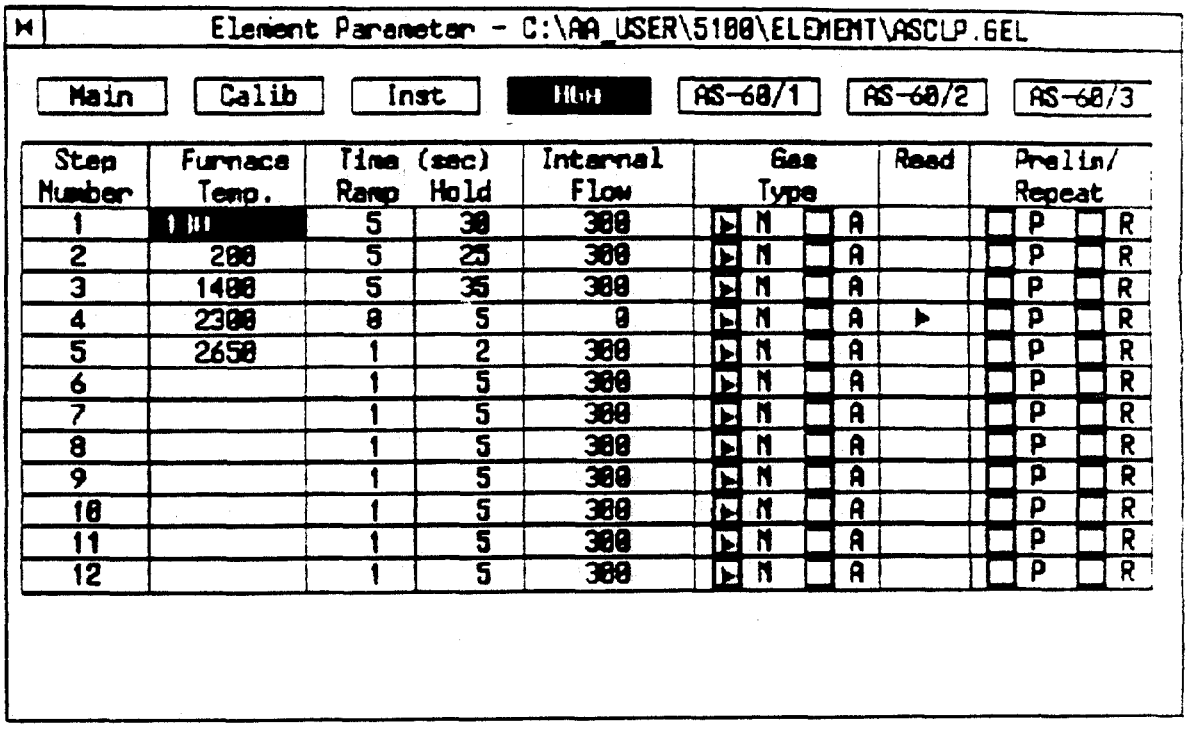
ELEMENT PARAMETERS

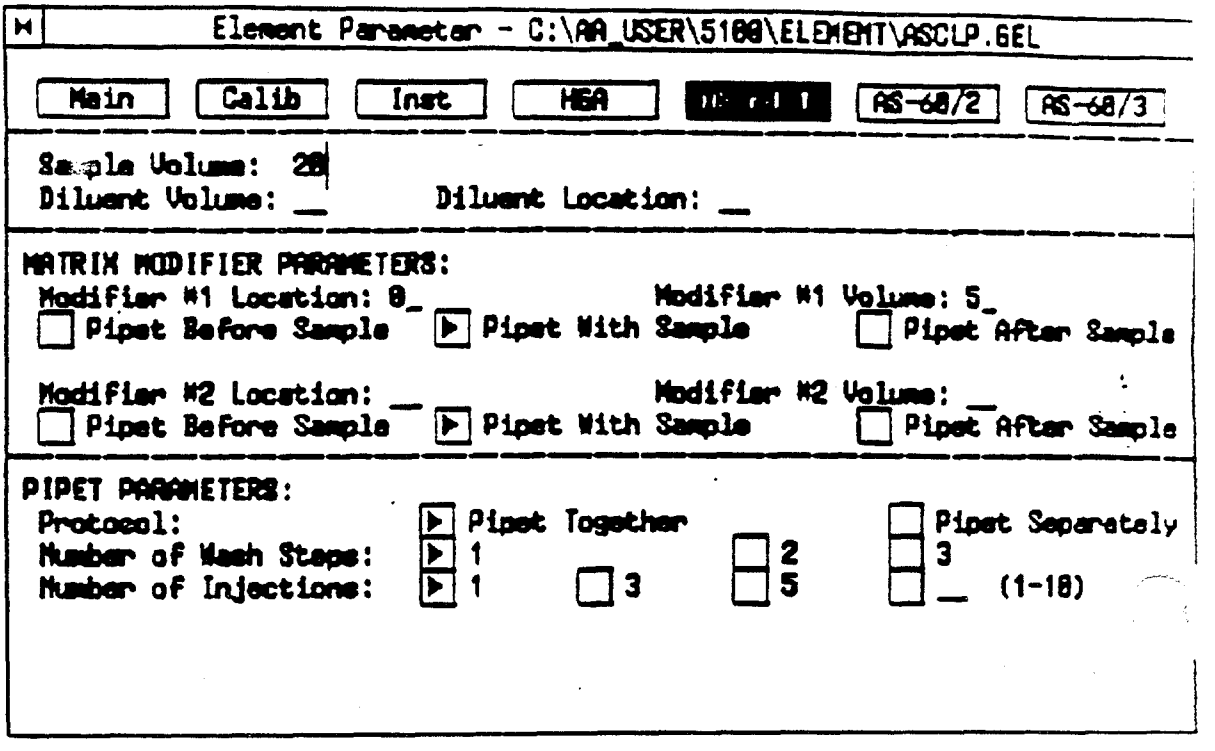

Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.GEL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
Element: Ag
Analyst: DFD
PRINTER:
Means Only
Main Data
Main+Suppl.
OPTIONS:
Calib. Curves
Peak Plots
Sampler Volumes
PEAK STORAGE:
None
All
1 Repl./Sample
REMARKS:


Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.6EL
Main
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
CALIBRATION TYPE:
Zero Intercept:
Nonlinear
Linear
Meth. of Add.
Add. Calib.
Calculated Intercept:
Linear
Meth. of Add.
STANDARD UNITS:
g/L
mg/L
ug/L
ng/L
pg/L
: Factor
STANDARD CONC.: S1: 10.0 S2: 50.0 S3: 100.0 S4:
S5: S6: S7: S8: Rslp:
SAMPLE UNITS:
g/L
ng/g
mg/L
wt%
ug/L
ng/L
Wt/Vol
pg/L
Wt/Wt
mg/g
ug/g
: Factor
REPLICATES:
1
3
5
2_ (1-99)
If conc <
use __,
If conc >
use __,
If between
&
use __,
else use
(1-99)


Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.6EL
Main
Calib
HGA
AS-60/1
AS-60/2
AS-60/3
INTEGRATION TIME (sec): 1 3 5 10 (1-60)
READ DELAY (sec): 0 2 5 (0-20)
WAVELENGTH (nm): 193.7 (190-860
Slew
Peak
Rollover (Abs.):
SLIT (nm): 0.2 0.7 2.0
BOC TIME (sec): 1 2 3 4
MEASUREMENT TYPE: Peak Area Peak Height
PEAK HEIGHT SMOOTHING (points): None 5 9 19 37

301140


Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.GEL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
Step Number
Furnace Temp.
Time (sec) Ramp
Hold
Internal Flow
Gas Type
Read
Prelin/ Repeat
1 100 5 30 300 N A P R
2 200 5 25 300 N A P R
3 1400 5 35 300 N A P R
4 2300 8 5 0 N A P R
5 2650 1 2 300 N A P R
6 1 5 300 N A P R
7 1 5 300 N A P R
8 1 5 300 N A P R
9 1 5 300 N A P R
10 1 5 300 N A P R
11 1 5 300 N A P R
12 1 5 300 N A P R


Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.6EL
Main
Calib
Inst
HGA
AS-60/2
AS-60/3
Sample Volume: 20
Diluent Volume: __
Diluent Location: __
MATRIX MODIFIER PARAMETERS:
Modifier #1 Location: 0_
Modifier #1 Volume: 5_
Pipet Before Sample
Pipet With Sample
Pipet After Sample
Modifier #2 Location: __
Modifier #2 Volume: __
Pipet Before Sample
Pipet With Sample
Pipet After Sample
PIPET PARAMETERS:
Protocol:
Pipet Together
Pipet Separately
Number of Wash Steps:
1
2
3
Number of Injections:
1
3
5
__ (1-18)

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Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.GEL

Main | Calib | Inst | HGA | AS-60/1 | [redacted] | AS-60/3

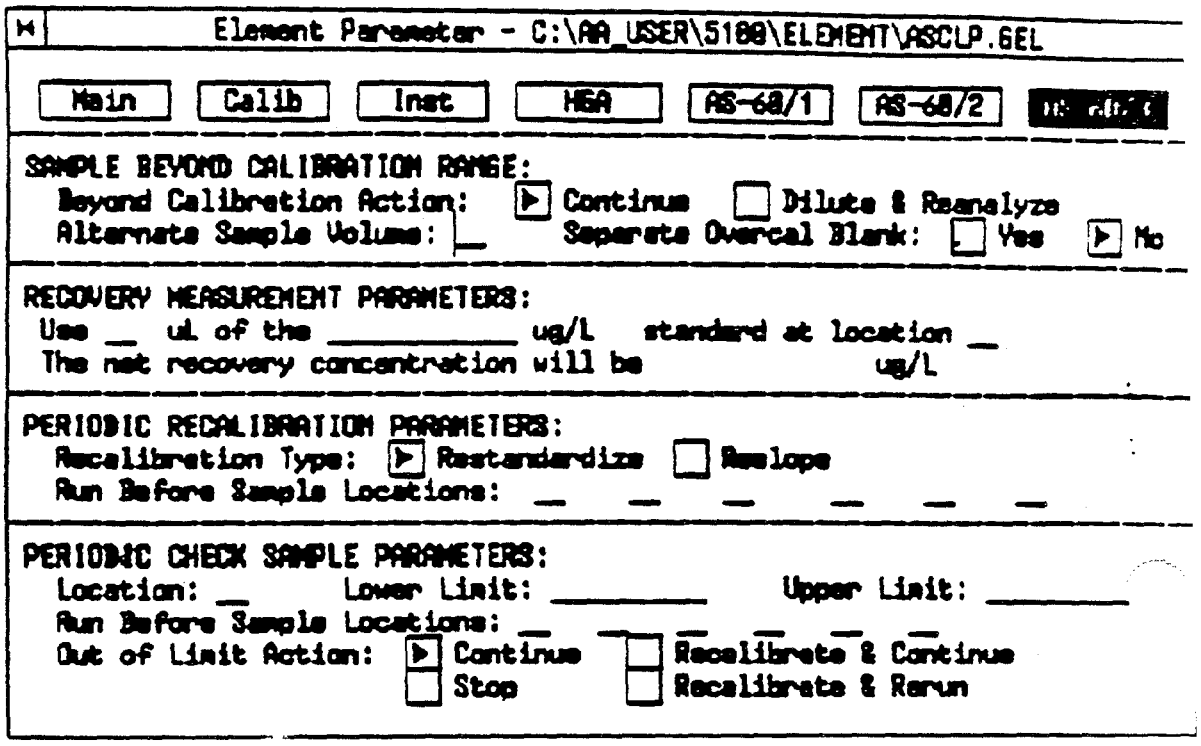
REFERENCE STANDARDS PARAMETERS:

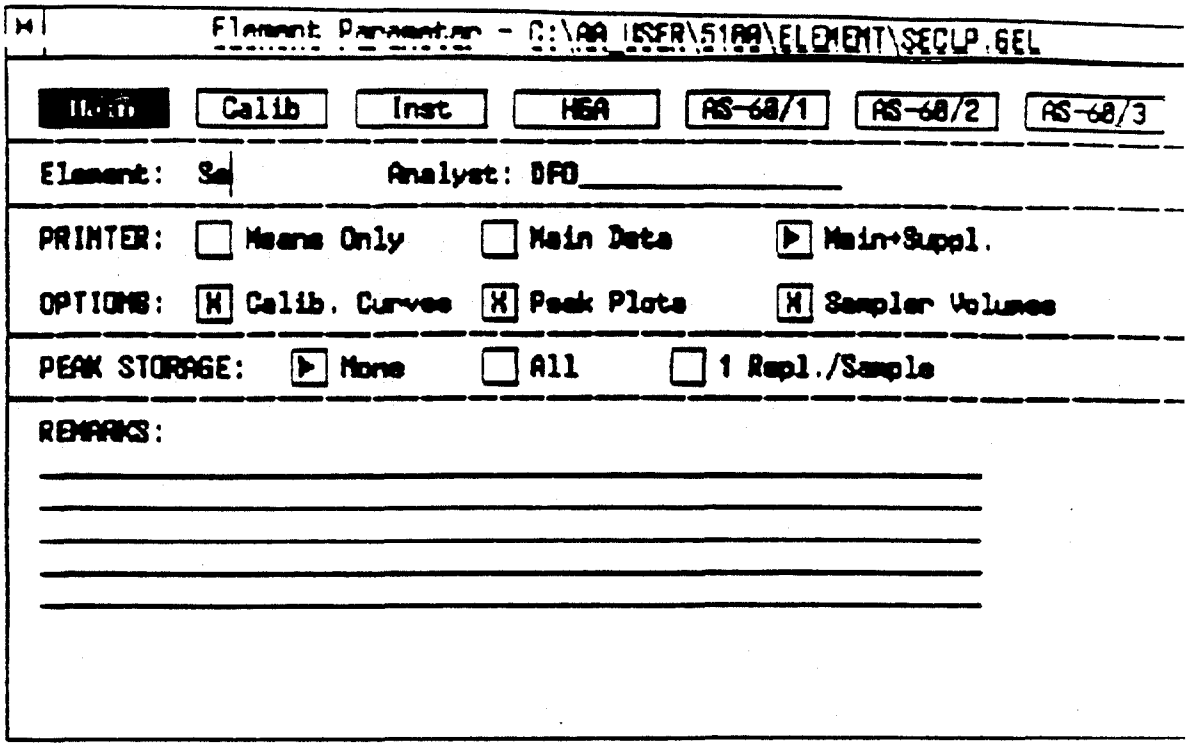
Solutions	Location	Volume	Diluent Volume	Modifier #1 Volume	Modifier #2 Volume
Std. Blank	[redacted]	20		5	
Standard 1	2	20		5	
Standard 2	3	20		5	
Standard 3	4	20		5	
Standard 4					
Standard 5					
Standard 6					
Standard 7					
Standard 8					
Reslope					

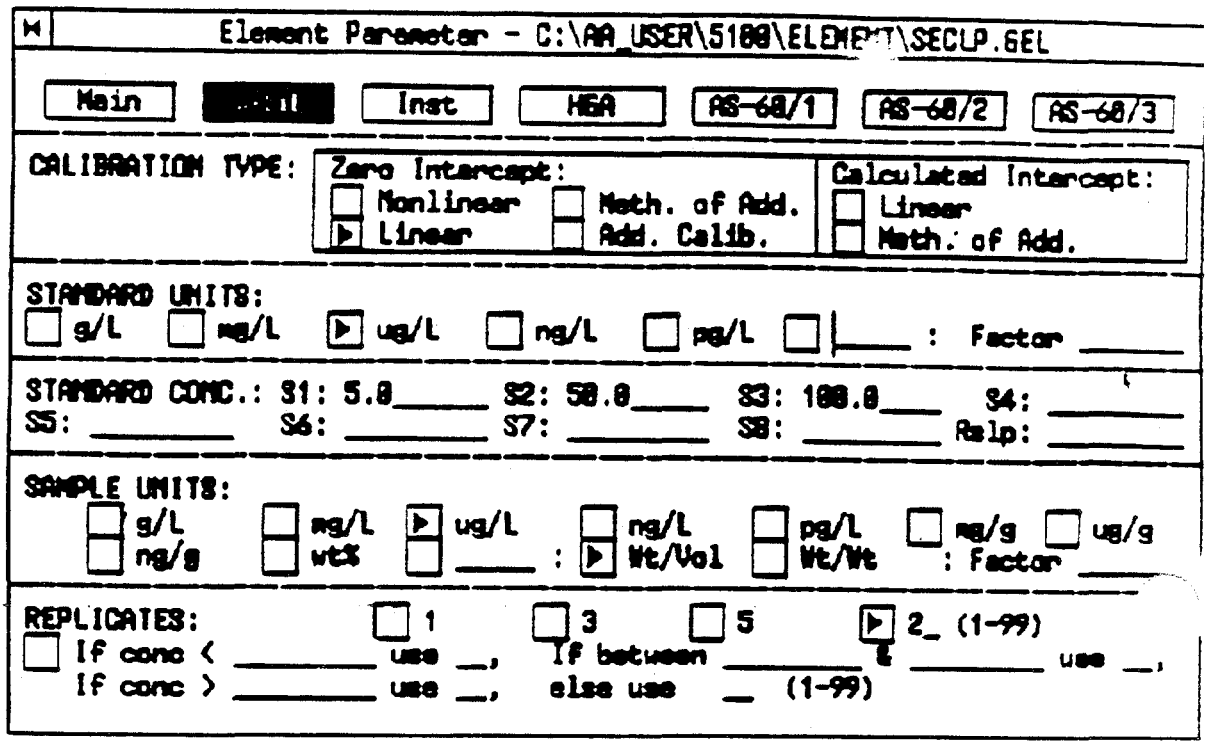
Sample Blank Location: _

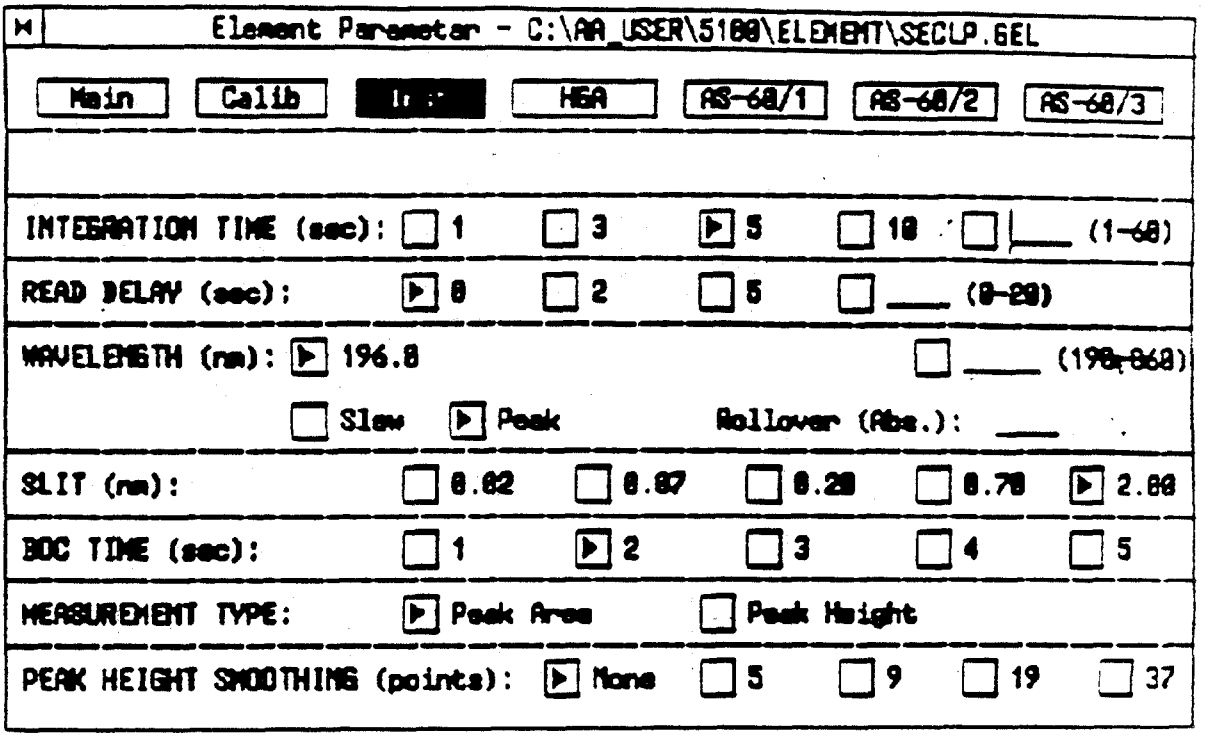
Additions Definition: ▶ Automated Additions ☐ Premixed Additions

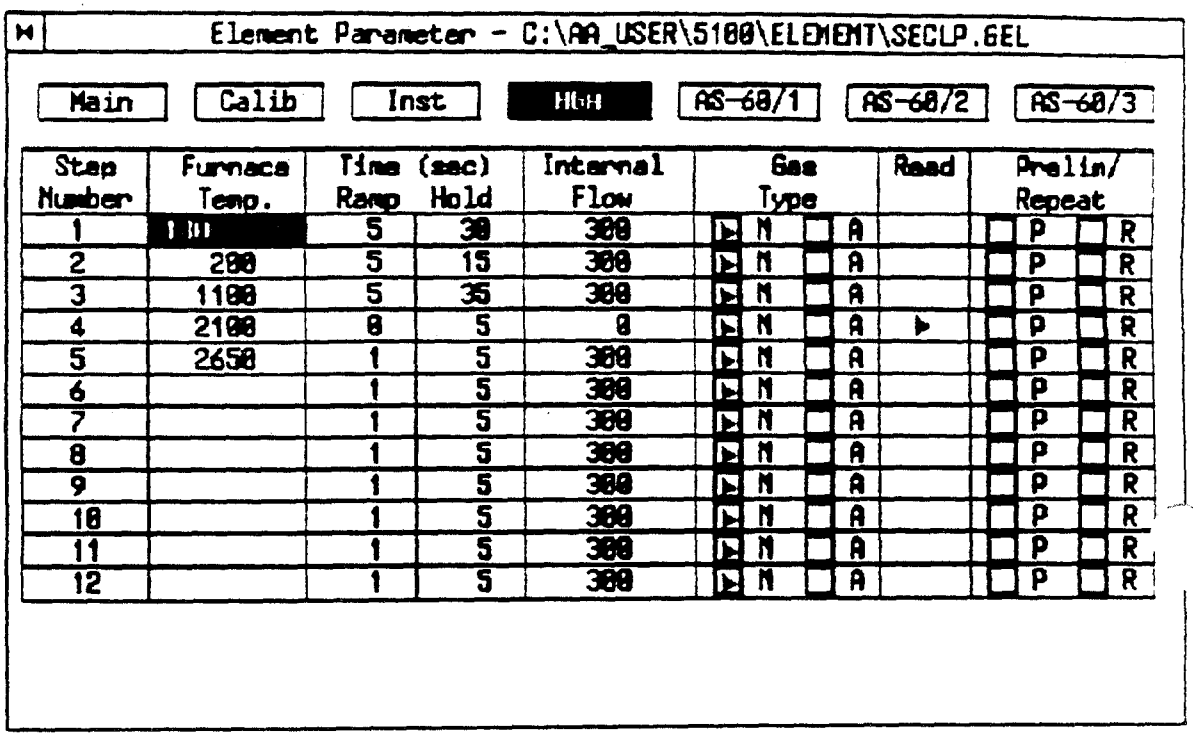
301143


Element Parameter - C:\AA_USER\5100\ELEMENT\ASCLP.GEL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
SAMPLE BEYOND CALIBRATION RANGE:
Beyond Calibration Action: Continue Dilute & Reanalyze
Alternate Sample Volume: Separate Overcal Blank: Yes No
RECOVERY MEASUREMENT PARAMETERS:
Use __ uL of the _____ ug/L standard at location __
The net recovery concentration will be ug/L
PERIODIC RECALIBRATION PARAMETERS:
Recalibration Type: Restandardize Reslope
Run Before Sample Locations: __ __ __ __ __ __
PERIODIC CHECK SAMPLE PARAMETERS:
Location: __ Lower Limit: _____ Upper Limit: _____
Run Before Sample Locations: __ __ __ __ __ __
Out of Limit Action: Continue Recalibrate & Continue
Stop Recalibrate & Rerun

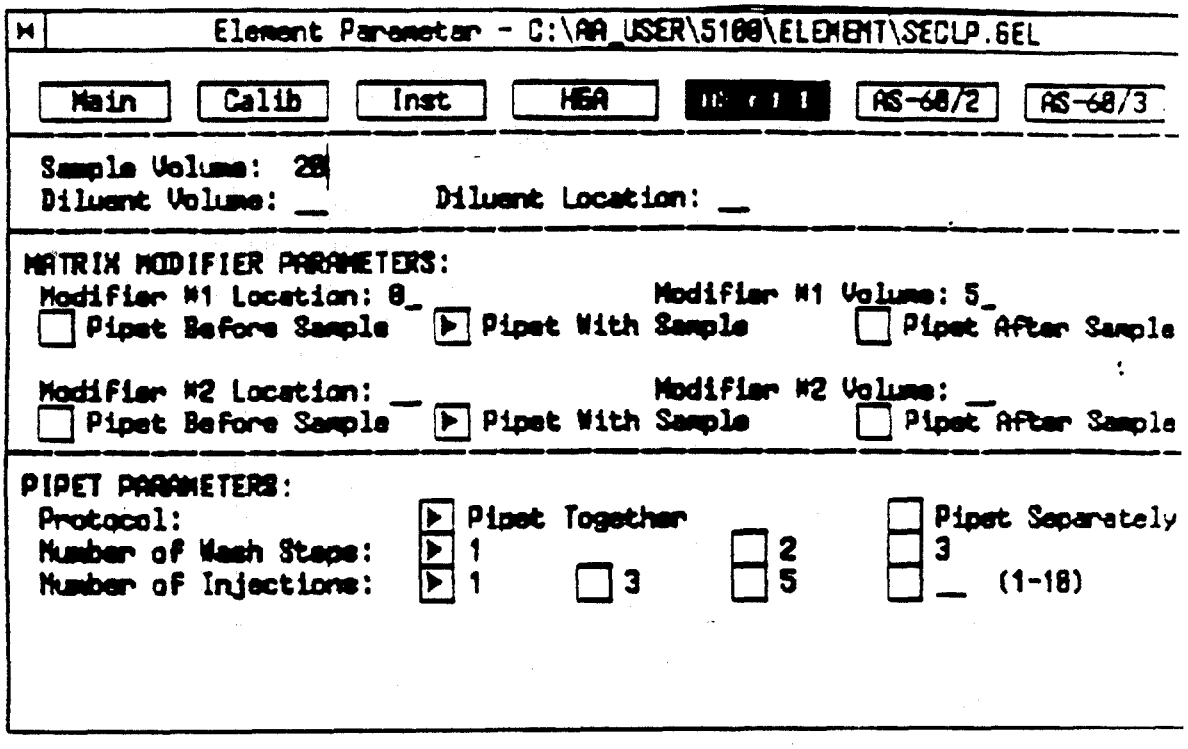

Element Parameter - C:\AA_USER\51AA\ELEMENT\SECLP.6EL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
Element: Se
Analyst: DFD
PRINTER:
Means Only
Main Data
Main+Suppl.
OPTIONS:
Calib. Curves
Peak Plots
Sampler Volumes
PEAK STORAGE:
None
All
1 Repl./Sample
REMARKS:

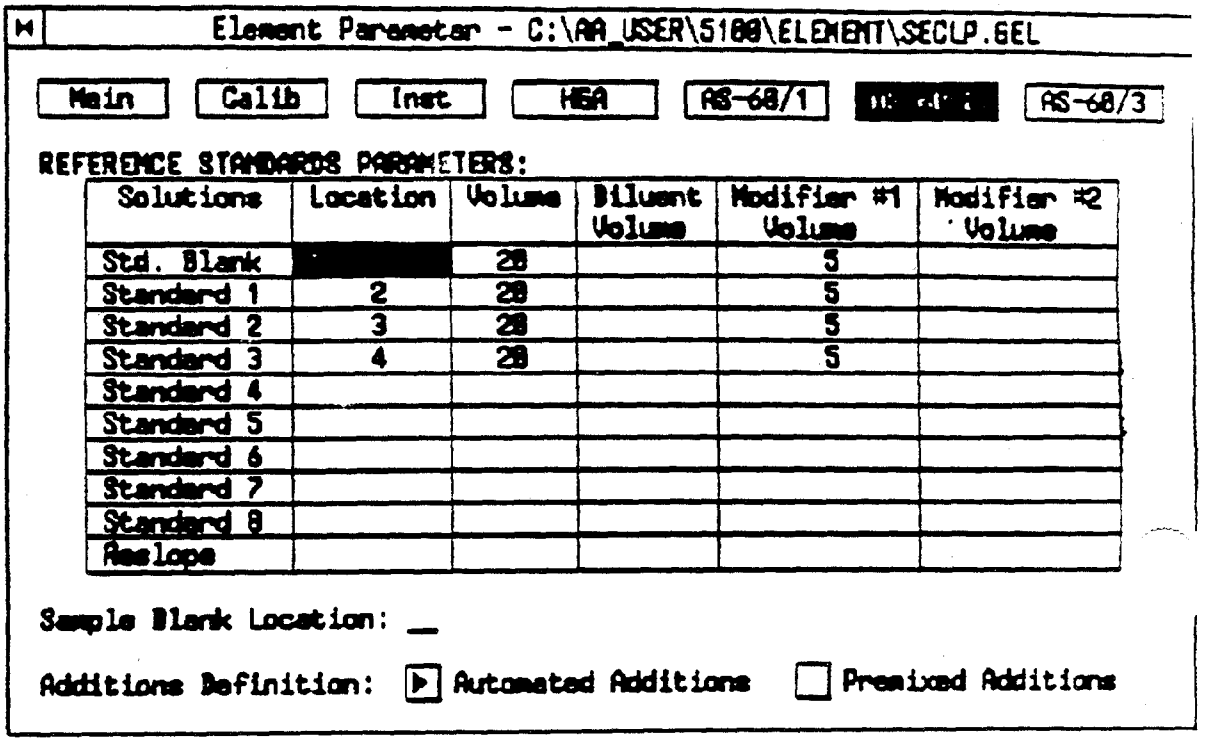

Element Parameter - C:\AA_USER\5100\ELEMENT\SECLP.GEL
Main
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
CALIBRATION TYPE:
Zero Intercept:
Nonlinear
Linear
Meth. of Add.
Add. Calib.
Calculated Intercept:
Linear
Meth. of Add.
STANDARD UNITS:
g/L
mg/L
ug/L
ng/L
pg/L
: Factor
STANDARD CONC.: S1: 5.0 S2: 50.0 S3: 100.0 S4:
S5: S6: S7: S8: Rslp:
SAMPLE UNITS:
g/L
ng/g
mg/L
wt%
ug/L
ng/L
Wt/Vol
pg/L
Wt/Wt
mg/g
ug/g
: Factor
REPLICATES:
1
3
5
2_ (1-99)
If conc <
use _,
If between
&
use _,
If conc >
use _,
else use _ (1-99)


Element Parameter - C:\AA_USER\5100\ELEMENT\SECLP.GEL
Main
Calib
HGA
AS-60/1
AS-60/2
AS-60/3
INTEGRATION TIME (sec): 1 3 5 10 (1-60)
READ DELAY (sec): 0 2 5 (0-20)
WAVELENGTH (nm): 196.0 (196-860)
Slew
Peak
Rollover (Abs.):
SLIT (nm): 0.02 0.07 0.20 0.70 2.00
BOC TIME (sec): 1 2 3 4 5
MEASUREMENT TYPE: Peak Area Peak Height
PEAK HEIGHT SMOOTHING (points): None 5 9 19 37


Element Parameter - C:\AA_USER\5100\ELEMENT\SECLP.GEL
Main
Calib
Inst
HGH
AS-60/1
AS-60/2
AS-60/3
Step Number
Furnace Temp.
Time (sec) Ramp
Hold
Internal Flow
Gas Type
Read
Prelim/ Repeat
1 100 5 30 300 N A P R
2 200 5 15 300 N A P R
3 1100 5 35 300 N A P R
4 2100 0 5 0 N A P R
5 2650 1 5 300 N A P R
6 1 5 300 N A P R
7 1 5 300 N A P R
8 1 5 300 N A P R
9 1 5 300 N A P R
10 1 5 300 N A P R
11 1 5 300 N A P R
12 1 5 300 N A P R

301148


Element Parameter - C:\AA_USER\5100\ELEMENT\SECLP.6EL
Main
Calib
Inst
HGA
AS-60/2
AS-60/3
Sample Volume: 20
Diluent Volume: __
Diluent Location: __
MATRIX MODIFIER PARAMETERS:
Modifier M1 Location: 0_
Modifier M1 Volume: 5_
Pipet Before Sample
Pipet With Sample
Pipet After Sample
Modifier M2 Location: __
Modifier M2 Volume: __
Pipet Before Sample
Pipet With Sample
Pipet After Sample
PIPET PARAMETERS:
Protocol:
Pipet Together
Pipet Separately
Number of Wash Steps:
1
2
3
Number of Injections:
1
3
5
__ (1-18)


Element Parameter - C:\AA_USER\5100\ELEMENT\SECLP.GEL
Main
Calib
Inst
HGA
AS-60/1
AS-60/3
REFERENCE STANDARDS PARAMETERS:
Solutions
Location
Volume
Diluent Volume
Modifier #1 Volume
Modifier #2 Volume
Std. Blank
20
5
Standard 1
2
20
5
Standard 2
3
20
5
Standard 3
4
20
5
Standard 4
Standard 5
Standard 6
Standard 7
Standard 8
Reslope
Sample Blank Location: _
Additions Definition:
Automated Additions
Premixed Additions

301150

Element Parameter - C:\AA_USER\5100\ELEMENT\SECLP.GEL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | [illegible]

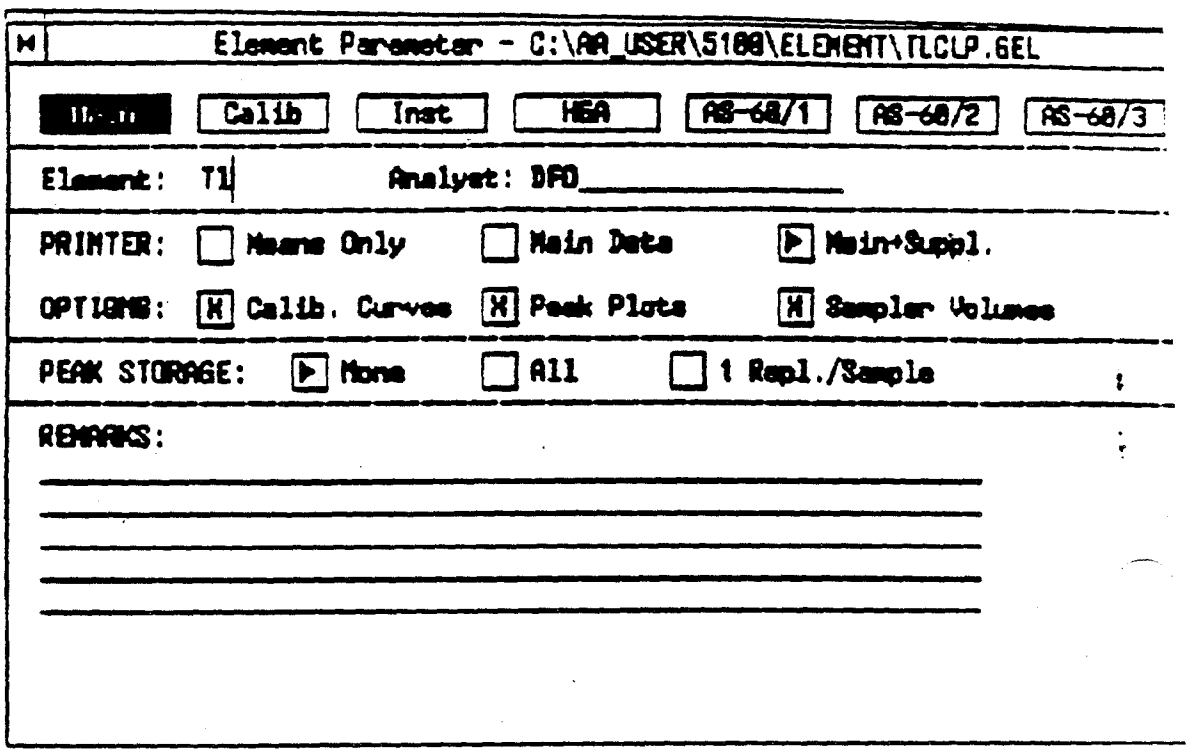
SAMPLE BEYOND CALIBRATION RANGE:
Beyond Calibration Action: ▶ Continue ☐ Dilute & Reanalyze
Alternate Sample Volume: __ Separate Overcal Blank: ☐ Yes ▶ No

RECOVERY MEASUREMENT PARAMETERS:
Use __ uL of the _____ ug/L standard at location __
The net recovery concentration will be ug/L

PERIODIC RECALIBRATION PARAMETERS:
Recalibration Type: ▶ Restandardize ☐ Reslope
Run Before Sample Locations: __ __ __ __ __ __

PERIODIC CHECK SAMPLE PARAMETERS:
Location: __ Lower Limit: _____ Upper Limit: _____
Run Before Sample Locations: __ __ __ __ __ __
Out of Limit Action: ▶ Continue ☐ Stop ☐ Recalibrate & Continue ☐ Recalibrate & Rerun

301151


Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.GEL
Calib
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
Element: Tl
Analyst: DFD
PRINTER:
Means Only
Main Data
Main+Suppl.
OPTIONS:
Calib. Curves
Peak Plots
Sampler Volumes
PEAK STORAGE:
None
All
1 Repl./Sample
REMARKS:

Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.6EL

Main | ... nl | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

CALIBRATION TYPE:

Zero Intercept:
- [▶] Nonlinear
- [ ] Linear
- [ ] Meth. of Add.
- [ ] Add. Calib.

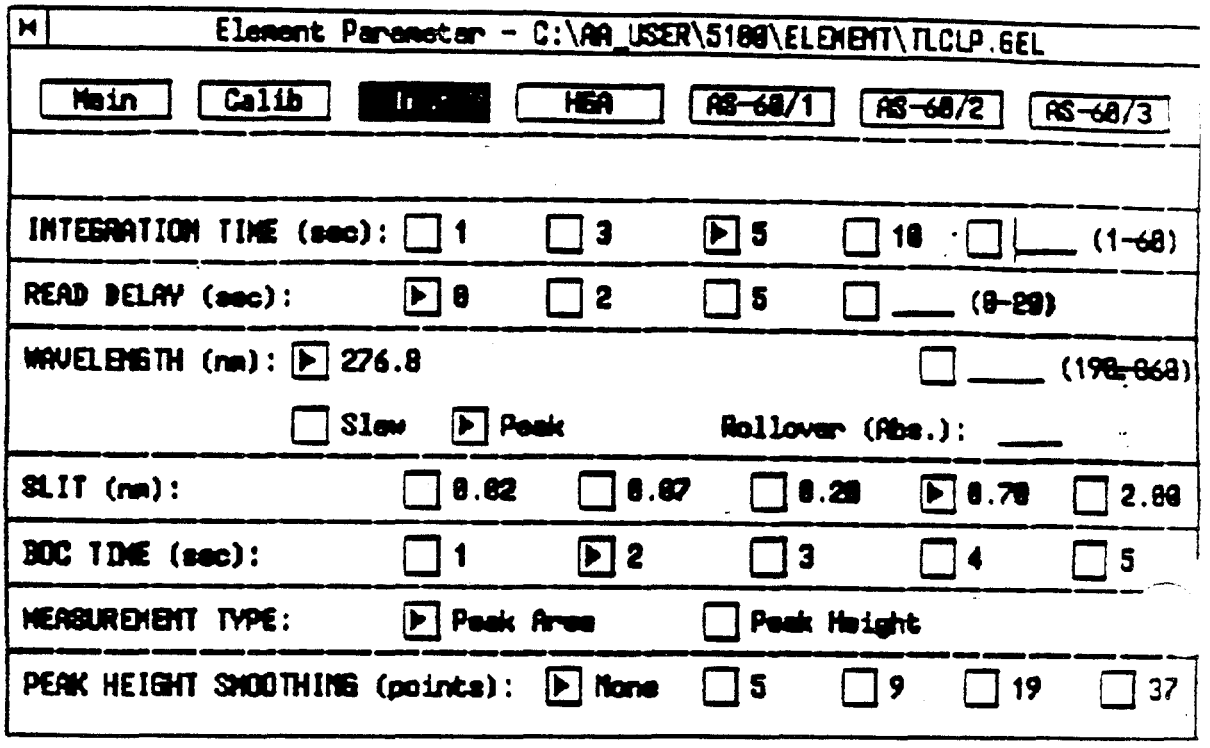
Calculated Intercept:
- [ ] Linear
- [ ] Meth. of Add.

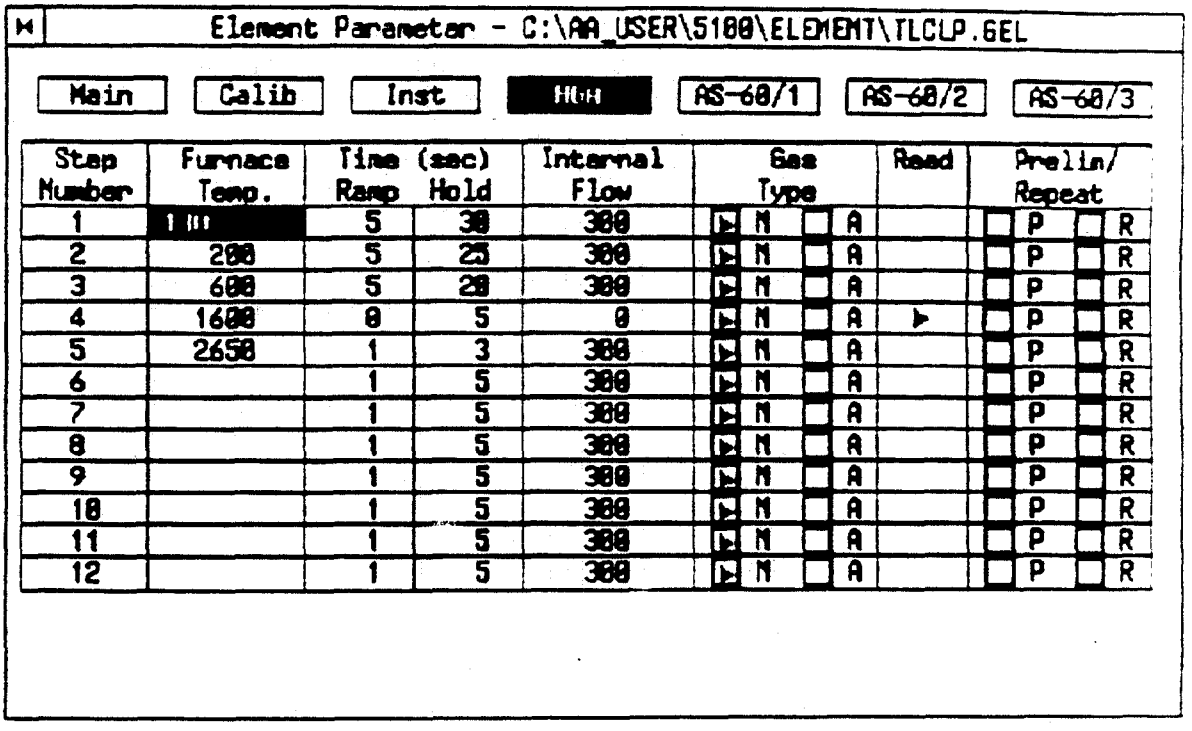
STANDARD UNITS:
[ ] g/L [ ] mg/L [▶] ug/L [ ] ng/L [ ] pg/L [ ] ____ : Factor _____

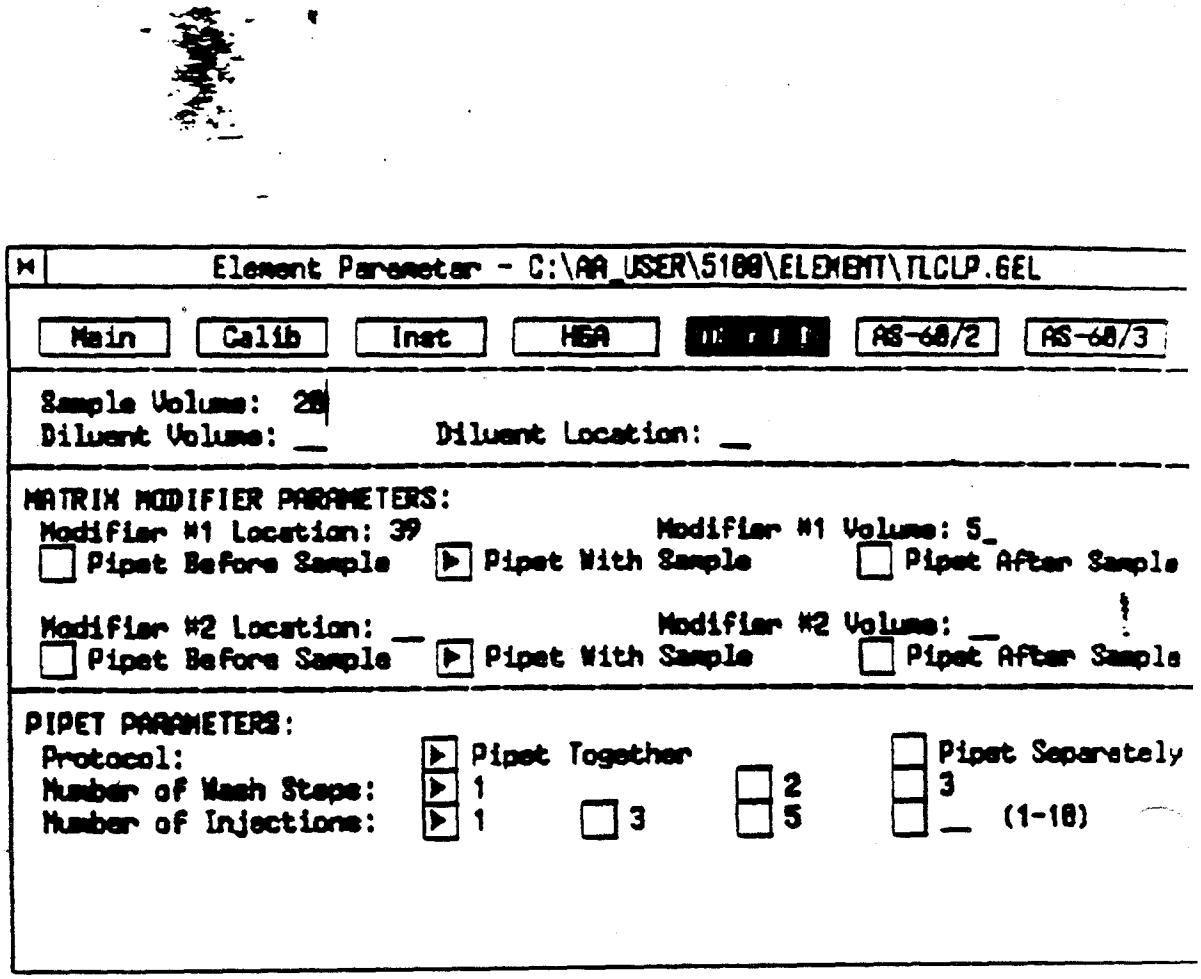
STANDARD CONC.: S1: 10.0____ S2: 50.0____ S3: 100.0____ S4: _____
S5: _____ S6: _____ S7: _____ S8: _____ Rslp: _____

SAMPLE UNITS:
[ ] g/L [ ] mg/L [▶] ug/L [ ] ng/L [ ] pg/L [ ] mg/g [ ] ug/g
[ ] ng/g [ ] wt% [ ] _____ : [▶] Wt/Vol [ ] Wt/Wt : Factor _____

REPLICATES: [ ] 1 [ ] 3 [ ] 5 [▶] 2_ (1-99)
[ ] If conc < _____ use __, If between _____ & _____ use __,
If conc > _____ use __, else use __ (1-99)


Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.6EL
Main
Calib
HGA
AS-60/1
AS-60/2
AS-60/3
INTEGRATION TIME (sec): 1 3 5 10 (1-60)
READ DELAY (sec): 0 2 5 (0-20)
WAVELENGTH (nm): 276.8 (190-860)
Slew
Peak
Rollover (Abs.):
SLIT (nm): 0.02 0.07 0.20 0.70 2.00
BOC TIME (sec): 1 2 3 4 5
MEASUREMENT TYPE: Peak Area Peak Height
PEAK HEIGHT SMOOTHING (points): None 5 9 19 37


Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.GEL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
Step Number
Furnace Temp.
Time (sec) Ramp
Hold
Internal Flow
Gas Type
Read
Prelim/ Repeat
1 100 5 30 300 M A P R
2 200 5 25 300 M A P R
3 600 5 20 300 M A P R
4 1600 0 5 0 M A P R
5 2650 1 3 300 M A P R
6 1 5 300 M A P R
7 1 5 300 M A P R
8 1 5 300 M A P R
9 1 5 300 M A P R
10 1 5 300 M A P R
11 1 5 300 M A P R
12 1 5 300 M A P R


Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.GEL
Main
Calib
Inst
HGA
AS-60/2
AS-60/3
Sample Volume: 20
Diluent Volume: _
Diluent Location: _
MATRIX MODIFIER PARAMETERS:
Modifier #1 Location: 39
Modifier #1 Volume: 5_
Pipet Before Sample
Pipet With Sample
Pipet After Sample
Modifier #2 Location: _
Modifier #2 Volume: _
Pipet Before Sample
Pipet With Sample
Pipet After Sample
PIPET PARAMETERS:
Protocol:
Pipet Together
Pipet Separately
Number of Wash Steps:
1
2
3
Number of Injections:
1
3
5
_ (1-10)

Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.GEL

Main | Calib | Inst | HGA | AS-60/1 | [illegible] | AS-60/3

REFERENCE STANDARDS PARAMETERS:

Solutions	Location	Volume	Diluent Volume	Modifier #1 Volume	Modifier #2 Volume
Std. Blank		20		5	
Standard 1	2	20		5	
Standard 2	3	20		5	
Standard 3	4	20		5	
Standard 4					
Standard 5					
Standard 6					
Standard 7					
Standard 8					
Reslope					

Sample Blank Location: _

Additions Definition: [▶] Automated Additions [ ] Premixed Additions

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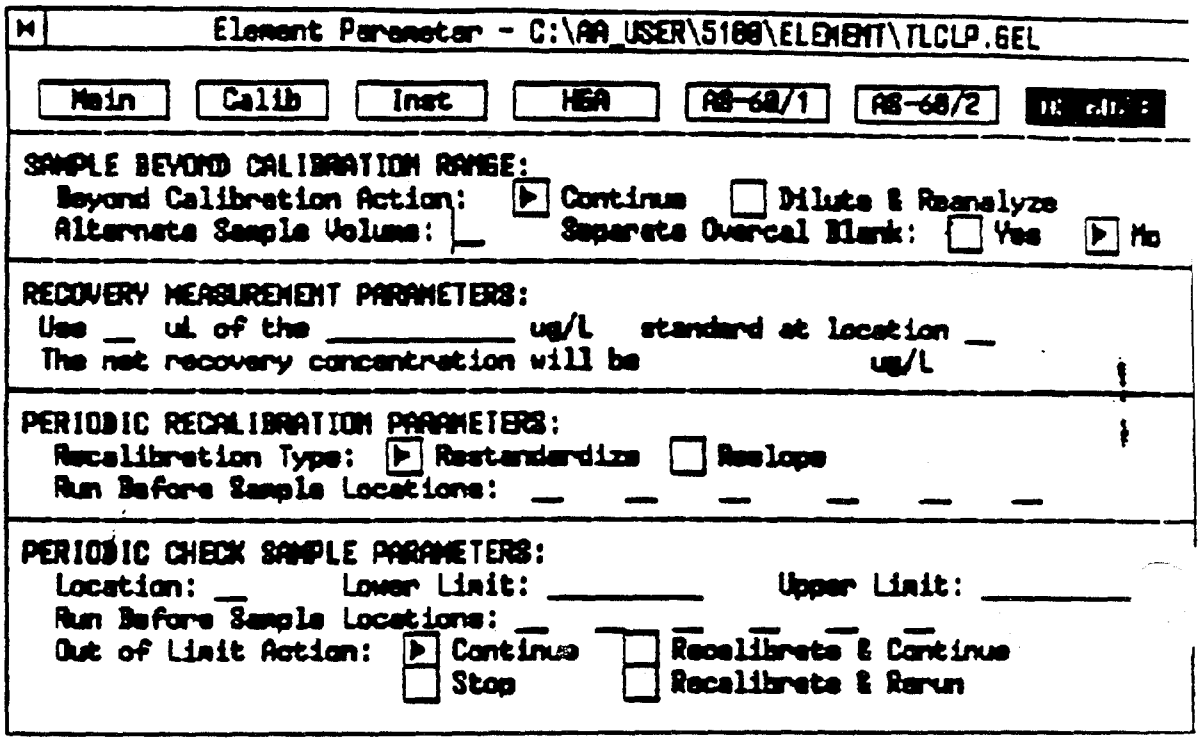
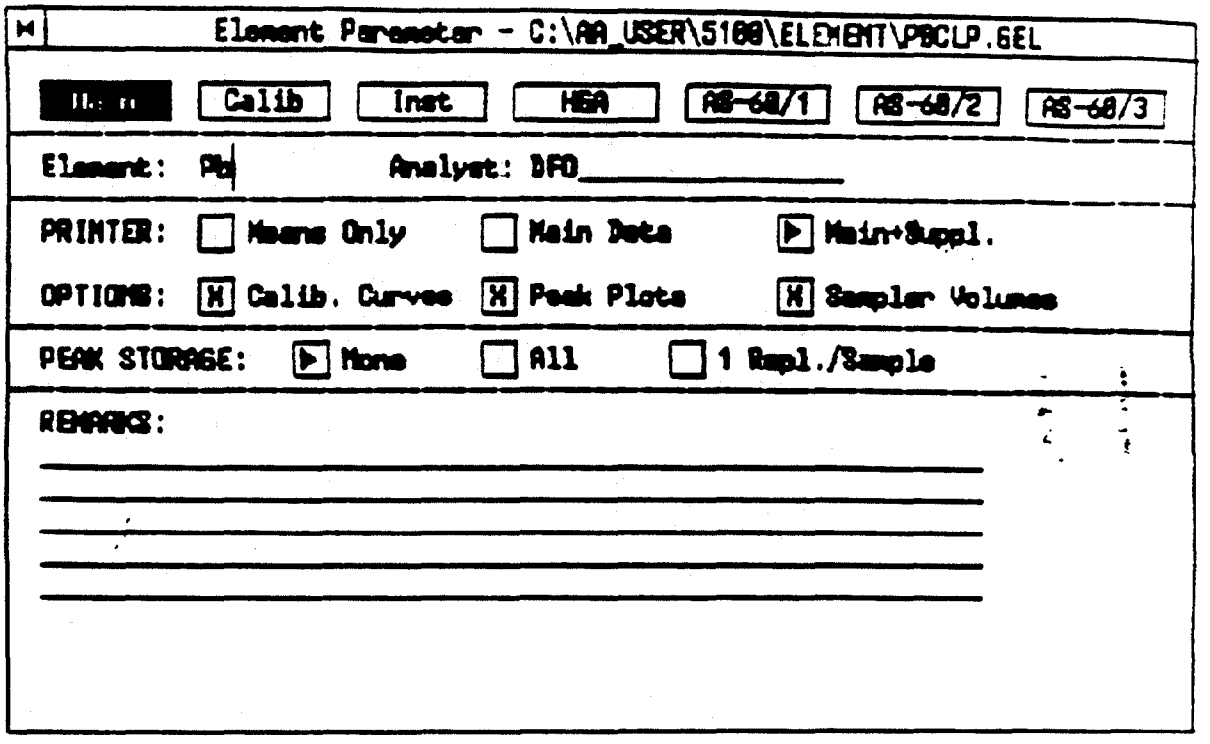

Element Parameter - C:\AA_USER\5100\ELEMENT\TLCLP.GEL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
SAMPLE BEYOND CALIBRATION RANGE:
Beyond Calibration Action: Continue Dilute & Reanalyze
Alternate Sample Volume:
Separate Overcal Blank: Yes No
RECOVERY MEASUREMENT PARAMETERS:
Use __ ul of the _____ ug/l standard at location __
The net recovery concentration will be ug/l
PERIODIC RECALIBRATION PARAMETERS:
Recalibration Type: Restandardize Reslope
Run Before Sample Locations: __ __ __ __ __ __
PERIODIC CHECK SAMPLE PARAMETERS:
Location: __ Lower Limit: _____ Upper Limit: _____
Run Before Sample Locations: __ __ __ __ __ __
Out of Limit Action: Continue Recalibrate & Continue
Stop Recalibrate & Rerun


Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.6EL
Main
Calib
Inst
HGA
AS-60/1
AS-60/2
AS-60/3
Element: Pb
Analyst: DFD
PRINTER:
Means Only
Main Data
Main+Suppl.
OPTIONS:
Calib. Curves
Peak Plots
Sampler Volumes
PEAK STORAGE:
None
All
1 Repl./Sample
REMARKS:

Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.GEL

Main | [redacted] | Inst | HGA | AS-60/1 | AS-60/2 | AS-60/3

CALIBRATION TYPE:

Zero Intercept:
- [x] Nonlinear
- [ ] Linear
- [ ] Meth. of Add.
- [ ] Add. Calib.

Calculated Intercept:
- [ ] Linear
- [ ] Meth. of Add.

STANDARD UNITS:
- [ ] g/L
- [ ] mg/L
- [x] ug/L
- [ ] ng/L
- [ ] pg/L
- [ ] ____ : Factor ____

STANDARD CONC.: S1: 3.0____ S2: 50.0____ S3: 100.0____ S4: ____
S5: ____ S6: ____ S7: ____ S8: ____ Rslp: ____

SAMPLE UNITS:
- [ ] g/L
- [ ] ng/g
- [ ] mg/L
- [ ] wt%
- [x] ug/L
- [ ] ____ :
- [ ] ng/L
- [x] Wt/Vol
- [ ] pg/L
- [ ] Wt/Wt
- [ ] mg/g
- [ ] ug/g
: Factor ____

REPLICATES:
- [ ] 1
- [ ] 3
- [ ] 5
- [x] 2_ (1-99)

- [ ] If conc < ____ use __, If between ____ & ____ use __,
If conc > ____ use __, else use __ (1-99)

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Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.GEL

Main | Calib | [illegible] | HGA | AS-60/1 | AS-60/2 | AS-60/3

INTEGRATION TIME (sec): ☐ 1 ☐ 3 ☑ 5 ☐ 10 ☐ ___ (1-60)

READ DELAY (sec): ☑ 0 ☐ 2 ☐ 5 ☐ ___ (0-20)

WAVELENGTH (nm): ☑ 283.3 ☐ ___ (190-860)

☐ Slew ☑ Peak Rollover (Abs.): ___

SLIT (nm): ☐ 0.02 ☐ 0.07 ☐ 0.20 ☑ 0.70 ☐ 2.00

BOC TIME (sec): ☐ 1 ☑ 2 ☐ 3 ☐ 4 ☐ 5

MEASUREMENT TYPE: ☑ Peak Area ☐ Peak Height

PEAK HEIGHT SMOOTHING (points): ☑ None ☐ 5 ☐ 9 ☐ 19 ☐ 37

301161

Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.GEL

Main | Calib | Inst | HGH | AS-60/1 | AS-60/2 | AS-60/3

Step Number	Furnace Temp.	Time (sec) Ramp	Hold	Internal Flow	Gas Type	Read	Prelin/ Repeat
1	100	5	30	300	N A		P R
2	200	5	25	300	N A		P R
3	900	5	20	300	N A		P R
4	1800	0	5	0	N A	▶	P R
5	2650	1	5	300	N A		P R
6		1	5	300	N A		P R
7		1	5	300	N A		P R
8		1	5	300	N A		P R
9		1	5	300	N A		P R
10		1	5	300	N A		P R
11		1	5	300	N A		P R
12		1	5	300	N A		P R

Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.6EL

Main | Calib | Inst | HGA | [redacted] | AS-60/2 | AS-60/3

Sample Volume: 20
Diluent Volume: __ Diluent Location: __

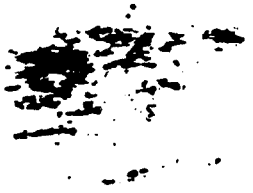
MATRIX MODIFIER PARAMETERS:

Modifier #1 Location: 40 Modifier #1 Volume: 5_
☐ Pipet Before Sample ▶ Pipet With Sample ☐ Pipet After Sample

Modifier #2 Location: __ Modifier #2 Volume: __
☐ Pipet Before Sample ▶ Pipet With Sample ☐ Pipet After Sample

PIPET PARAMETERS:

Protocol: ▶ Pipet Together ☐ Pipet Separately
Number of Wash Steps: ▶ 1 ☐ 2 ☐ 3
Number of Injections: ▶ 1 ☐ 3 ☐ 5 ☐ __ (1-10)



301163

Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.GEL

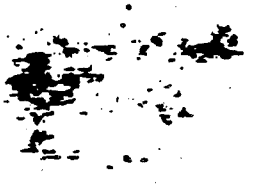
Main | Calib | Inst | HGA | AS-60/1 | [redacted] | AS-60/3

REFERENCE STANDARDS PARAMETERS:

Solutions	Location	Volume	Diluent Volume	Modifier #1 Volume	Modifier #2 Volume
Std. Blank	[redacted]	20		5	
Standard 1	2	20		5	
Standard 2	3	20		5	
Standard 3	4	20		5	
Standard 4					
Standard 5					
Standard 6					
Standard 7					
Standard 8					
Blanks					

Sample Blank Location: _

Additions Definition: ▶ Automated Additions ☐ Premixed Additions



Element Parameter - C:\AA_USER\5100\ELEMENT\PBCLP.6EL

Main | Calib | Inst | HGA | AS-60/1 | AS-60/2 | [redacted]

SAMPLE BEYOND CALIBRATION RANGE:
Beyond Calibration Action: ▶ Continue ☐ Dilute & Reanalyze
Alternate Sample Volume: __ Separate Overcal Blank: ☐ Yes ▶ No

RECOVERY MEASUREMENT PARAMETERS:
Use __ ul of the _____ ug/L standard at location __
The net recovery concentration will be ug/L

PERIODIC RECALIBRATION PARAMETERS:
Recalibration Type: ▶ Restandardize ☐ Reslope
Run Before Sample Locations: __ __ __ __ __ __

PERIODIC CHECK SAMPLE PARAMETERS:
Location: __ Lower Limit: _____ Upper Limit: _____
Run Before Sample Locations: __ __ __ __ __ __
Out of Limit Action: ☐ Continue ☐ Recalibrate & Continue
☐ Stop ▶ Recalibrate & Rerun

APPENDIX A

APPENDIX A

Stock Standard Solution - Pipette 1.0 mL each of 1,000 mg/L (ppm) of arsenic, selenium, thallium, and lead standards into a 200 mL volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 5,000 ppb of each element.

CRA Stock Standard Solution - Pipette 1.0 mL each of 1,000 mg/L (ppm) arsenic and thallium, 0.5 mL of 1,000 mg/L (ppm) selenium and 0.3 mL of 1,000 mg/L (ppm) lead into a 200 ml volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 5,000 ppb arsenic and thallium, 2,500 ppb/selenium and 1,500 ppb lead.

Analytical Spike Solution - Pipette 0.4 mL each 1,000 mg/L (ppm) of arsenic, thallium, and lead and 0.2 mL of 1,000 mg/L (ppm) selenium into a 200 mL volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 2,000 ppb arsenic, thalium, and lead, 1,000 ppb selenium.

In-house Continuing Calibration Verification Solution - Pipette 1.0 mL each of 1,000 mg/L (ppm) arsenic, selenium, thallium, and lead into a 200 ml volumetric flask. Bring to volume with 0.5 percent nitric acid. This solution contains 5,000 ppb for all elements.

STOCK STANDARD SOLUTIONS

Reference	Elements	Stock ID	mg/L	Vol.	Acid	Date	Init.
SQL	As, Se, Tl, Pb	ICP-P-66,65,64,63,66	1.0	200	HNO_3 1:9	6/22/89	
10	As	ICP-P-0070	1.0	200	HNO_3 2%	10/20/89	JC
10	Tl	ICP-P-0041	1.0				
10	Se	ICP-P-0057	1.0				
10	Pb	ICP-P-0066	1.0				
SQL	As, Se, Tl, Pb	ICP-P-0070, 41, 57, 66	1.0			10/20/89	JC
11	As, Tl	ICP-P-0070, 41	1.0			10/20/89	JC
11	Se	ICP-P-57	0.5				
11	Pb	ICP-P-0066	0.3	↓	↓	↓	↓
10	As	ICP-P-0070	1.0	200	HNO_3 2%	10/20/89	JC
10	Tl	ICP-P-0041	1.0				
10	Se	ICP-P-0057	1.0				
10	Pb	ICP-P-0066	1.0				
SQL	As, Se, Tl, Pb	ICP-P-0070, 41, 57, 66	1.0			10/30/89	JC
11	As, Tl	ICP-P-0070, 41	1.0			10/30/89	JC
11	Se	ICP-P-57	0.5				
11	Pb	ICP-P-66	0.3	↓	↓	↓	↓
SQL	As, Se, Tl, Pb	ICP-P-0070, 41, 57, 66	1.0	200	HNO_3 2%	11/07/89	JC
10	As	ICP-P-0064	1.0	200	HNO_3 2%	11/08/89	JC
10	Tl	ICP-P-0065	1.0				
10	Se	ICP-P-0063	1.0				
10	Pb	ICP-P-0066	1.0				
SQL	As, Se, Tl, Pb	ICP-P-64,65, 63,66	1.0			11/10/89	JC
11	As, Tl	ICP-P-64,65	1.0			11/10/89	JC
11	Se	ICP-P-63	0.5				
11	Pb	ICP-P-66	0.3	↓	↓	↓	↓
SQL	As, Se, Tl, Pb	ICP-P-65, 64, 63, 66	1	200	HNO_3 2%	12/[illegible]/89	JC
10	As	ICP-P-0064	1			12/[illegible]/89	JC
10	Se	ICP-P-0063	1				
10	Tl	ICP-P-0065	1				
10	Pb	ICP-P-0066	1				
11	As, Tl	ICP-P-0064, 0065	1			12/[illegible]/89	JC
11	Se	ICP-P-0063	0.5				
11	Pb	ICP-P-0066	0.3	↓	↓	↓	↓

301168

APPENDIX B

APPENDIX B

Working Standard Solutions 100 ppb Standard - Pipette 4.0 of the stock standard solution into a 200 ml volumetric flask and bring to volume with 0.5 percent nitric acid.

50 ppb Standard - Pipette 2.0 mL of the stock standard solution into a 200 mL volumetric flask and bring to volume with 0.5 percent nitric acid.

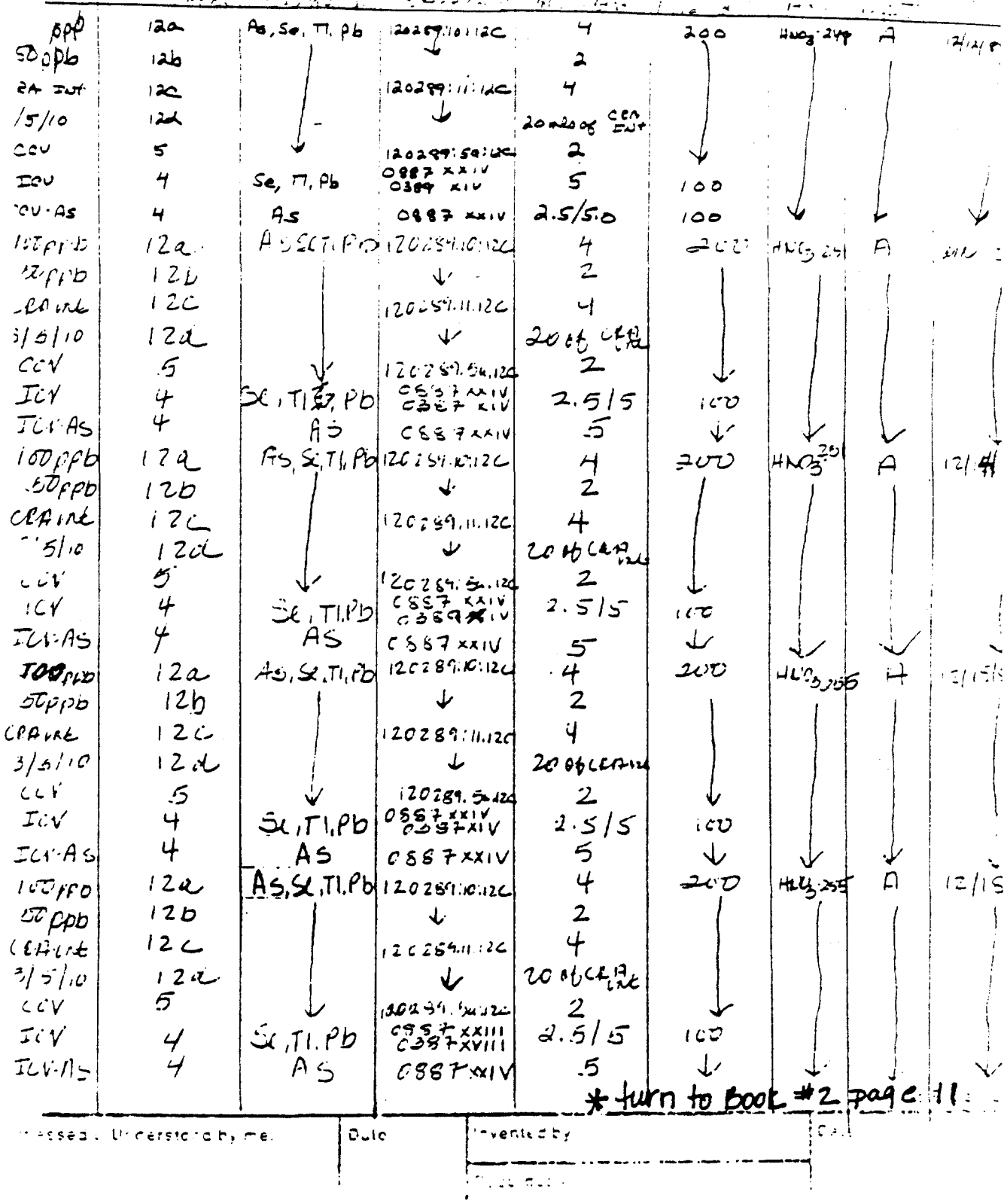
Intermediate CRA Solution - Pipette 4.0 mL of the stock CRA solution into a 200 mL volumetric flask and bring to volume with 0.5% nitric acid.

CRA Solution - 10.0 ppb As, Tl, 5.0 ppb Se, 3.0 ppb Pb - Pipette 20.0 mL of the intermediate CRA solution into a 200 mL volumetric flask and bring to volume with 0.5% nitric acid.

Blank Solution - Add 0.5 nitric acid to 100 mL nanopure water.

In-house Continuing Calibration Verification Solution (CCU) - Pipette 2.0 mL of the stock CCU solution into a 200 mL volumetric flask and bring to volume with 5 percent nitric acid. This solution contains 50.0 ppb all elements.

Appendix 5
Working Standard Solutions



Witnessed, understood by me.

Date

Invented by

Date

301171

APPENDIX C

703 716 0515

Appendix C
EPA ICV Solution

ICF TECHNOLOGY INCORPORATED

October 13, 1989

INITIAL CALIBRATION VERIFICATION SOLUTIONS FOR THE
CONTRACT LABORATORY PROGRAM
EPA-EMSL AND ICF TECHNOLOGY, INC.

The Initial Calibration Verification Solutions (ICVs) are to be used to evaluate the accuracy of the initial calibrations of ICP and AA instruments. These solutions may also be used as aqueous Laboratory Control Solutions.

The values for each element in the ICVs are listed below in ug/L (ppb) for the solutions that result after the concentrates have been diluted according to the following instructions:

1. ICV-1 (0489) -- For ICP use, dilute the ICV-1 concentrate 10-fold with 2% (v/v) nitric acid; pipet 10 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

2. ICV-2 (0989) -- For furnace AA use, dilute the ICV-2 concentrate 20-fold with 2% (v/v) nitric acid; pipet 5 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

3. ICV-3 (0189) -- For ICP use, dilute the ICV-3 concentrate 10-fold with 2% (v/v) nitric acid; pipet 10 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid. For furnace AA use, dilute the ICV-3 concentrate 100-fold with 2% (v/v) nitric acid; pipet 1 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

4. ICV-4 (0389) -- For the furnace AA determination of lead and thallium, dilute the ICV-4 concentrate 10-fold with 2% (v/v) nitric acid; pipet 10 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid. For the furnace AA determination of silver and cadmium, dilute the ICV-4 concentrate 100-fold with 2% (v/v) nitric acid; pipet 1 mL of the concentrate into a 100-mL volumetric flask and dilute to volume with 2% (v/v) nitric acid.

Appendix C
EPA ICU Solution

ICV-1 (0489):

(after 10-fold dilution)

Element	Value (ug/L)
Al	2010
Ba	1960
Be	483
Cd	502
Ca	48700
Cr	510
Co	502
Cu	515
Fe	2040
Pb	4800
Mg	24600
Mn	504
Ni	480
K	49000
Ag	500
Na	49600
V	488
Zn	3100

ICV-2 (0989):

(after 20-fold dilution)

Element	Value (ug/L)
As	52.6
Se	52.6

ICV-3 (0189):

(after 10-fold dilution)

Element	Value (ug/L)
Sb	978

ICV-4 (0389):

(after 10-fold dilution)

Element	Value (ug/L)
Cd	95.8
Pb	97.5
Ag	98.6
Tl	97.0

APPENDIX D

APPENDIX D

Modifier Solutions

1. Nickel Nitrate for arsenic and selenium determination. Dissolve 4.95 gm of ACS reagent grade nickel nitrate, $Ni\ (NO_3)_2 \times 6H_2O$ in 100 mL nanopure water.

2. Ammonium monobasic phosphate, magnesium nitrate for lead determinations. Dissolve 4.0 gm ACS reagent grade $(NH_4)_2H_2PO_4$ and 0.2 gm $Mg\ (NH_3)_2 \times 6H_2O$ in 100 mL nanopure water.

3. One percent Sulfuric Acid for thallium determinations. Add 1.0 mL concentrated sulfuric acid to 99.0 mL nanopure water.

MODIFIERS SOLUTIONS

Compound	Concentration	Your Stock #	Amount Used	Final Volume	Code	Initials
[illegible]	5	mix KMDA	4gm	100	031390M	JFO
$Mg(NO_3)_2 \cdot 6H_2O$	5	Fisher 724199	0.2gm	100	041389M	JFO
$Ni(NO_3)_2 \cdot 6H_2O$	7	N04999	4.9g	100	032189M	JFO ET
[illegible]	6	Fisher 724199	2g	200	032389M	JFO
[illegible]	7	N04999	9.8gm	200	032389M	JFO
$Ni(NO_3)_2 \cdot 6H_2O$	7	mx 6384 KXXB	4.9gm	100 ml	033089M	JFO 3/2
H_2SO_4	9	H_2SO_4 #3	5ml	500ml	033089M	JFO 3/2
$Ni(NO_3)_2 \cdot 6H_2O$	7	N04999	4.9gm	100	041889M	041889/M
$(NH_4)H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4gm, 0.2gm	100	050189M	050189
$Ni(NO_3)_2 \cdot 6H_2O$	7	Fisher 724199	4.92gm	100	050389M	050389/M
$(NH_4)H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4gm, 0.2gm	100	053089M	053089/M
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.92 gm	100	060589M	060589/M
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4gm, 0.2gm	100	060889	060889/M
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	4gm / 0.2gm	100	061989	061989 JM
H_2SO_4	9	H_2SO_4 #	~~5ml~~ 2ml	~~500~~ 200	061989	061989 JM
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.90 gm	100	061989	061989 JM
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	490 gm	100	032690	032690 JM
$(NH_4)H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	(4gm/0.2gm)x2	200	032690	032690 JM
H_2SO_4	9	H_2SO_4 #37	5 ml	500	041090	041090 JM
$Ni(NO_3)_6 \cdot 6H_2O$	7	Fisher 724199	4.90	100	041090	41090 JM
$(NH_4)_2H_2PO_4 \cdot MgNO_3$	8	Fisher 72199	(4gm/0.2gm) x 4	400	041090	41090 JM

301177

APPENDIX E

Appendix E

Daily Sensitivity Check

Mo = 15.0

Sens: 35 ppb = 0.130

Date	Lamp	Energy	Blank	10.0ppb	50.0ppb	100.0ppb	150.0ppb
11/6/89			0.051	0.052	0.278	0.548	
11/16/89			-0.003	0.063	0.297	0.575	
11/20/89			0.002	0.055	0.280	0.560	
			-0.004	0.053	0.263	0.495	
12/1/89			-0.002	0.058	0.273	0.540	
12/2/89			0.004	0.049	0.247	0.498	
12/4/89			0.001	0.054	0.265	0.513	
12/5/89			-0.000	0.052	0.256	0.484	
12/11/89			-0.001	0.048	0.253	0.526	
12/12/89			0.002	0.054	0.270	0.536	
12/13/89			0.001	0.054	0.275	0.521	
12/14/89			0.000	0.047	0.264	0.517	
12/14/89			0.001	0.050	0.267	0.522	
12/13/89			-0.001	0.053	0.264	0.518	
12/14/89			-0.001	0.057	0.271	0.517	
12/14/89			0.001	0.055	0.255	0.554	
12/14/89			-0.001	0.052	0.240	0.481	
12/21/89			-0.002	0.051	0256	0.493	
12/22/89			-0.002	0.056	0.275	0.536	
12/27/89			0.005	0.049	0.234	0.480	
12/28/89			0.001	0.051	0.256	0.552	
12/29/89			0.001	0.051	0.277	0.539	
1/3/90			0.028	0.058	0.211	0.423	

[redacted]

Appendix E

Maintenance Log

Date	Initials	Hours Run	Routine Maint.	NonRoutine Maint.	QC Failure	Elements & Samples Run
4/4/90	dmc	3	T+P			
4/4/90	JTV	16	N/A			
4/5/90	dmc	16	T+P			
4/7/90	JTV	4	T+P			
4/11/90	JTV/dmc	1	T+P			
4/11/90	dmc	8	T+P			
4/12/90	JTV	4	T+P			
4/16/90	dmc/JTV	4	T+P			
4/16/90	JTV	16	T+P			As, Se, Pb
4/17/90	JTV	12	T+P			
~~4/20/90~~	~~JTV~~	2	T+P			
4/19/20	DMA	16	T+P			
4/20/90	JTV	12	T+P			
4/23/90	DMA	4	T+P			
4/23/90	dmc	10	N A			
~~4/24/90~~	~~dmc~~					
5/1/90	JTV	16	T+P			All
5/2/90	DMA	1	T+P			
5/2/90	DMA	16	N A			
5/3/90	dmc	8	T+P			TL-925
5/4/90	dmc	15	T+P			all
5/5/90	dmc	12	T+P			all
5/6/90	dmc	4	T+P			
5/6/90	dmc	6	N A			
5/7/90	dmc	6	T+P			

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an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX: (203) 268-5346

STANDARD OPERATING PROCEDURE

COLD VAPOR ATOMIC ABSORPTION (CVAA)

FOR THE

DETERMINATION OF MERCURY IN WATER

AS:021590:3

Whippany, New Jersey 201-428-8181
Schaumburg, Illinois 708-705-0740
Miramar, Florida 407-989-0928
Essex Junction, Vermont 802-878-5138
N. Billerica, Massachusetts 617-272-5212
Cary, North Carolina 919-677-0090

STANDARD OPERATING PROCEDURE
COLD VAPOR ATOMIC ABSORPTION (CVAA)
FOR THE DETERMINATION OF MERCURY IN WATER

INTRODUCTION

This procedure describes the manual cold vapor analysis of mercury in water and leachate samples.

It is the intent of this procedure to comply with the IEA business ethics policy.

Abstract

The addition of HNO_3 and H_2SO_4 serve to oxidize mercury. This is necessary as mercury in its elemental state is highly volatile. The addition of $KMnO_4$ ensures that the mercury is in its most oxidized state while the addition of $K_2O_8S_2$ ensures that all organic mercurials are oxidized. The reaction continues and is driven to completion in a heating step; it is probably most advantageous to bring the procedure to this point before letting any significant holding time occur. This is the best way to guarantee total oxidation of any mercury present in the samples. Hydroxylamine hydrochloride is added to reduce the permanganate and to rid the sample of any excess oxidizing agents; it will not, however, reduce mercury. Stannous chloride is used to reduce mercury back to its elemental state where it is easily volatilized and swept through the absorption cell for measurement.

1.0 SCOPE AND APPLICATION

1.1 Other related SOP's - Glassware Cleaning Procedure, Standard and Sample Prep Logs, Documentation SOP and Corrective Action SOP.

1.2 In addition to inorganic forms of mercury, organic mercurials may also be present. These organo-mercury compounds will not respond to the CVAA technique without being converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, and potassium persulfate has been found to give approximately 100 percent recovery when used as the oxidant with compounds only partially oxidized by potassium permanganate. Consequently, a persulfate oxidation step following the addition of permanganate has been included to guarantee that any organo-mercurials present will be oxidized to the mercuric ion prior to measurement. A heating step is required for methyl mercuric chloride when present in or spiked to a natural system. For nanopure water this heating step is not necessary.

1.3 Using a 100 mL sample, a quantitation limit of 0.2 ug/L mercury can be achieved.

2.0 SUMMARY OF METHOD

2.1 The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

3.0 INTERFERENCES

3.1 Possible interference from sulfide is elminiated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

3.2 Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

3.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 25 mL). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation of 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent (25 mL). In addition, the dead air space in the BOD bottle must be purged before the addition of stannous chloride. Both inorganic and organic mercury spikes have been quantitatively recovered from sea water using this technique.

3.4 Interference from certain volatile organic materials which will absorb at this wavelength is also possible. A preliminary run without reagents should determine if this type of interference is present. This is mentioned only to caution the analyst of the possibility; EMSL has not encountered such samples.

4.0 APPARATUS AND MATERIALS

4.1 Spectro Products, Inc. Mercury Analyzer (model HG-4)

4.2 Kipp and Zonen Strip Chart Recorder (model BD40)

4.3 KNF Neuberger, Inc. Vacuum Pump (1.5 amps)

4.4 Flowmeter

4.5 Aeration Tubing

4.6 Drying Tube

5.0 REAGENTS

5.1 Sulfuric acid, concentrated (H_2SO_4)

A) Sulfuric acid, 0.5N: Dilute 14 mL H_2SO_4 to 1 L with nanopure water.

5.2 Nitric acid, concentrated (HNO_3)

5.3 Potassium permanganate, 5 percent w/v ($KMnO_4$): Dissolve 125 g of $KMnO_4$ in 2.5 L nanopure water.

5.4 Potassium persulfate, 5 percent w/v ($K_2O_8S_2$): Dissolve 125 g of $K_2O_8S_2$ in 2.5 L nanopure water.

5.5 Hydroxylamine hydrochloride: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in 1 L nanopure water.

5.6 Stannous chloride: Dissolve 100 g stannous chloride in 1 L 0.5N H_2SO_4.

5.7 Preparation of working mercury standards (to be prepared fresh daily).

A) Standard A (10 mg/L mercury): 1 mL 1,000 mg/L mercury stock diluted to 100 mL in 1 percent HNO_3.

B) Standard B (100 ug/L mercury): 1 mL Standard A diluted to 100 mL in 1 percent HNO_3.

C) Standard C (10 ug/L mercury): 10 mL Standard B diluted to 100 mL in 1 percent HNO_3.

5.8 Record the standard preparation in the appropriate standard logbook according to the procedures outlined in the standard and sample preparation logbook SOP - AS:071587:2.

6.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

6.1 Samples should be preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection.

6.2 Holding times are according to the appropriate procedures:

A) SW846 - 28 days for water samples; 28 days after leaching for leachates

B) EPA SOW 7/88 - 26 days for water samples

C) Refer to any other applicable protocol requested by the client

7.0 PROCEDURE

7.1 The first step in sample preparation is to determine which samples to prepare, and beginning the associated worksheets. This is done by generating a LIMS printout of samples that are in-house requiring mercury analysis.

A) Type "S MTL,134,METALS" at the * prompt of any LIMS terminal to sign on to the atomic spectroscopy group account.

B) Type in "MASTER/G", then 5, then 4 on the selections that follow. Finally, type "MERCURY" to send a printout to the printer in the classical chemistry department. See Figure 3 for a detailed explanation of this printout.

C) Choose appropriate samples to prepare and fill out the run log (Figure 2) and prep sheet (Figure 4). The samples are entered on the left side of the form; standards, CCV's, CCB's, duplicates, spikes and prep blanks are on the right side of the form.

D) Remove the samples from Sample Control, taking care to sign out the samples in the appropriate log, and any other applicable chains of custody such as the New Jersey chain of custody. Actual preparation of standards and samples can now proceed.

7.2 Preparation of Standards and QA/QC Samples

A) Blank: Pour 100 mL of nanopure water into the first BOD bottle.

B) 0.5 ug/L mercury: Pour 95 mL of nanopure water and 5 mL Standard C into the next BOD bottle.

C) 1.0 ug/L mercury: Pour 99 mL of nanopure water and 1 mL Standard B into the next BOD bottle.

D) 2.0 ug/L mercury: Pour 98 mL of nanopure water and 2 mL Standard B into the next BOD bottle.

E) 5.0 ug/L mercury: Pour 90 mL of nanopure water and 10 mL Standard B into the next BOD bottle.

F) 10.0 ug/L mercury: Pour 90 mL of nanopure water and 10 mL Standard B into the next BOD bottle.

G) Initial Calibration Verification (ICV): Dilute 1 mL EPA ICV5 stock solution to 100 mL and pour into a BOD bottle.

H) Initial Calibration Blank (ICB): Pour 100 mL nanopure water into a BOD bottle.

I) Continuing Calibration Verification (CCV): Pour 95 mL of nanopure water and 5 mL Standard B into a BOD bottle.

J) Continuing Calibration Blank (CCB): Pour 100 mL of nanopure water into a BOD bottle.

K) Preparation Blank (PB): Pour 100 mL of nanopure water into a BOD bottle.

L) Spike = 1.0 ug/L: Add 1 mL Standard B to designated spike sample, except in the case of TCLP leachate spikes, which are spiked before preservation of the sample.

M) Reagents are to be added to the standards in the same fashion as samples.

7.3 Shake sample to obtain homogeneity. After shaking, no particulate matter should be adhering to the sample container. If more than one aliquot is required for preparation, the sample must be shaken before each additional aliquot is taken in order to maintain homogeneity.

7.4 Transfer a 100 mL sample aliquot to a 300 mL BOD bottle using an acid-washed 100 mL graduated cylinder pre-rinsed with sample. Exception: when analyzing EPTOX or TCLP leachates, transfer only 10 mL of sample to the BOD bottle and dilute to 100 mL with nanopure water.

7.5 Add 5 mL H_2SO_4 and 2.5 mL HNO_3 to each BOD bottle. Swirl to mix the sample with the reagents.

7.6 Add 15 mL of 5 percent $KMnO_4$, mix and let stand for 15 minutes. The purple color must persist throughout the digestion since a loss of color indicates that the oxidizing agents are being used up. If the color dissipates at this point, add another 15 mL of 5 percent $KMnO_4$ to the sample. If the purple color does not persist for at least 15 minutes, discard contents of the BOD into acid waste and

clean the BOD thoroughly. Take a new aliquot of sample at a volume ≤50 mL and dilute to 100 mL with nanopure water. Proceed with preparation from Section 7.5.

7.7 Add 8 mL of 5 percent $K_2O_8S_2$ and swirl to mix.

7.8 Autoclave samples on slow for 15 minutes at 120°C and 15 lbs pressure, following the set-up procedure stated below.

A) Fill the autoclave with water to the bottom lip of the chamber, just below where the door meets the chamber.

B) Load the autoclave with samples and close the door (maximum load - 36 samples).

C) Set the exhaust selector switch on slow.

D) Set the mixer knob to 15 minutes.

E) The timer will shut the autoclave off automatically. Wait until the autoclave has had time to release pressure and cool down to approximately 60°C (about 45 minutes). Carefully open the door and unload the autoclave. (NOTE: Use high temperature gloves.)

F) Drain the autoclave and fill in the autoclave logbook as shown in Figure 5.

7.9 Set up the mercury analyzer as described below:

A) Turn the instrument power on.

B) Adjust lamp B to 100 percent absorbance.

C) Wait approximately 5 minutes for the instrument to stabilize.

D) Readjust lamp B to 100 percent absorbance.

E) Turn the AGC on; instrument should read 100 percent absorbance.

F) Adjust lamp A to 0 percent absorbance.

G) Allow the lamps to warm up for at least 45 minutes.

H) Set up air flow system (see Figure 1).

I) Readjust lamp A to 0 percent absorbance.

J) Turn the strip chart recorder on.

K) Adjust the damping as necessary.

L) Turn the air flow system on. The flow meter should read between 3 and 4.

M) Allow the system to equilibrate, usually about 5 minutes.

7.10 Add 6 mL hydroxylamine hydrochloride to each sample and swirl to mix.

7.11 As chlorine is known to interfere with this procedure, the analyst is instructed to sparge the sample after the hydroxylamine has been added and immediately before the addition of 6 mL of stannous chloride. If chlorine is present, it will be swept through the cell at this point, causing a sharp peak response. Sparge until no further response is noted and then proceed with the addition of stannous chloride. Note: It may not be possible to entirely clear the sample of such interferences. If this is the case, the sample must be prepared at a dilution and rerun.

7.12 Immediately after the addition of stannous chloride, place the sparger in the sample to be analyzed, creating a closed system. Should no peak occur, remove sparger after approximately 15 seconds. If peak occurs, remove sparger after peak has reached its maximum height.

7.13 Repeat steps 7.9 - 7.11 for each standard and sample, following the analytical sequence outlined in Figure 2.

7.14 After analysis is complete, shut the system down.

A) Turn the air flow system off.

B) Turn the strip chart recorder off.

C) Turn the AGC off.

D) Center damping and zero set controls.

E) Turn lamps A and B off.

F) Turn instrument power off.

G) Disassemble the air flow system.

H) Wipe out the hood.

I) Clean out the BOD bottles using the procedure outlined in the glassware cleaning SOP.

7.15 Maintenance logs are kept for recording of routine and non-routine maintenance performed on the instrument.

A) Tubes are changed and repacked after each analytical run that exceeds 30 minutes in duration.

B) The absorption cell is cleaned about once a month using 1:1 HNO_3 and nanopure water.

C) Lamps are changed when necessary - refer to manufacturer's instructions.

8.0 QUALITY CONTROL

8.1 The calibration curve is composed of a blank and five standards: 0.5 ug/L mercury, 1.0 ug/L mercury, 2.0 ug/L mercury, 5.0 ug/L mercury and 10.0 ug/L mercury.

8.2 Samples are to be diluted if they are more concentrated than the highest standard or if they fall between the 5.0 and the 10.0 standards of the curve.

8.3 Calibration is verified initially with an independently prepared check standard (see ICV, section 7.2 G) and blank (see ICB, section 7.2 H).

8.4 Calibration is verified throughout the course of analysis by running check standards and blanks every 10 samples (see CCV, section 7.2 I and CCV, section 7.2 J). In the event the continuing calibration fails, the samples prior to and following the failed standard(s) must be rerun. Refer to the current SOW.

8.5 Batch QC consists of a minimum of one preparation blank (PBW), duplicate and spike per every 20 samples. Every EPA SDG must have its own PBW, duplicate and spike. If the prep blank exceeds the contract required detection limits (0.2 ug/L) then the samples associated with that prep blank must be reprepped and reanalyzed. Refer to the current SOW.

8.6 An abbreviated method of standard additions is used for the analysis of all EP and TCLP extracts (see method 7000, section 8.7, SW846).

9.0 METHOD PERFORMANCE

9.1 Instrument detection limits are determined quarterly per the current EPA contract.

9.2 Control charts are kept for tracking of accuracy (spike recoveries), and precision (RPD on the duplicate analysis).

10.0 DATA REDUCTION AND DATA HANDLING

10.1 Label all peaks on the strip chart and draw a baseline for each peak. Measure and record the height (in mm) of each peak on the chart.

10.2 Generate report sheets using the "MERCURY" program on LIMS.

A) Type "S MTL,134,METALS" at the * prompt of any LIMS terminal to sign on to the atomic spectroscopy group account.

B) Type in "MERCURY" when the * prompt returns to access the data processing program for mercury analysis. This program is self-explanatory with a few exceptions. Enter 100 mL as the initial volume for all CCV's, CCB's and PB's. When finished entering samples, type "END" and hit return. Type in the requested information and proceed. When processing is complete, the program will request that a printer destination be specified. Printer 0 is in the LIMS office, printer 1 is in GC and printer 3 is in classical chemistry.

C) After the report sheets have been generated type "OFF" at the * prompt to sign off the LIMS system.

10.3 After checking these report sheets for data entry errors, initial each page and turn in the entire data package for review. After being reviewed the data will be returned.

A) Make complete copies of the data for each CLP job and copies of the data report sheets for each regular job.

B) Record the reported values for each regular job on final report sheets and have them double checked by another member of the group.

C) Sign all pertinent chains of custody and return the folder to the file cabinet.

10.4 After recording all mercury data for a particular job, if the job is complete, place the entire folder on the supervisor's desk for data review. If the job requires additional analysis, return the folder to the file cabinet. For CLP-type jobs that require computer-generated forms, return the completed folder to the file cabinet and notify the computer operator when the job is complete and ready for data entry.

11.0 SPECIAL PROCEDURES - N/A

12.0 USE OF OTHER REQUIRED SOP'S - N/A

13.0 DOCUMENTATION OF PROBLEMS

13.1 Corrective Action Reports - If any discrepancy occurs during the analysis that requires the attention of others (such as low sample volume, standard or instrument problems), a corrective action report is generated per atomic spectroscopy corrective action report procedure - AS:040590.

13.2 All CLP-type reports and regular reports require case narratives that specifically address any anomalies and their possible causes that may have occurred during analysis.

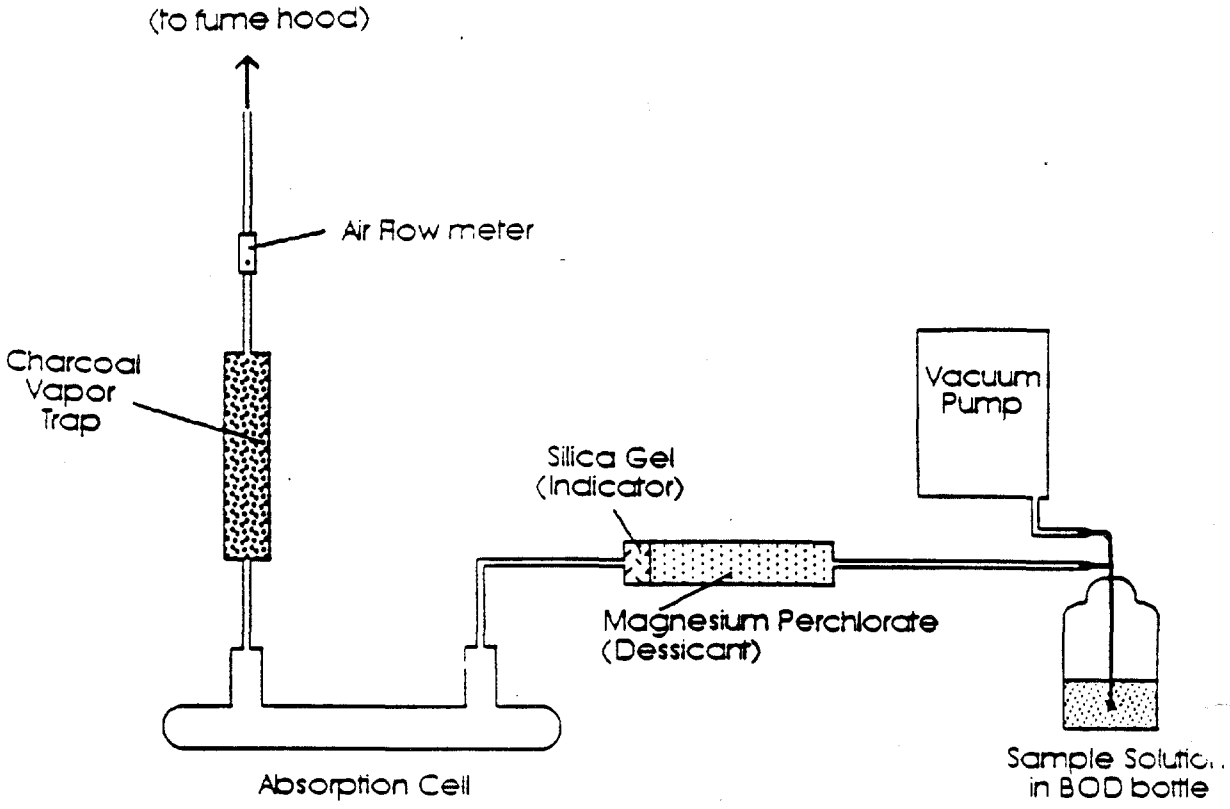
14.0 REFERENCES

14.1 Methods for Chemical Analysis of Water and Wastes, EPA 600/4-82-055, December 1982, Method 245.1.

14.2 USEPA Contract Laboratory Program, SOW 7/88.

14.3 Test Methods for Evaluating Solid Waste, Volume 1A, USEPA SW846, 3rd Edition, November 1986, Method 7470.

FIGURE 1.



NOTE: Tubes should be changed and repacked after each mercury run unless the length of the run is less than 30 minutes. The absorption cell should be cleaned approximately once a month using 1:1 HNO3 acid and nanopure water. The cell can be dried using nitrogen. The nitrogen tank can be located in the Extractions Department. Care must be taken that the cell is free of any water.

ORDER OF PACKING:

1. **Silica Gel (indicator)**
2. **Magnesium Perchlorate (Dessicant)**
3. **Charcoal Vapor Trap**

FIGURE 2.

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

Example of water run log

MERCURY RUN LOG

Matrix: (water) soil
Job #: 0100, 0200, 0123, 0333

Analyst: Lily P.
Date: 3/5/92
Page 1 of 1

Sample ID	Bottle ID	Time	Sample ID	Bottle ID	Time
Blank	1	1000	0200003	24	
0.5	2		-004	25	
1.0	3		0123001	26	
2.0	4		-002	27	
5.0	5		-003	28	
10.0	6		-004	29	
ICV	7	1006	-005	30	
ICB	8	1007	CCV2	31	1030
PBW	9		CCB2	32	1031
0100001	10		0223001	33	
-002	11		-002	34	
-0020	12		-003	35	
-0025	13		-003D	36	
-003	14		-0035	37	
-004	15		PBW2	38	
-005	16		CCV3	39	1038
-006	17		CCB3	40	1039
-007	18				
CCV1	19	1018			
CCB1	20	1019			
0100008	21				
0200001	22				
-002	23				

301194

FIGURE 3.

PRODUCED: 05/04/90 16:12

Procedure to follow when analyzing data.
→ Refrigerator Number

SAMPLE	TEST	PROTOCOL	MATRIX	LOCATION	SAM ID	CASE #	INSTRUCT	DATE
0477004LCS00	MS-PREP	CLP	(1) + H_2O	17	*BEN78	13421		
0651024 00	MS-PREP	SOP	3	32	COMPOSITE #17			
0651025 00	MS-PREP	SOP	3	32	COMPOSITE #18			
0650010DUP00	MS-PREP	CLP	1	EPA4	*BBZ14	13583		
0805003 00	MS-PREP	SW846	2	4,36,25,35,6	X03	RA789	VO	
0892004DUP00	MS-PREP	SW846	1	3,7,34	006-G	RA789		
0892004LCS00	MS-PREP	SW846	1	3,7,34	X06-G	RA789		
0892004SPK00	MS-PREP	SW846	1	3,7,34	006-G	RA789		
0950001 00	MS-PREP	SW846	(3) other	CAB1	01	R389		
0950002 00	MS-PREP	SW846	3	CAB1	02	R389		
0950003 00	MS-PREP	SW846	3	CAB1	03	R389		
0950004 00	MS-PREP	SW846	3	CAB1	04	R389		
0950005 00	MS-PREP	SW846	3	CAB1	05	R389		
0950006 00	MS-PREP	SW846	3	CAB1	03E	R389		
0950007 00	MS-PREP	SW846	3	CAB1	04E	R389		
0922007 00	MS-PREP	SOP	1	31,27,19	MM3			
0922008 00	MS-PREP	SOP	1	31,27,19	MM4			
0922009 00	MS-PREP	SOP	1	31,27,19	MM5A			
0931001 00	MS-PREP	SOP	1	31,29	INGE-002		AS	
0951001 00	MS-PREP	SOP	1	21	DGA-7/91-1358			
0951002 00	MS-PREP	SOP	1	21	BL-4/90-1216			
0951003 00	MS-PREP	SOP	1	21	BL-9/90			
0951004 00	MS-PREP	SOP	1	21	SL-L-7/90-1748			
0951005 00	MS-PREP	SOP	1	21	SPL-6/91-1725			
0951006 00	MS-PREP	SOP	1	21	ITW-SL-3/89-217			
0951007 00	MS-PREP	SOP	1	21	ITW-7/91-1517			
0951008 00	MS-PREP	SOP	1	21	AGA-12/89-1036			
0951009 00	MS-PREP	SOP	1	21	AGA-6/91-947			
0951010 00	MS-PREP	SOP	1	21	3M-3/91-940			
0951011 00	MS-PREP	SOP	1	21	SPH-9/90-544			
0951012 00	MS-PREP	SOP	1	21	SPHH-12/90-249			
0951013 00	MS-PREP	SOP	1	21	SPBL-12/90-5048			
0951014 00	MS-PREP	SOP	1	21	ILITW-8/90-4628			
0951015 00	MS-PREP	SOP	1	21	ITW-12/90-3758			
0951016 00	MS-PREP	SOP	1 soil	21	SLBL-7/90-4048			
0940001 00	MS-PREP	OTHER	(2) →	7,35,22,19,29,33	W-40 39'-41'			
0940002 00	MS-PREP	OTHER	2	7,35,22,19,29,33	W-36 35-36			
0940003 00	MS-PREP	OTHER	1	7,35,22,19,29,33	FIELD BLANK #1 4-12-			
0981002 00	MS-PREP	SOP	1	28,30,20,34	ZE0141		VO	
1034001 00	MS-PREP	SW846	2	34,5	29054-I		AS,CC,VO	
1034002 00	MS-PREP	SW846	2	34,5	29054-II		AS,CC,VO	
1034003 00	MS-PREP	SW846	2	34,5	29054-III		AS,CC,VO	
0012275 00	MS-PREP	SOP	2	7,34	R-19-110-SL(5-7)		VO	
0012276 00	MS-PREP	SOP	2	7,34	R-19-102-SL(10-11)		VO	
0012277 00	MS-PREP	SOP	2	7,34	R-19-111-SL(12-14)		VO	
0947001 00	MS-PREP	CLP	1	EPA2	MXZ595	13849		
0947001DUP00	MS-PREP	CLP	1	EPA2	MXZ595	13849		
0947001LCS00	MS-PREP	CLP	1	EPA2	MXZ595	13849		
0947001SPK00	MS-PREP	CLP	1	EPA2	MXZ595	13849		
0947002 00	MS-PREP	CLP	1	EPA2	MXZ596	13849		

301195

FIGURE 4.

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

023

Example of water prep log:

METALS (WATER)/SOIL PREPERATION LOG - MERCURY

Top Loading Balance Calibration N/A
Analytical Balance Calibration N/A

Page 1 of

IEA ID	IFB ID	Wt(g) Vi(mL)	Vf(mL)	SDG ID	QA/QC ID	Wt/Vi	QC add	Vf(mL)	SDG
0100001		100	100	10	0.5	99.5	0.5mL B	100	
-002				11	1.0	99.0	1.0mL B		
-003				14	2.0	98.0	2.0mL B		
-004				15	5.0	95.0	5.0mL B		
-005				16	10.0	90.0	10.0mL B		
-006				17	ICV	99.0	1mL ICV5		
-007				18	CCV1				
-008				21	CCV2				
0200001				22	CCV3	↓	↓		
-002				23	ICB	100			
-003				24	CCB1				
-004				25	CCB2				
0123001				26	CCB3				
-002				27	Blank				
-003				28	PBW1				
-004				29	PBW2				
-005				30	0100020				
0223001				33	0100025		1mL B		
-002				34	0223003D				
-003		↓	↓	35	0223003S	↓	1mL B	↓	

QA/QC

Standard ID = MTL-W-705B
ICV ID = ICV50689E1
CCV ID = CCV50689E1
LCS ID = N/A
Spike ID = MTL-W-705B

Prepared by: Libby Pujda
Date: 3/15/91

Accepted by:
Date:

Lot #: Hydroxylamine Hydrochloride 28 Stannous Chloride 15 KMnO4 12
H2SO4 58 HNO3 560 K2O8S2 4 NaCl 6

301196

FIGURE 5. AUTOCLAVE LOG BOOK

DATE	INITIALS	TEMP	TIME	LOAD
15 DEC 89	CMN	[illegible]	[illegible]	[illegible]
19 DEC 89	JTV			
20 DEC 89	CMN			
28 DEC 89	CMN			
31 DEC 89	CMN			
1 JAN 90	CMN			
3 JAN 90	ASB	"	30	SPC Aq
1/4/90	JTV	"	15	Hg Prep
7 JAN 90	CMN	"	"	
1/8/90	JTV	"	"	"
1/9/90	JTV	"	"	"
1/11/90	JTV	"	"	"
1/17/90	JTV	"	"	"
1/19/90	SM	"	"	Hg PR
1/19/90	JTV	"	"	" X
1/22/90	JTV	"	"	" X
1/24/90	JTV	"	"	" X
1/25/90	JTV	"	"	" X
1/30/90	JTV	"	"	" X
1/31/90	JTV	"	"	" X
2/5/90	JTV	"	"	" X

SOP/FORM MODIFICATION
APPROVAL SHEET

SOP/Form Modification Requested By: JOSEPH VENEZIA Date: 5/4/90

Deadline for Modification of SOP/Form: 5/11/90

Deadline for Implementation of Modified SOP/Form: _____

Date SOP/Form Modified: 5/3 - 5/4/90

Date Typing of Modification Sent to Typist: 9/26/90

Date Typing of Modification Completed: 10/1/90

Code of SOP/Form: AS:02159013

Pages Changed: all

Approval of Modification to SOP/Form

Group/Section Leader: D M Vasique, Date: 5/7/90

Lab Manager: _____, Date: _____

QA Director: _____, Date: _____

Lab Director: _____, Date: _____

Date Modification to be Implemented: _____

Copies of Final Approved Modification Sent to:

Section/Group Members, Lab Manager, Lab Directors (all labs), Marketing Manager, QA Directors (all labs)

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

THERMO JARRELL ASH ICAP61

AS:020689:0

Whippany, New Jersey 201-428-8181
Schaumburg, Illinois 708-705-0740
Miramar Florida 407-989-0928
Essex Junction, Vermont 802-878-5138
N. Billerica, Massachusetts 617-272-5212
Cary North Carolina 919-677-0090

STANDARD OPERATING PROCEDURE
THERMO JARRELL ASH ICAP61

Abstract

This method outlines the simultaneous analysis of water and soil samples through the measurement of atomic emission by an optical spectroscopic technique. The samples are aspirated through a nebulizer, which results in an aerosol that is then introduced into the plasma. In the plasma excitation of the sample results in characteristic atomic emission spectra. The spectra are dispersed by a grating with the resultant intensities monitored by specific photomultipliers located along the focal curve.

It is the intent of this procedure to comply with the IEA business ethics policy.

INTRODUCTION

This procedure describes the analysis of water and soil samples utilizing the Thermo Jarrell Ash inductively coupled argon plasma (ICAP).

1.0 Scope and Application

1.1 Other related SOP's - Glassware cleaning procedure, standard & sample preparation logs, standard & sample preparation SOP's and corrective action SOP.

1.2 The analytes determined using this procedure are:

Aluminum	Cobalt	Potassium
Antimony	Copper	Selenium
Arsenic	Iron	Silver
Barium	Lead	Sodium
Beryllium	Magnesium	Tin
Cadmium	Manganese	Titanium
Calcium	Molybdenum	Vanadium
Chromium	Nickel	Zinc

2.0 Summary of Method

2.1 The sample is digested with the appropriate acids according to the sample preparation SOP.

2.2 The Thermo Jarrell Ash ICAP61 is standardized using 7 working standards.

2.3 Appropriate QC check standards are measured according to EPA or routine protocols at the beginning, throughout the duration and at the end of the analysis to assure measurement is accurate.

2.4 Linearity of high analytes is monitored and measured. Check solutions are run to assure all applicable interelement corrections are applied.

2.5 The data files are copied onto a floppy disk for transfer to Smartlog software for CLP deliverable generation.

3.0 Interferences - N/A

4.0 Apparatus and Materials

4.1 Thermo Jarrell Ash ICAP61 (Inductively Coupled Argon Plasma)

4.2 Gilson Miniplus 2 Peristaltic Pump including sample tubing (0.30 ID) and rinse tubing (0.06 ID)

4.3 Thermo Jarrell Ash Autosampler Model #ICS-240

4.4 IBM AT Personal Computer and 5.25 floppy diskettes

4.5 1,000 uL variable Eppendorf pipette

4.6 Serological pipettes

4.7 10 mL capacity polypropylene test tubes

4.8 ICAP working standards as described in the ICAP standards logbook

4.9 EPA QC standard solutions as described per EPA in the atomic spectroscopy reagents logbook, or equivalent

4.10 Thermo Jarrel Ash Operator's Manual - January 1988 Part #125791-01

5.0 Reagents

5.1 ICAP working standards for each analyte

5.2 ICAP QC standard solutions

5.3 Nitric acid

5.4 Hydrochloric acid

5.5 Hydrogen peroxide

5.6 Triton x-100

6.0 Sample Collection, Preservation and Storage - N/A

7.0 Procedure

7.1 Startup of the ICAP61

WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator should be familiar with the operator's manual supplied by Thermo Jarrell Ash.

B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.

C) Check that the pressure of the argon supply is 50 psi.

D) On the front of the instrument check that the standby (SB) and fatigue (FAT) lights are lit. At the back of the instrument check that the high voltage (HV) power switch is on. If the lights are not on, consult the Thermo Jarrell Ash operator's manual to conduct a "cold startup" (TJA 5.1.1).

E) Check that the drain tube is immersed in at least 8 inches of water in the plastic waste container. Ensure there are no crimps in this tubing.

F) On the RF generator panel (front of the instrument) (Figure 1.0), working from the top down, and left to right, the controls should be set as follows:

1) Automatic power switch in MANUAL mode

2) Automatic forward power control set at approximately 3.7

3) Forward power switch - ON

4) Auto tuner control - AUTOMATIC

5) Reflected power alarm (preset per manufacturer's instructions)

6) Forward power manual control rheostat - set the knob as far as it will go counterclockwise

7) Control switch OFF and line switch ON

G) Turn the control switch up to the on position. The white power on lamp should be illuminated.

H) Connect the rinse and sample tubing on the peristaltic pump. Check that there is sufficient rinse solution in the rinse supply container. Start the pump, making sure that the sipper is immersed in the rinse solution.

I) On the gas flow control panel turn the toggle switches for the torch and the sample argon flows on. The torch flow meter should register about 15 or 16 when measuring on the center of the bead. The sample flow bead measures at a reading of 0.6 or more, depending on the optimum conditions for that specific torch (See the TJA manual for optimizing the torch).

J) Check that the blue RF off lamp on the RF generator control panel is illuminated. It normally comes on approximately 5 minutes after the generator is turned on. If it is not on, check the argon supply and cooling water to be sure they are in adequate supply. Also check that the plasma chamber door is closed properly.

K) Open the plasma access door. Check that the torch is aligned properly per manufacturer's instructions, the tesla wire is connected to the top arm of the torch, and the arms of the torch are equidistant from the horizontal position of the spectrometer to prevent arching. Close door.

L) When the argon has been flushing the torch system for at least 3 minutes, shut off the argon sample toggle switch.

M) Press the red RF on button.

N) To ignite the plasma, slowly rotate the forward power manual control rheostat knob clockwise until the forward power meter reads about 0.5 kW.

O) Press the ignitor button while watching the torch through the window. A faint spiral of argon gas can be observed in the outer chamber of the torch. Continue turning the forward power rheostat until the plasma ignites. The ignitor button may need to be pressed several times before the plasma is successfully ignited.

P) Turn the automatic power control switch to the AUTOMATIC mode.

Q) Turn the forward power rheostat to the full clockwise position.

R) Very slowly, flip the argon sample toggle switch to the ON position. The plasma is fully on and requires about one hour to stabilize. If the torch is extinguished by this step, push the blue RF off button, press the reset button, turn back the automatic power control switch to the MANUAL position and turn completely counterclockwise the forward power rheostat. Begin the procedure again from step L.

7.2 Sample Evaluation

A) Locate the samples to be analyzed and compare them to the applicable prep log in order to determine if they require CLP or routine analysis protocols. Check also if the samples require analytical spikes, as in the case of E.P. toxicity/TCLP samples. Determine which elements are required and prepare the

appropriate calibration standards according to the standards preparation procedures outlined in the ICAP standards logbook. Complete all entries in the logbook regarding stock solutions and acids used for their preparation.

NOTE: Elements for an E.P. toxicity/TCLP include those normally analyzed by furnace AA - i.e. As, Se and Pb. In order to analyze for these elements using the ICAP a third initial and continuing calibration verification standard denoted ICV1B must be employed in the analysis. However, E.P. toxicity samples do not require Sb, and therefore do not need the EPA-supplied ICV3, etc.

B) Visually inspect the samples, noting which samples were in duplicate and were spiked. Also note initial and final sample volumes, which laboratory control samples were used and also the sample weights. Check this against the sample prep log for completeness for all these mentioned items. During this inspection any highly colored samples (usually an indication of high analyte concentrations) should be noted. These samples may require a rinse cup after them during analysis to minimize carryover effects.

C) Estimate the number of sample cups necessary for the autosampler table. If CLP protocol is required, count also the possibility of all spiked samples needing a post-digest spike, as well as a serial dilution for each sample matrix.

7.3 Program Setup

A) On the main menu of the thermospec software cursor to the SET-UP option and press ENTER.

B) The method name is selected at this time. The name can be either typed in or by pressing the <F6> function key to list all available programs. Currently, method name IEALAB is used for all CLP and routine samples and should be entered here.

C) The first page of the program will appear on the monitor. On the lower righthand portion of the screen the function keys will be listed with a variety of options. Select <F3> (SampInfo).

D) Cursor down 13 times to the default table names, specifically AUTOSAMPLER. Enter the name of the autosampler table you are about to create using the format: WMMDDYY. For instance, if today's date is February 6, 1989, then the autosampler table entered here would be: W020689.

E) Cursor to the default file names portion and enter under the data file the same name as the autosampler table, in this case: W020689.

F) Press the function key <F9> (done/keep) until back to the set-up option (entered twice). The program now has stored the name of the new autosampler table and the name of the new data file in which the information is to be stored.

7.4 Setup of the Autosampler Table

A) On the main menu cursor once again to the setup mode and press enter.

B) Cursor through the setup options to AUTOSAMPLER and press enter.

C) As with the program portion, here you will be required to enter an autosampler table name. Use the name selected in the program portion, which, in this case is: W020689 and press enter.

D) The autosampler screen will display information regarding this specific table. At this point under "set # description", it will read, "this table is empty". Choose the <F3> (add set) function key to enter the information for this table.

E) For CLP work use the following entries here:

"SET DESCRIPTION" - leave blank
"METHOD NAME" - IEALAB or current method in use
"DEFAULT COMMENT" - enter the SDG number
"DEFAULT OPERATOR ID" - enter the initial of the ICAP operator
"LABORATORY ID" - enter the inititals of the person who prepared the samples
"CUSTOMER ID" - enter the date that the samples wer prepared using MM/DD/YY format
"AUTO-RINSE BETWEEN SAMPLES?" - leave YES
"RINSE TIME" - enter 90 seconds
"# SAMPLE POSITIONS NEEDED" - enter the number of cups required, remembering to include extras for rinse cups and any high analytes that may be needed to check for interelement corrections (IEC's).

Cursor down to "default limit check table name" - and type in LIMITS.

For non-CLP type work the only entries required are:

"METHOD NAME" - IEALAB
"DEFAULT OPERATOR ID" - ICAP operator's initials "RINSE TIME" - 60 seconds
"# SAMPLE POSITIONS NEEDED" - as required
"DEFAULT LIMIT CHECK TABLE NAME" - LIMITS

This page is complete. Now press <F1> (ed samples) to set up the sample cup designations.

F) Press <F5> (ins stds). This allows the insertion for a standardization standard. Type STD2 then enter. Once again press <F5> (ins stds).

Type STD3 then press enter. Use this same sequence to enter STD4, STD5, STD6, STD7, and STD1. All the standards are now entered for standardization of the instrument.

STD2
STD3
STD4
STD5
STD6
STD7
STD1

G) Initial QC must now be entered. Press <F3> (insQC) and type ICV1 then press enter. Cursor to the check tables column and type in again ICV1 and press enter. Cursor back to the next sample name column and press <F3> again. This time type ICV1A. Use this same procedure to enter:

<F3> ICV1---------ICV1
<F3> ICV1A--------ICV1A
<F3> ICV1B--------ICV1B
<F3> ICB----------ICB
<F3> CRII---------CRII
<F3> ICSAI--------ICSAI
<F3> ICSABI-------ICSABI

All initial QC is now entered.

H) To enter the sample names a specific format must be utilized for CLP protocol in order to facilitate proper data storage in the appropriate SMARTLOG software files.

Init vol or wt/ (5 characters)	Final volume/ (5 characters)	EPA sample name/ (6 characters)	Suffix (1 character)

The initial and final volumes are 5 characters including decimals. The EPA sample name is the sample name provided by the EPA - NOT the in-house job name. The suffix is for duplicate (D), spike (S), post-digest (P) and serial dilution (L) designations.

Examples:

Water sample, EPA001, having initial and final volumes of 100 mLs is entered: 1.0001.000EPA001, DUP = 1.0001.000 EPA001D.

Soil sample, EPA002, having an initial weight of 1.07 grams, and a final volume of 200 mLs is entered: 1.0700.200EPA002.

NOTE: The difference in decimal placement in 100 and 200 mLs for water and soil sample final volumes is to accommodate the mg/Kg calculation for soil samples.

Prep blanks are entered: 1.0001.000PBW (water samples), 1.0000.200PBS (soil samples).

Laboratory Control Samples (LCS) are entered: 1.0001.000LCSW (water samples), 1.XXX0.200LCSS (soil samples).

I) When entering sample names for non-CLP samples, the in-house job name and sample number may be used i.e. Job 123, sample 001 may be entered as: 0123-001.

In both CLP and non-CLP work, only an entry in the sample name column is necessary.

J) According to contract, CLP samples must have QC run after every 10 samples. The difference in time between two samples and QC may not exceed the lowest difference in time between any two consecutive analytical samples. In the case of initial QC, the CRI, ICSA and ICSAB count as samples. Therefore, only 7 actual samples can be analyzed before the next series of QC. When running CLP work several things must be kept in mind, calculations must be made during the analysis to determine if spike recoveries are within the 75-125 percent windows. Also, all prep blank and laboratory control samples (LCS) must pass all parameters, otherwise the sample preparation may need to be repeated. Therefore, it is prudent to

place these samples near the beginning to allow sufficient time for calculation and determination of all acceptable parameters, whether post-digest spike samples are necessary, and which sample(s) is appropriate for serial dilution. An example setup could be:

STD2
STD3
STD4
STD5
STD6
STD7
STD1

ICV1
ICV1A
ICB
CRII
ICSAI
ICSABI

RINSE
1.0001.000AAA002
1.0001.000AAA002D
1.0001.000AAA0002S
1.1000.200AAA003D
1.1100.200AAA0003S

CCV1A
CCV1
CCB1

1.0001.000PBW
1.0001.000PBS
1.0001.000LCSW
1.0000.200LCSS
1.0001.000AAA001

QC(x)

1.0001.000AAA002P
1.1000.200AAA003P
1.0005.000AAA L
1.0005.000AAA L

FINAL QC

K) The final QC is run in the following order: CRIF, ICSAF, ICSABF, CCV_A, CCV_, and CCB_. As in the case of the initial QC, the CRIF, ICSAF and ICSABF count as samples, therefore only 7 samples can precede them.

L) Double check autosample table making sure all samples and QC are entered correctly.

M) In order to store the completed autosampler table press <F9> (done/keep), then press again to get back to the main menu.

N) Cursor through setup and go to the autosampler section again to check that the table is in memory. Check paper supply to be sure there is enough for the entire run. Then press <F2> to print out the table. Get two printouts, one to keep on file in the ICAP run logbook and one to go with the data. Both copies should be dated and initialed by the ICAP operator. Press <F9> (done/keep) to get back to the main menu.

O) Using the printed autosampler table, pour fresh standards and all the samples into the cups and test tubes and place them in their designated positions on the autosampler tray.

7.5 Operation of the ICAP

A) Cursor to the main menu. Select the operation mode and press enter. Select ANALYSIS and press enter. Once again the method name will be requested. Type IEALAB and press enter.

B) Open the front portion of the instrument that contains the mercury (Hg) lamp, and place the lamp in front of the entrance slit of the spectrometer. Select <F5> (profile) in order to optimize the light path before operation. Profiling the instrument may be done manually or automatically per manufacturer's instructions, on the monitor. However, if the manual mode is selected, check the profile setting using the automatic mode. Do NOT begin analysis unless the offset value at the end of the profiling procedure is less than 0.1. Move the mercury lamp out of the path of the entrance slit and shut the door of this compartment.

C) To initiate the analysis, press <F9> (autosampler). This will call the autosampler table from memory. On the monitor should be the first sample, usually STD2. Check

that the sipper is firmly in the autosampler, and the height is adequate for both standard cup and test tube levels. To start the analysis, press <F1> (run). The autosampler will move to the first sample and begin operation.

8.0 Quality Control

8.1 Frequent monitoring of the results during an analysis is crucial. Check the precision during the standardization stage to assure that the three replicate exposures are similar. Also check to determine that all initial QC passes. If it does not, stop the analysis, determine the problem and restart the analysis.

8.2 Check the ICSA sample for any IEC's attributed to iron, aluminum, calcium or magnesium. Normally, the one that recurs is slight changes of beryllium resulting from iron.

8.3 Once the sample results are available, check the sample and duplicate to see if their values are close. Then calculate the spike recoveries for both water and soil samples, remembering to calculate using proper weights and percent solids. Determine if any post-digest spike samples are necessary and add these to the autosampler tray in the spaces that were pre-set on the autosampler table. On the autosampler table note which elements required a post-digest spike and the level of the spike. If any discrepancies occur with the sample, its duplicate or spike, a corrective action report should immediately be submitted to a supervisor to evaluate the situation.

9.0 Method Performance

9.1 Check the prep blanks and LCS's to make sure they pass all requirements. If they do not, submit a corrective action report to a supervisor immediately.

9.2 Once most of the samples are run, determine which samples would be suitable for a serial dilution, making sure one of each matrix, i.e. soil and water is made in a 1:5 dilution. On the autosampler table note which sample was diluted.

9.3 If any analyte exceeds the linear range (determined quarterly) a dilution must be made to bring the analyte into range. If this sample was chosen as the serial dilution, further 1:5 dilution is needed for those analytes exceeding the linear range.

9.4 Assure all QC is within limits and final QC passes. The analysis is complete.

9.5 Once the analysis is finished, check all values again, all QC and then do all appropriate calculations of IEC's from other elements are necessary. Standard prep dates must be recorded on each standard printout.

10.0 Data Reduction and Data Handling

10.1 Making Diskette for Data Transfer to Smartlog

A) On main menu cursor to EXIT and press enter.

B) The computer will respond with "C>". Type CD\STATION\DATA\ ANALYTIC then press enter.

C) Type DIR and press enter. A directory of all analytical tables will appear.

D) Place a formatted floppy disk in the disk drive and type COPY SAMPLES.DBF A: and press enter. The red light over the disk drive will turn on. The light will go out and the computer will respond with "C> 1 file copied" when complete.

E) Type COPY W020689.DBF A: (using same example as before), which is the data file used to store the analysis, entered in the program setup mode. Once again, the light will be on during the copying sequence. Again, the computer returns with the prompt "C> 1 file copied" and the light will be off. Remove the disk and affix a label to it on which is the case or job name, data file name and the sample file name that it will be changed to.

F) Change the samples file to another file name, such as 01SAMPLE.DBF. To do this type RENAME SAMPLES.DBF 01SAMPLE.DBF and press enter. Now the samples associated with that specific analysis are called 01SAMPLE.DBF. Approximately once a month old files should be deleted in order to allow sufficient memory space for new files. To delete a file type DEL followed by the file name to be deleted.

G) To return to the Thermospec software, type AUTOEXEC and enter.

10.2 Data Reporting Procedure

A) Once all values for CLP analysis are complete, and all associated IEC's are calculated, sign the appropriate chain of custody, which may include a New Jersey chain of custody and enter the date(s) of analysis.

B) For non-CLP reporting, select the appropriate report sheets, including the QC batch sheet and enter all pertinent information, including all required elements, prep blanks, laboratory control samples, duplicates and spike recoveries. After all forms are completed, sign the applicable chain of custody and enter the date(s) of analysis. Note any discrepancies or problems encountered with analysis that should be included in a case narrative.

11.0 Special Procedures

11.1 Instrument Shutdown

A) Press the blue RF off button.

B) Turn the automatic forward power switch to MANUAL and turn forward power manual control rheostat fully counterclockwise.

C) Shut off the peristaltic pump and loosen the tubing.

D) Flip the control power switch down to the OFF position. Leave the line switch on.

E) Shut off the torch and sample argon toggle switches.

F) On the computer main menu, cursor to EXIT. Press enter.

G) Shut off the computer and monitor.

H) The plasma system is now shut down.

11.2 Preventive Maintenance

A) At least once a week or as necessary, replace rinse and sample tubing on peristaltic pump.

B) Once a week vacuum fan filter and screens in back of ICAP and fans on IBM PC.

C) Periodically check torch for contamination. If buildup does occur, remove torch and wash in aqua regia. Rinse well, dry and replace.

D) Record all standard and QC preparation in logbook and record prep data and initials on flask.

E) Periodically empty drain bucket half way and dispose of waste acid carefully into a proper waste acid container.

F) Document all maintenance, both routine and non-routine, including service calls in the logbook.

R.F. GENERATOR CONTROL PANEL

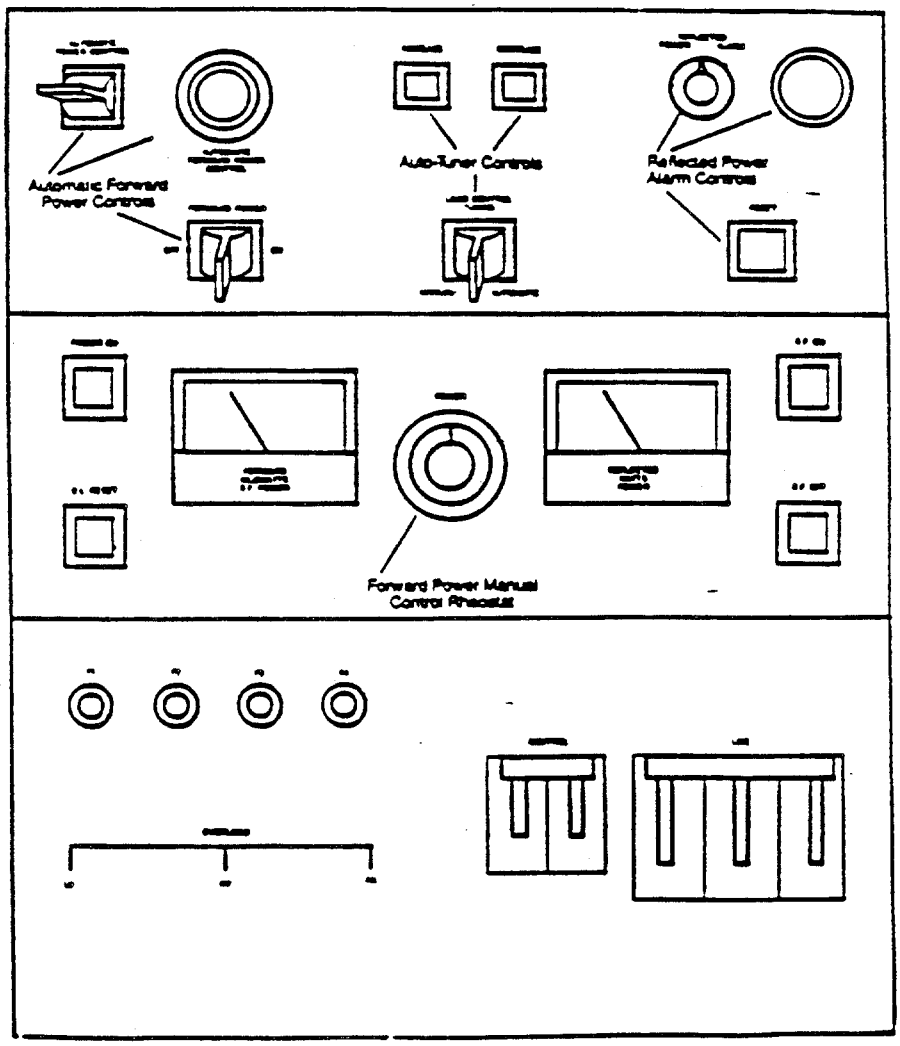


FIGURE 1.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

STANDARD OPERATING PROCEDURE

ICP SET UP

PE6500

AS:070887:1

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

STANDARD OPERATING PROCEDURE
ICP SET UP PE6500

Abstract

This method outlines the analysis of water and soil samples through the measurement of atomic emission by an optical spectroscopic technique. The samples are aspirated through a nebulizer which results in an aerosol that is then introduced into the plasma. In the plasma, excitation of the sample results in characteristic atomic emission spectra.

INTRODUCTION

This procedure describes the analysis of water and soil samples utilizing the Perkin-Elmer 6500 Inductively Coupled Argon Plasma (ICAP).

It is the intent of this procedure to comply with the IEA, Inc. business ethics policy.

1.0 Scope and Application

Related SOP's - Glassware cleaning procedure, standards and sample prep logs, standard and sample preparation SOP's and corrective action SOP.

2.0 Summary of Method

2.1 The sample is digested with the appropriate acids according to the sample preparation SOP.
2.2 The PE6500 is standardized separately for each analyte.
2.3 Appropriate QC check standards are measured according to EPA or routine protocols at the beginning, throughout the duration, and at the end of the analysis to assure measurement is accurate.
2.4 Linearity of high analytes is monitored and measured.

3.0 Interferences - N/A

4.0 Apparatus and Materials

4.1 Perkin-Elmer ICAP 6500
4.2 Gilson Miniplus 2 Peristaltic Pump including sample tubing (0.30 ID)
4.3 Perkin-Elmer 7700 PC
4.4 1,000 uL variable Eppendorf pipette
4.5 Seriological pipettes
4.6 ICAP working standards as described in ICAP standards logbook
4.7 EPA QC standard solutions as described per EPA in the Atomic Spectroscopy Reagents logbook

5.0 Reagents

5.1 ICAP working standards for each element
5.2 ICAP QC standard solutions
5.3 Nitric acid
5.4 Hydrochloric acid
5.5 Hydrogen peroxide
5.6 Triton X-100

6.0 Sample Collection, Preservation and Storage - N/A

7.0 Procedure

7.1 Startup of the ICAP PE6500

WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator should be familiar with the operators manual supplied by Perkin-Elmer.
B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.
C) Check pressure of argon supply.
D) Turn on power to the RF generator.
E) Turn on the power to the torch control box.
F) Turn on gases by pressing "PLAS", "NEB", and "AUX" buttons. Flows should be 16, 1.1 and 0.3 respectively. Allow 3 minutes for argon to fill torch chamber.
G) Connect sample tubing (0.30 ID) to peristaltic pump and place tubing in nanopure water. Turn pump on, make sure flow is smooth.
H) Check water, argon and door interlocks on torch box, all lights should be out. If light is on, check for problem.
I) Within 15 minutes of initial startup the RF ready light will come on.
J) Turn off "NEB" and "AUX" by pressing buttons.
K) Press RF ON button (light should come on). RF power should read 900. When RF light comes on press "ICP IGN".
L) Press on "NEB" and "AUX".

7.2 Instrument Operation

A) Turn on "power" and "run" switches on left side of ICAP.
B) Turn on power to the Perkin-Elmer 7700 personal computer and printer.
C) Type "idris (enter)", this will load idris program.
D) Type "icp" to load software.
E) At this time the computer screen should display a blank METHOD MODE screen.
F) Determine what elements are required for the samples to be analyzed. Experience has shown that the best instrument performance is achieved by following a few basic rules:

Each analytical run should contain those elements whose wavelengths are closest to each other (eg. low end - Sb, Zn, Pb, Cd, Cr, Ni; high end - Be, Cu, Ag)

G) Enter the utilities MODE by pressing the "Utilities" special function key. Select "Library" soft key. Select "List Method" soft key. The screen will display all methods stored and available for use. Select appropriate method by using arrow keys to position

cursor (blue highlight) on desired method. Hit "Recall" special function key.

H) The system will enter the GRAPHICS MODE and automatically set up operating parameters.

I) Hit "Print Elem Parameters" special function key. A listing of the element parameters for selected method will automatically be printed on the PR-210 printer.

Check the calibration standard against the standard cores listed on printout. They should match.

J) Hit "Run Manual" special function key. The 6500 should begin initialization sequence. (This can be noted by printout on PR-210.) Screen will display "depress Hold then Print key and depress Enter" - DO SO. (The Hold must be hit before the Print key.)

K) Start aspirating the calibration standard.

L) System will prompt "Aspirate Standard 1". Depress "Standard" special function key. The system will set gain and run standard. If systems misreads standard 1 it will display "Peak offset aspirate standard 1". Repeat #7 above (????????). This occurrence is not unusual; however, if it happens again there may be a significant problem, consult with supervisor.

M) Depending on the number of calibration standards in the method the system will continue the prompt sequence as follows:

for 2 calibration standards - Aspirate Standard 2
for 3 calibration standards - Aspirate Standard 3

Up to 5 standard positions.

N) After all standards are run the system will prompt "aspirate BLANK and depress read" hit "Blank" special function key. If system displays "Standards out of sequence" after blank is run a significant problem exists, consult supervisor.

O) System will next prompt "Aspirate sample and depress read", from this point on all analyses will be conducted by hitting the "Sample" special function key.

To complete analysis and begin a new one:

P) Hit "Method" special function key and proceed as in #16 (??????).

To complete analysis and shut down:

Q) Hit "Quit" special function key. Screen will prompt "Confirm desire to exit ICP 6500 Program? Y or N", type "Y".

R) Screen will return to "login:" prompt. Turn monitor knob counterclockwise to turn off screen.

S) Remove sample capillary from rinse water and wait approximately one minute. Press "RF OFF" button on torch box, the plasma should immediately go out. Hit gas buttons to turn flows off. Turn off peristaltic pump and release side area and relax tubing.
T) Turn power to torch box off.
U) Turn 6500 to stand by.
V) Turn RF power supply off.

8.0 QUALITY CONTROL

8.1 The instrument must be calibrated at the start of each new analytical run. (The analytical run is based on the specific elements being run within a specific method: i.e. one run may contain all low wavelength elements while another run will contain high wavelength elements, each of these runs requires a separate calibration.) The calibration consists of a standard at the end of the linear range for each element analyzed (standards can be in lower concentrations in regular sample analysis). The calibration is ended with a calibration blank (reagent water).

8.2 Immediately following calibration an Initial Calibration Verification solution (ICV) must be run for each wavelength analyzed. The concentration found for this standard must fall within ± 10 percent of the true concentration or the run is voided.
NOTE: The ICV is prepared from EPA-supplied solutions. In the event EPA standards are not available a certified solution may be used or a mixture of independent standards may be prepared. The standards must be from a source other than that used to prepare calibration and continuing calibration standards.

8.3 To confirm the linearity at the lower end of ICP instrumental detection a standard equivalent to 2x CRDL must be run for each wavelength analyzed at the beginning and end of each run or every 8 hours.

8.4 To confirm appropriate background correction factors two ICP interference check samples (ICS) must be run starting with ICSA (only interferents) followed by ICSAB (both interferents and analytes). If results obtained fall outside ± 20 percent of true concentrations the analysis is terminated. ICSA and ICSAB must be run at the beginning and end of each analytical run or every eight hours.

8.5 A CCV must be run at the beginning of each analytical run; at a frequency of 10 percent during the run (i.e. every 10 samples) and after the last analytical sample. The CCV must not exceed ± 10 percent of the true concentration or the analysis is terminated and all samples run since the last good CCV must be reanalyzed.

8.6 After <u>each</u> <u>ICV</u> <u>and</u> <u>CCV</u> (continuing calibration verification) standard a calibration blank (CB) must be analyzed, this reagent blank must not exceed the CRDL (contract required detection limit) for any analyte run, if it does the analysis is terminated.

8.7 The following represents QC which is associated with a given batch of samples. For a batch to be considered complete, the following must be analyzed for each wavelength required:

1) Preparation Blank - PB - reagent blank put through same digestion procedure as samples. The PB must not exceed the CRDL or all samples associated with that PB must be redigested.

2) Laboratory Control Sample - LCS - liquid or solid depending on sample batch (i.e. water or soils). This sample is usually provided by the EPA and represents a sample with known and certified levels of analytes tested for. The LCS is digested with each batch of samples and provides information on digestion efficiency. As such, the concentration found for each analyte must not exceed ± 20 percent of true or all samples associated with that LCS must be redigested.

3) Duplicate Sample Analysis - For each batch digested at least one sample must be digested in duplicate.

4) Spiked Sample Analysis - For each batch digested at least one sample must be spiked with a known quantity of the analytes of interest. The concentration found must not exceed ± 25 percent of the true (except in the case where the sample itself has the analyte present at a level greater than 4 times the spike concentration). When the spike is out of required control limits a post-digestion spike must be performed. Spike the unspiked aliquot of sample at 2x the concentration present in that sample or at 2x the CRDL, whichever is greater.

TABLE 1
ICP ANALYTICAL RUN

	Calibration
1	ICVS
2	CB (Cal Blk)
3	2x CRI (2x CRDL)
4	ICSA
5	ICSA - Dilution (if necessary)
6	ICSAB
7	ICSAB - Dilution (if necessary)
8	CCVS
9	CB
10	PB
11	LCS
12	Sample 1
13	Sample 1D (duplicate)
14	Sample 1S (spike)
15	Sample 2
16	Sample 3
17	Sample 4
18	Sample 5
19	Sample 6
20	CCV
21	CB
22	Sample 7
23	Sample 8
24	CCV
25	CB
26	2x CRI
27	ICSA
28	ICSA (dilution)
29	ICSAB
30	ICSAB (dilution)
	END

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

ICP SET UP

PE6500

AS:070887:1

Miramar, Florida 305-989-0926
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

STANDARD OPERATING PROCEDURE
ICP SET UP PE6500

Abstract

This method outlines the analysis of water and soil samples through the measurement of atomic emission by an optical spectroscopic technique. The samples are aspirated through a nebulizer which results in an aerosol that is then introduced into the plasma. In the plasma, excitation of the sample results in characteristic atomic emission spectra.

INTRODUCTION

This procedure describes the analysis of water and soil samples utilizing the Perkin-Elmer 6500 Inductively Coupled Argon Plasma (ICAP).

It is the intent of this procedure to comply with the IEA, Inc. business ethics policy.

1.0 Scope and Application

Related SOP's - Glassware cleaning procedure, standards and sample prep logs, standard and sample preparation SOP's and corrective action SOP.

2.0 Summary of Method

2.1 The sample is digested with the appropriate acids according to the sample preparation SOP.
2.2 The PE6500 is standardized separately for each analyte.
2.3 Appropriate QC check standards are measured according to EPA or routine protocols at the beginning, throughout the duration, and at the end of the analysis to assure measurement is accurate.
2.4 Linearity of high analytes is monitored and measured.

3.0 Interferences - N/A

4.0 Apparatus and Materials

4.1 Perkin-Elmer ICAP 6500
4.2 Gilson Miniplus 2 Peristaltic Pump including sample tubing (0.30 ID)
4.3 Perkin-Elmer 7700 PC
4.4 1,000 uL variable Eppendorf pipette
4.5 Seriological pipettes
4.6 ICAP working standards as described in ICAP standards logbook
4.7 EPA QC standard solutions as described per EPA in the Atomic Spectroscopy Reagents logbook

5.0 Reagents

5.1 ICAP working standards for each element
5.2 ICAP QC standard solutions
5.3 Nitric acid
5.4 Hydrochloric acid
5.5 Hydrogen peroxide
5.6 Triton X-100

6.0 Sample Collection, Preservation and Storage - N/A

7.0 Procedure

7.1 Startup of the ICAP PE6500

WARNING: Ensure that no pacemaker users are in the vicinity of the spectrometer. RF generator radiation may interfere with pacemaker operation.

A) Prior to initiation of the plasma, the operator should be familiar with the operators manual supplied by Perkin-Elmer.
B) Before starting the instrument, check the exhaust vents and cooling water to ensure they are operating.
C) Check pressure of argon supply.
D) Turn on power to the RF generator.
E) Turn on the power to the torch control box.
F) Turn on gases by pressing "PLAS", "NEB", and "AUX" buttons. Flows should be 16, 1.1 and 0.3 respectively. Allow 3 minutes for argon to fill torch chamber.
G) Connect sample tubing (0.30 ID) to peristaltic pump and place tubing in nanopure water. Turn pump on, make sure flow is smooth.
H) Check water, argon and door interlocks on torch box, all lights should be out. If light is on, check for problem.
I) Within 15 minutes of initial startup the RF ready light will come on.
J) Turn off "NEB" and "AUX" by pressing buttons.
K) Press RF ON button (light should come on). RF power should read 900. When RF light comes on press "ICP IGN".
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7.2 Instrument Operation

A) Turn on "power" and "run" switches on left side of ICAP.
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F) Determine what elements are required for the samples to be analyzed. Experience has shown that the best instrument performance is achieved by following a few basic rules:

Each analytical run should contain those elements whose wavelengths are closest to each other (eg. low end - Sb, Zn, Pb, Cd, Cr, Ni; high end - Be, Cu, Ag)

G) Enter the utilities MODE by pressing the "Utilities" special function key. Select "Library" soft key. Select "List Method" soft key. The screen will display all methods stored and available for use. Select appropriate method by using arrow keys to position

cursor (blue highlight) on desired method. Hit "Recall" special function key.

H) The system will enter the GRAPHICS MODE and automatically set up operating parameters.

I) Hit "Print Elem Parameters" special function key. A listing of the element parameters for selected method will automatically be printed on the PR-210 printer.

Check the calibration standard against the standard cores listed on printout. They should match.

J) Hit "Run Manual" special function key. The 6500 should begin initialization sequence. (This can be noted by printout on PR-210.) Screen will display "depress Hold then Print key and depress Enter" - DO SO. (The Hold must be hit before the Print key.)

K) Start aspirating the calibration standard.

L) System will prompt "Aspirate Standard 1". Depress "Standard" special function key. The system will set gain and run standard. If systems misreads standard 1 it will display "Peak offset aspirate standard 1". Repeat #7 above (????????). This occurrence is not unusual; however, if it happens again there may be a significant problem, consult with supervisor.

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Up to 5 standard positions.

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To complete analysis and begin a new one:

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To complete analysis and shut down:

Q) Hit "Quit" special function key. Screen will prompt "Confirm desire to exit ICP 6500 Program? Y or N", type "Y".

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S) Remove sample capillary from rinse water and wait approximately one minute. Press "RF OFF" button on torch box, the plasma should immediately go out. Hit gas buttons to turn flows off. Turn off peristaltic pump and release side area and relax tubing.
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8.4 To confirm appropriate background correction factors two ICP interference check samples (ICS) must be run starting with ICSA (only interferents) followed by ICSAB (both interferents and analytes). If results obtained fall outside ± 20 percent of true concentrations the analysis is terminated. ICSA and ICSAB must be run at the beginning and end of each analytical run or every eight hours.

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8.6 After <u>each</u> <u>ICV</u> <u>and</u> <u>CCV</u> (continuing calibration verification) standard a calibration blank (CB) must be analyzed, this reagent blank must not exceed the CRDL (contract required detection limit) for any analyte run, if it does the analysis is terminated.

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1) Preparation Blank - PB - reagent blank put through same digestion procedure as samples. The PB must not exceed the CRDL or all samples associated with that PB must be redigested.

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3) Duplicate Sample Analysis - For each batch digested at least one sample must be digested in duplicate.

4) Spiked Sample Analysis - For each batch digested at least one sample must be spiked with a known quantity of the analytes of interest. The concentration found must not exceed ± 25 percent of the true (except in the case where the sample itself has the analyte present at a level greater than 4 times the spike concentration). When the spike is out of required control limits a post-digestion spike must be performed. Spike the unspiked aliquot of sample at 2x the concentration present in that sample or at 2x the CRDL, whichever is greater.

TABLE 1
ICP ANALYTICAL RUN

	Calibration
1	ICVS
2	CB (Cal Blk)
3	2x CRI (2x CRDL)
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7	ICSAB - Dilution (if necessary)
8	CCVS
9	CB
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12	Sample 1
13	Sample 1D (duplicate)
14	Sample 1S (spike)
15	Sample 2
16	Sample 3
17	Sample 4
18	Sample 5
19	Sample 6
20	CCV
21	CB
22	Sample 7
23	Sample 8
24	CCV
25	CB
26	2x CRI
27	ICSA
28	ICSA (dilution)
29	ICSAB
30	ICSAB (dilution)
	END

STANDARD OPERATING PROCEDURE

ANALYSIS OF SAMPLES

FOR TOTAL CYANIDE CLP PROTOCOL

WC:070187:4

200 MONROE TURNPIKE • MONROE, CONNECTICUT 06468 • (203) 452 8200

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STANDARD OPERATING PROCEDURE
ANALYSIS OF ENVIRONMENTAL STANDARDS FOR TOTAL CYANIDE
CLP PROTOCOL

Abstract

This procedure is designed for the analysis of cyanide in water and soil samples by acidified distillation and collecting the hydrogen cyanide gas, HCN, in a sodium hydroxide, NaOH, scrubber and analyzing colorimetrically utilizing the pyridinebarbituric acid method or titrimetrically with silver nitrate.

METHOD FOR TOTAL CYANIDE ANALYSIS

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the determination of cyanide in drinking, surface and saline waters, soils, domestic and industrial wastes.

1.2 The titration procedure using silver nitrate with p-dimethylaminobenzalrhodanine indicator is used for measuring concentrations of cyanide exceeding 1 mg/L (0.25 mg/250 mL of absorbing liquid).

1.3 The manual colorimetric procedure is used for concentrations below 1 mg/L of cyanide and is sensitive to about 0.02 mg/L.

1.4 The detection limit for soils is dependent on the dry weight basis of the sample taken for analysis.

2.0 SUMMARY OF METHOD

2.1 The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a reflux-distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by volumetric titration or colorimetrically.

2.2 In the colorimetric measurement the cyanide is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at a pH less than 8 without hydrolyzing to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm for pyridine-barbituric acid. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

2.3 The titimetric measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.

3.0 INTERFERENCES

3.1 Interferences are eliminated or reduced by using the distillation procedure described in Procedure 7.1.

3.2 Sulfides adversely affect the colorimetric and titration procedures. If a drop of the distillate on lead acetate test paper indicates the presence of sulfides, treat 25 mL more of the sample than that

required for the cyanide determination with powdered cadmium carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

3.3 The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 anti-foam agent will prevent the foam from collecting in the condenser. Fatty acids will distill and form soaps under alkaline titration conditions, making the end point almost impossible to detect. When this occurs, one of the spectrophotometric methods should be used.

4.0 APPARATUS

4.1 Reflux distillation apparatus such as shown in Figure 1 or Figure 2. The boiling flask should be of 1 liter size with inlet tube and provision for condenser. The gas absorber maybe a Fisher-Milligan scrubber.

4.2 Microburet, 5.0 mL (for titration).

4.3 Spectrophotometer suitable for measurements at 578 nm with a 1.0 cm cell.

5.0 REAGENTS

5.1 Distillation and Preparation Reagents

5.1.1 Sodium hydroxide solution, 1.25N: Dissolve 50 g of NaOH in distilled water, and dilute to 1 liter with distilled water.

5.1.2 Cadmium carbonate: powdered

5.1.3 Ascorbic acid: crystals

5.1.4 Sulfuric acid: concentrated

5.1.5 Magnesium chloride solution: Weigh 510 g of $MgCl_2.6H_2O$ into a 1000 mL flask, dissolved and dilute to 1 liter with distilled water.

5.2 Stock Standards and Titration Reagents

5.2.1 Stock cyanide solution: Dissolve 2.51 g of KCN and 2 g KOH in 1 liter of distilled water. Standardize with 0.0192 N $AgNO_3$.

5.2.2 Standard cyanide solution, intermediate: Dilute 5.0 mL of stock (1 mL = 1 mg CN) to 100 mL with distilled water. The stock solution is to be titrated weekly to verify concentration.

5.2.3 Standard cyanide solution: Prepare fresh daily by diluting 2.0 mL of intermediate cyanide solution to 100 mL with distilled water and store in a glass-stoppered bottle. 1 mL = 1.0 ug CN (1.0 mg/L).

5.2.4 Standard silver nitrate solution, 0.0192 N: Prepare by crushing approximately 5 g $AgNO_3$ crystals and drying to constant weight at 40°C. Weight out 3.2647 g of dried $AgNO_3$, dissolve in distilled water, and dilute to 1000 mL (1 mL = 1 mg CN).

5.2.5 Rhodanine indicator: Dissolve 20 mg of p-dimethylaminobenzalrhodanine in 100 mL of acetone.

5.2.6 Sodium hydroxide solution, 0.25 N: Dissolve 10 g of NaOH in distilled water and dilute to 1 liter.

5.3 Manual Spectrophotometric Reagents

5.3.1 Sodium dihydrogenphosphate, 1 M: Dissolve 138 g of $NaH_2PO_4.H_2O$ in a liter of distilled water. Refrigerate this solution.

5.3.2 Chloramine-T solution: Dissolve 1.0 g of white, water soluble chloramine-T in 100 mL of distilled water and refrigerate until ready to use. Prepare fresh weekly.

5.3.3 Color Reagent: Pyridine-barbituric acid reagent: Place 15 g of barbituric acid in a 250 mL volumetric flask and add just enough distilled water to wash the sides of the flask and wet the barbituric acid. Add 75 mL of pyridine and mix. Add 15 mL of HCl (sp gr 1.19), mix, and cool to room temperature. Dilute to 250 mL with distilled water and mix. This reagent is stable for approximately six months if stored in a cool, dark place.

6.0 SAMPLE COLLECTION

6.1 All bottles must be thoroughly cleaned and rinsed to remove soluble material from containers.

6.2 Water samples are preserved with 2 mL of 10N sodium hydroxide per liter of sample (pH >12) and ascorbic acid at the time of collection.

6.3 Samples must be stored at 4°C (±2°C) and must be analyzed within 12 days of the verified time samples received.

7.0 PROCEDURE

7.1 Distillation - Test samples with pH paper to assure pH is >12.0. Record in distillation log whether pH is or is not greater than 12.0. Test samples for presence of chlorine by placing a drop of sample on potassium iodide starch test paper. A blue color indicates the presence of chlorine. If chlorine is present, add a few crystals of ascorbic acid at a time, until a drop of sample procures no color on the test paper. Record in distillation log whether chlorine was or was not present.

7.1.1 For waters place 500 mL of sample, or an aliquot diluted to 500 mL, in the 1 liter boiling flask. Add 50 mL, of sodium hydroxide (5.1.1) to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.

7.1.2 For solids, accurately weigh a representative 1-5 portion of wet sample and transfer it to a boiling flask. Add 500 mL of distilled water. Shake or stir the sample so that it is dispersed. Add 50 mL, of sodium hydroxide (5.1.1) to the absorbing tube and dilute if necessary with distilled water to obtain an adequate depth of liquid in the absorber. Connect the boiling flask, condenser, absorber and trap in the train.

7.1.3 Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum so that approximately one bubble of air per second enters the boiling flask through the air inlet tube.

NOTE: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It will be necessary to

readjust the air rate occasionally to prevent the solution in the boiling flask from backing up into the air inlet tube.

7.1.4 Slowly add 25 mL concentrated sulfuric acid (5.1.4) through the air inlet tube. Rinse the tube with distilled water and allow the airflow to mix the flask contents for 3 minutes. Pour 20 mL of magnesium chloride solution (5.1.5) into the air inlet and wash down with a stream of water.

7.1.5 Heat the solution to boiling, taking care to prevent the solution from backing up into and overflowing from the air inlet tube. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, disconnect absorber and close off the vacuum source.

7.1.6 Drain the solution from the absorber into a 250 mL volumetric flask and bring up to volume with distilled water washings from the absorber tube.

7.2 Titrimetric Determination

7.2.1 If the sample contains more than 1 mg of CN, transfer the distillate, or a suitable aliquot diluted to 250 mL, to a 500 mL Erlenmeyer flask. Add 10-12 drops of the benzalrhodanine indicator.

7.2.2 Titrate with standard silver nitrate to the first change in color from yellow to brownish-pink. Titrate a distilled water blank using the same amount of sodium hydroxide and indicator as in the sample.

7.2.3 The analyst should be familiar with the end point of the titration and the amount of indicator to be used before actually titrating the samples. A 5 or 10 mL microburet may be conveniently used to obtain a more precise titration.

7.3 Manual Spectrophotometric Determination

7.3.1 Withdraw 20 mL or less of the solution from the flask and transfer to a 50 mL volumetric flask. If less than 20 mL is taken dilute to 20 mL with 0.25N sodium hydroxide solution (5.2.6). Add 15.0 mL of sodium phosphate solution (5.3.1) and mix.

7.3.1.1 Pyridine-barbituric acid method: Add 2 mL of chloramine-T (5.3.2) and mix. After 1 to 2 minutes, add 5 mL of pyridine-barbituric acid solu

tion (5.3.3) and mix. Dilute to mark with distilled water and mix again. Allow 8 minutes for color development then read absorbance at 578 nm in a 1 cm cell within 15 minutes. Spectrophotometer operating instructions are available in Appendix II.

7.3.2 Prepare a minimum of 3 standards and a blank by pipetting suitable volumes of standard solution into 100 mL volumetric flasks. NOTE: One calibration standard must be at the contract required detection limit (CRDL). To each standard, bring to 20 mL volume with 0.25N sodium hydroxide solution (5.2.6). Standards must bracket the concentration of the samples. If dilution is required, use the blank solution.

As an example, standard solutions could be prepared as follows:

mL of Standard Solution (1.0 = 1 ug CN)	Conc. ug CN per 50 mL
0	Blank
1.0	1
2.0	2
3.0	3
4.0	4
5.0	5
6.0	6

7.3.2.1 It is not imperative that all standards be distilled in the same manner as the samples. At least one standard (mid-range) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ± 15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

7.3.2.2 Prepare a standard curve by plotting absorbance of standard vs. cyanide concentrations (per 50 mL).

7.4 Percent Solids Determination Procedure

7.4.1 Immediately following the weighing of the sample to be analyzed for cyanide, add 5-10 g of sample to a tared weighing dish. Weigh and record the weight to the nearest 0.01 g.

7.4.2 Place weighing dish plus sample, with the cover tipped to allow for moisture escape, in a drying oven maintained at 103-105°C. Sample handling and drying should be conducted in a well-ventilated area.

7.4.3 Dry the sample overnight (12-24 hours) but not longer than 24 hours. If dried less than 12 hours, it must be documented that constant weight was attained. Remove the sample from the oven and cool in a dessicator with the weighing dish cover in place before weighing. Weigh and record weight nearest 0.01 g. Do not analyze the dried sample.

7.4.4 Duplicate percent solids determinations are required at the same frequency as are other analytical determinations.

7.4.5 For the duplicate percent solids determinations, designate one sample aliquot as the "original" sample and the other aliquot as the "duplicate" sample. Calculate dry weight using the results of the "original" sample aliquot.

NOTE: For the purpose of Section 7.4.3, drying time is defined as the elapsed time in the oven; thus raw data must record time in and out of the oven to document the 12 hour drying time minimum. In the event it is necessary to demonstrate the attainment of constant weight, data must be recorded for a minimum of two repetitive weigh/dry/dessicate/weigh cycles with a minimum of one hour drying time in each cycle. Constant weight would be defined as a loss in weight of no greater than 0.01 g between the start weight and final weight the last cycle.

7.4.6 Calculate percent solids by the formula below. This value will be used for calculating analytical concentration on a dry weight basis.

$$\% \text{ Solids} = \frac{\text{Sample Dry Weight}}{\text{Sample Dry Weight}} \times 100$$

7.5 Quantification of Results

7.5.1 Using the titrimetric procedure, calculate concentration of CN as follows:

$$\text{CN, mg/L} = \frac{(A-B)\ 1{,}000\ \text{mL/L}}{\text{mL orig. sample}} \times \frac{250\ \text{mL}}{\text{mL of aliquot titrated}}$$

where A = volume of $AgNO_3$ for titration of sample (1 mL = 1 mg Ag)
where B = volume of $AgNO_3$ for titration of blank (1 mL = 1 mg Ag)

and: 250 mL = distillate volume (7.1.5)
1000 mL = conversion mL to L
mL original sample (see 7.1.1)
mL of aliquot titrated (see 7.2.1)

7.5.2 If the colorimetric procedure is used, calculate the cyanide, in ug/L, in the original sample as follows:

$$\text{CN, ug/L} = \frac{A \times 1{,}000\ \text{mL/L}}{B} \times \frac{D}{C}$$

where A = ug CN read from standard curve (per 50 mL)
where B = mL of original sample for distillation (see 7.1.1)

where C = mL taken for colorimetric analysis (see 7.3.1)
where D = total volume of absorbing solution from the distillation, mL

and: 1,000 mL/L = conversion mL to L

7.5.3 Titration Calculation (For Soils):

$$\text{CN, mg/Kg} = \frac{(A-B) \times \frac{250\ \text{mL}}{\text{mL aliquot titrated}} \times 1{,}000\ \text{g/Kg}}{C \times \frac{\%\ \text{solids}}{100}}$$

where A = mL of $AgNO_3$ for titration of sample (1 mL = 1 mg Ag)
where B = mL of $AgNO_3$ for titration of blank (1 mL = 1 mg Ag)
where C = wet weight of original sample in g

7.5.4 Colorimetric (Soil) Calculation:

$$CN, mg/Kg = \frac{AxB}{CxDxS}$$

where A = ug CN read standard curve (per 50 mL)
where B = total volume of abosrbing solution from the distillation, mL
where C = wet weight of original sample in g
where D = volume of absorbing solution used in colorimetric test, mL
where S = decimal percent solids in sample

8.0 QUALITY CONTROL

8.1 For organizational purposes all activities for cyanide analysis have strict conventions for coding standards and batching of samples.

8.1.1 Standards - Standards are prepared from ACS grade potassium cyanide salt and dilutions thereof. All standard preparations are logged and coded. Our convention for coding all cyanide standards is as follows:

CN:xxxxxx:n:n
where CN = cyanide
where xxxxxx = date of preparation
where the first n = page # of standards prep. log
where the second n = preparation log book #

Stock cyanide preparation log book must contain the following information:

Date of preparation
Analyst's initials
Weight of KCN used
Raw data generated for standardization of solution
Normality of stock cyanide solution

All stock cyanide standards are labeled for storage, labeling must include concentration (1000 ug/ml cyanide), date solution made, weight of KCN used, normality of solution, page number of standards prep log, log book number and analyst's initials.

8.2 Instrument Calibration

Spectrophotometer must be calibrated daily or once every 24 hours and each time instrument is set up. The instrument standardization date and military time (24-hour clock) started and time completed must be included in the raw data. Calibration standards must be prepared fresh each time an analysis is to be made and discarded after use. Prep a blank and a minimum of three standards in graduated amounts in the appropriate range. NOTE: One calibration standard must be at the Contract Required Detection Limit (CRDL). Standards must bracket the concentration of the samples. At least one additional standard (mid-range) must be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable. If the distilled standard does not agree within ± 15 percent of the undistilled standards, the operator should find and correct the cause of the apparent error before proceeding.

8.3 Initial Calibration Verification (ICV)

Immediately after the spectrophotometer has been calibrated, the accuracy of the initial calibration shall be verified and documented by the analysis of EPA Initial Calibration Verification Solution for cyanide. This solution must be distilled. When measurements exceed the control limits for initial and continuing calibration verification the analysis must be terminated, the problem corrected, the instrument recalibrated, and the calibration reverified. The control limit for cyanide is ± 15 percent of the true value as set by EPA. The values for the initial and subsequent continuing calibration verifications shall be recorded on Form II-IN (see Appendix III).

8.4 Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analysis run a mid-range standard must be prepared to be used for continuing calibration verification and must be analyzed at a frequency of 10 percent of samples or every two hours, whichever is more frequent. The standard must be analyzed at the beginning of the run, after ten samples and after the last analytical sample. The same continuing calibration standard must be used throughout the analysis runs for a case of samples received. If the deviation of the continuing calibration is greater than the control limit (± 15 percent) of the true value, the instrument must be recalibrated and the preceding ten analytical samples or all analytical samples analyzed

since the last good calibration verification must be analyzed again. Information regarding the continuing verification of calibration shall be recorded on FORM II-IN.

8.5 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB) and Preparation Blank (PB) Analyses

A calibration blank must be analyzed for cyanide analysis at the beginning of the run, after every initial and continuing calibration verification at a frequency of 10 percent of samples or every two hours, whichever is more frequent and after the last analytical sample. The results for the calibration blanks shall be recorded on FORM III-IN (see Appendix III). If the magnitude (absolute value) of the calibration result exceeds the instrument detection limit (IDL), the result must be reported in ug/L on FORM III-IN (see Appendix III), otherwise report as IDL-U. If the absolute value blank result exceeds the contract required detection limit (CRDL), terminate analysis, correct problem, recalibrate and reanalyze the preceding ten analytical samples or all analytical samples analyzed since the last good calibration blank.

At least one preparation blank (or reagent blank) processed through the distillation procedure must be prepared and analyzed with every sample delivery group (SDG) or with each batch of samples digested whichever is more frequent. A batch is a group of samples prepared at the same time.

The first batch of samples in an SDG is to be assigned to preparation blank one, the second batch of samples to preparation blank two, etc. FORM III-IN, Figure 2.0. Each data package must contain the results of all the preparation blank analyses associated with the samples in that SDG.

This blank is to be repeated for each SDG and used in all analyses to ascertain whether sample concentrations reflect contamination in the following manner:

1) If the absolute value of the concentration of the blank is less than or equal to the CRDL, no correction of sample results is performed.

2) If the cyanide concentration in the blank is above the CRDL, the lowest concentration of cyanide in the associated samples must be 10x the blank concentration. Otherwise, all samples associated with the blank with the cyanide concentration less than 10x the blank concentration and above the CRDL must be redigested and reanalyzed (except for an identified aqueous soil field blank).

3) If the concentration of the blank is below the negative CRDL, then all samples reported below 10x CRDL associated with the blank must be redigested and reanalyzed.

The values for the preparation blank must be recorded in ug/L for aqueous samples and in mg/Kg for solid samples on FORM III-IN (see Appendix III).

8.6 Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the soil matrix on the digestion and measurement methodology. The spike is added prior to any distillation steps. At least one spike sample analysis must be performed on each group of samples of a similar matrix (i.e. water, soil) or for each sample delivery group, whichever is more frequent. Samples identified as field blanks cannot be used for spike sample analysis.

The analyte spike must be added in the concentration of 100 ug/L for both water and soil matrices. If the spike recovery is not within the limits of 75-125 percent, the data of all samples associated with that spike sample must be flagged with the letter "N" on FORM I-IN and V-IN (see Appendix III). An exception to this is when the sample concentration exceeds the spike level by a factor of four or more. In this case the data shall be reported unflagged even if the percent recovery does not meet the 75-125 percent recovery criteria.

When the pre-distillation spike recovery falls outside the control limits and the sample result does not exceed 4x the spike added, a post-distillation spike must be performed. Spike the unspiked aliquot of the sample at 2x the indigenous level or 2x CRDL, whichever is greater. Results of the post-distillation spike must be reported on FORM V (Part 2)-IN (see Appendix III).

Spike recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR-SR)}{SA} \times 100$$

where SSR = Spiked Sample Result
where SR = Sample Result
where SA = Spike Added

When sample concentration is less than the instrument detection limit, use SR=0 for calculating percent recovery. The spike sample results, samples results and percent recovery must be reported on FORM V-IN (see Appendix III).

8.7 Duplicate Sample Analysis (D)

One duplicate must be analyzed from each group of samples of a similar matrix type (i.e. water, soil). Duplicates cannot be averaged for reported on FORM I-IN (see Appendix III).

Duplicate sample analyses are required for percent solids. Samples identified as field blanks cannot be used for duplicate analysis. The relative percent differences (RPD) are calculated as follows:

$$RPD = \frac{(S-D)}{(S+D)/2} \times 100$$

where RPD = relative percent difference
where S = first sample value (original)
where D = second sample value (duplicate)

The results of the duplicate sample analyses must be reported on FORM VI-IN in ug/L for aqueous samples and mg/Kg dry weight basis for solid original and duplicate analyses (see Appendix III). A control limit of 20 percent for RPD shall be used for original and duplicate sample values greater than or equal to 5x the CRDL. A control limit of ± the CRDL must be used for sample values less than 5x the CRDL, and the absolute value of the control limit (CRDL) must be entered in the "control limit" column of FORM VI-IN (see Appendix III).

If one result is above the 5x the CRDL level and the other is below, use the + CRDL criteria. If both sample values are less than the IDL, the RPD is not calculated on FORM VI-IN. For solid sample or duplicate results <5x CRDL, enter the absolute value of the CRDL, corrected for sample weight and percent solids, in the "control limit" column. If the duplicate sample results are outside the control limits, flag all the data for samples associated with the duplicate sample with an asterisk (*) on FORMS I-IN and VI-IN.

8.8 Laboratory Control Sample (LCS) Analysis

Solid laboratory control samples supplied by the EPA must be analyzed for cyanide using the same sample preparations, analytical methods and QA/QC procedures employed for the EPA samples received. Percent solids determination is not required on the EPA-provided LCS sample. One solid LCS must be prepared and analyzed for every group of solid samples in a SDG, or for each batch of samples digested, whichever is more frequent.

All LCS results and percent recovery (%R) will be reported on FORM VII-IN (see Appendix III). If the results for the solid LCS fall outside the control limits established by the EPA, the analysis must be terminated, the problem corrected and the previous samples associated with that LCS redistilled and reanalyzed.

8.9 Instrument Detection Limit (IDL) Determination

Before any field samples are analyzed the instrument detection limits (in ug/L) must be determined at the start of the contract and at least quarterly (every three calendar months).

The instrument detection limits (in ug/L) shall be determined by multiplying by three, the average of the standard deviations obtained on three non-consecutive days from the analysis of a standard solution at a concentration 3x-5x the IDL, with seven consecutive measurements per day.

IDL's must be reported on FORM XI-IN (see Appendix III) submitted with each data package.

8.10 Summary of Quality Assurance/Quality Control

Listed below is the required QA/QC and order of events to be followed when analyzing samples for cyanide.

- Instrument Calibration - Blank, three standards in graduated amounts, one of which must be at the CRDL (non-distilled) include date, start analysis time (military time, 24-hour clock) and finish analysis time.

- Initial Calibration Verification (ICV) and Continuing Calibration Verification (CCV) - Analysis of distilled EPA known standard, (ICV), then a mid-range standard (not distilled) must be analyzed, (CCV). The CCV is then analyzed after every tenth sample and after the last analytical sample.

- Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) - Calibration blank (non-distilled) must be analyzed at the beginning of the run, after every initial and continuing calibration verification. Preparation blank - Consisting of distilled water processed through the distillation procedure must be analyzed with each batch of samples.

- Spiked Sample - One spike of a sample must be performed on each batch of samples of a similar matrix. Field blanks cannot be used as spikes.

- Duplicate Sample Analysis - One duplicate sample must be performed on each batch of samples of a similar matrix. Field blanks cannot be used as duplicates. For soil samples, duplicate sample analyses are required for percent solids.

- Laboratory Control Sample (LCS) - Solid laboratory control samples must be prepared and analyzed for every batch of solid samples analyzed.

The recorded absorbances for each standard must be checked to ensure that they are within the acceptable range. If standards do not fall within the acceptable range, call your supervisor. (Acceptable ranges for CN- standards are attached in Appendix IV).

8.11 Instrument Maintenance

8.11.1 Instrument maintenance logs are kept for each spectrophotometer.

8.11.2 An instrument performance log is kept for all spectrophotometers. The absorbance of the 2.0 ug standard is recorded weekly in order to monitor the lamp stability.

9.0 METHOD PERFORMANCE

9.1 A method detection limit (MDL) study performed on 03/14/90 yielded a 7.7 ug/L MDL using the Milton Roy Spectrophotometer.

9.2 The last 20 water sample matrix spikes (100 ug/L CN) average recovery was 97.0 percent as of 12/20/89.

9.3 The last 20 soil sample matrix spikes average recovery was 88.0 percent as of 12/11/89.

10.0 DATA REDUCTION AND DATA HANDLING

10.1 All distillation data including sample ID, sample volume, and volume distilled are recorded in each analyst's notebook. Separate logs for distillation, development and titration are kept for CLP protocols (see Appendix III).

10.2 All results are entered into LIMS for reporting and sample tracking purposes.

10.3 Technical Data Review

10.3.1 The technical data review includes verifying that samples were preserved and holding times were met. Absorbances of standards and batch quality control were within criteria

(see Appendix IV). All result calculations are verified and each log is checked for transcription error.

11.0 DOCUMENTATION OF PROBLEMS

11.1 Corrective Action Reports - must be filed if any problems were associated with the analysis (see Appendix III).

11.2 Case Narratives - should include any deviation from this SOP or any difficulties encountered in the procedure.

12.0 REFERENCES

1. Methods for "Chemical Analysis of Water and Wastes", March 1979, EPA publication #600/4-79-02.

2. "Operation RN Manual for Technicon Auto Analyzer IIC System", 1980. Technical publications #TA9-0460-00. Technicon Industrial Systems, Tarrytown, NY 10591.

3. "Users Guide for the Continuous Flow Analyzer Automation System", EMSL U.S. EPA, Cincinnati, OH (1981).

4. "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", USEPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, August 1977, Revised October 1980.

5. Op. cit. (#4), Methods 335.2.

APPENDIX I

(FIGURES)

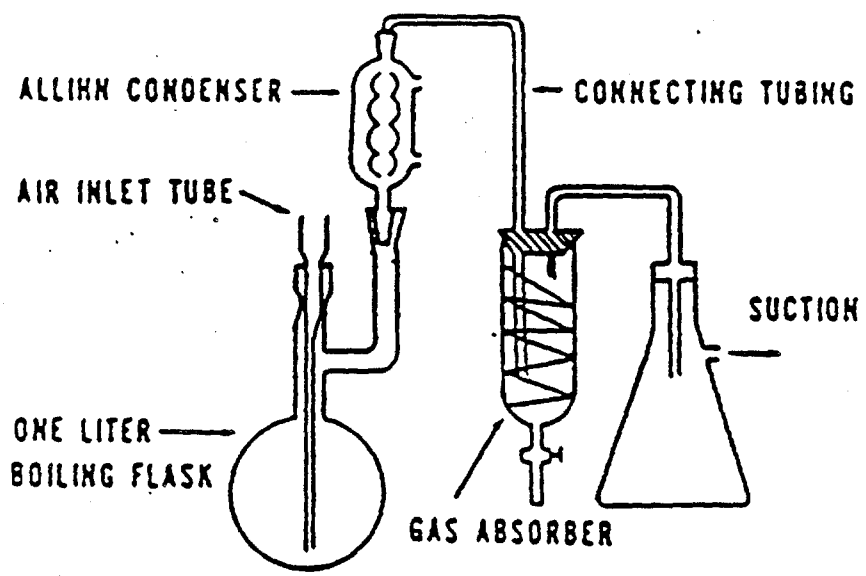

FIGURE 1

— Cyanide distillation apparatus

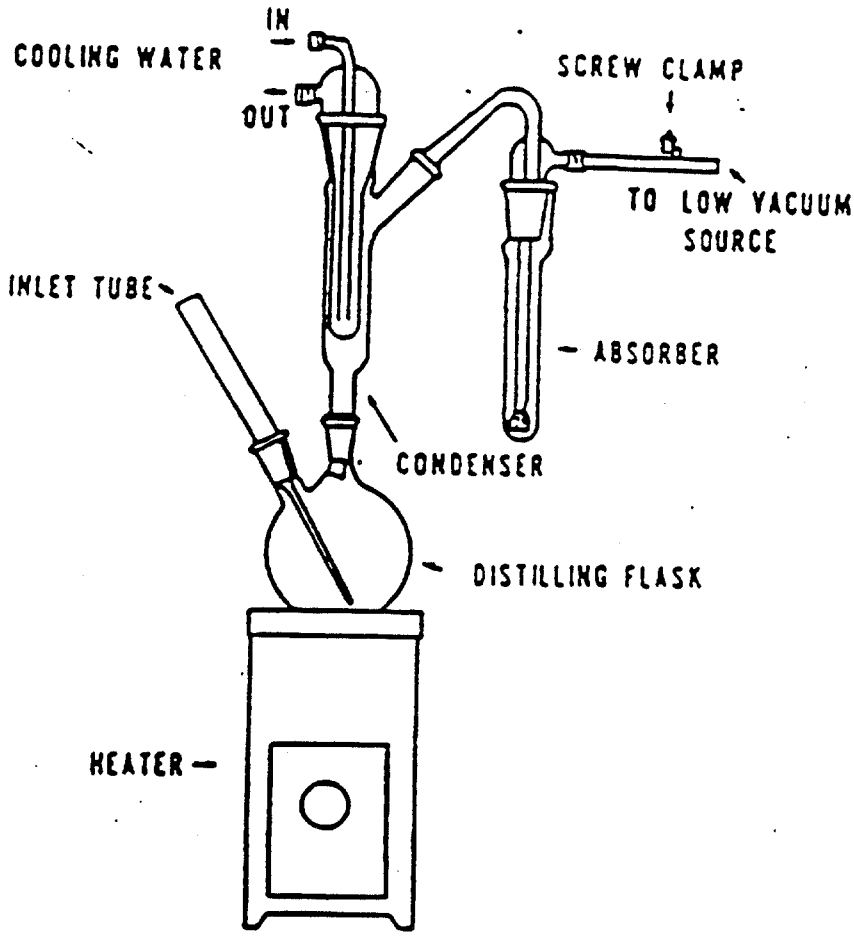

FIGURE 2

Cyanide distillation apparatus

APPENDIX II

INSTRUCTIONS FOR THE PERKIN-ELMER 08 SPECTROPHOTOMETER

1. Preparation

Power W damp - on (not D_2 damp)
Pull out Vis lamp knob
Slit - set at 2.0 nm
Response - Med
Suppression - Off
Scan - Off
NM - ignore (used with scan)
Wavelength - put at 500 - reads in absorbance or % T
Push % T (= % Transmittance)
w/o cells - adjust digital readout to 100% T with the knob on top (always reads ± when it's +)

Block front (sample) cell path with board - should read 0% T if not, adjust to 0% T with 0% T knob.
Remove board.
Back Cell = Blank
Front Cell = Sample - Lettering on cells should always point to the right when in the spec.

2. Operation

Abs - depress 0-2 scale
Clean cells; rinse out 1 time with blank and dry sides with Kimwipes
Put blank cells in front and back
Adjust wavelength to that required for the analysis.
With the blanks in, adjust 0 abs to read 0.

Now take the front cell out; rinse twice with the sample, fill with sample, put in the spec and take down the absorbance reading.
When done - just take out cells, clean them and leave on top to dry.

3. Shut Down

Switch off the W lamp and main power.

INSTRUCTIONS FOR THE MILTON ROY SPECTROPHOTOMETER

1. Turn the power on, and allow the instrument to warm up for 30 minutes.
2. Enter the desired wavelength and press "Go to λ"
3. Set the test mode to ABS.
4. Insert the sample cell with calibration blank into the spectrophotometer, close the sample door and press "Auto Zero".
5. Rinse the cuvette with sample twice and dry sides with kimwipes. Fill th cell with sample, place the cell back into the spec, close the sample door and record the absorbance.

APPENDIX III

INORGANICS QA/QC SUMMARY

Analyte:_____ Date:_____

Notebook Ref:_____

SPIKES

Lab Sample ID	Spike Result	Sample Result	Amount Added	Percent Recovery

DUPLICATES

ab Sample ID	Result 1	Result 2	Mean	RPD

BLANKS

Blank ID	Result

Samples in Batch:	Comments:

York Labs/CT

Classical Chemistry

CORRECTIVE ACTION REPORT/CASE NARRATIVE REPORT

Client:_____ Date:_____

Job/Case:_____ Analyst:_____

Sample(s):_____

Category of Problem:
__ Preservative
__ Sample Matrix
__ Missing Sample
__ Other:_____
__ Holding Time
__ Protocol
__ Communication

Description of Problem:_____

_____

_____

_____

_____

_____

Corrective Action Taken:_____

_____

_____

_____

Distribution: Lab Manager, Project Manager, Client Services,
(circle) QA Officer, Job Folder, Other:_____

301259

301260

York Laboratories
200 Monroe Turnpike
Monroe, CT 06468
(203) 261-4458
Lab Code YORK

Contract # 68-W8-0017
Case #
SAS #
SDG #
Page #

CYANIDE DISTILLATION LOG

Batch No.	Client	Date	YWC Sample ID	EPA Sample ID	Dist. App. No.	Sample Volume or Weight	pH >12.0	Cl_2 Present?	Volume Distillate	Sulfide Present	Initials	Comments

Reviewed by Classical Chemistry Supervisor

Date

York Laboratories
200 Monroe Turnpike
Monroe, CT 06468
(203) 261-4458
Lab Code YORK

Contract # 68-W8-0017
Case #
SAS #
SDG #
Page #

CYANIDE DEVELOPMENT LOG

Batch No.	Client	Date	YNC Sample ID	EPA Sample ID	Sample Volume or Weight	Percent Solids	Volume Distillate	Volume for Color Development	ABS at 578 nm	ug Cyanide	Total Cyanide	units, mg/Kg or ug/L	Initials	Comments

Standard Cyanide Code ID_____
Cyanide Curve Slope_____
Cyanide Curve y Intercept_____

Cyanide Curve Correlation_____
Time Analysis Start_____
Time Analysis End_____

_____
Reviewed by Classical Chemistry Supervisor

Date _____

York Laboratories
200 Monroe Turnpike
Monroe, CT 06468
(203) 261-4458
Lab Code York

Contract # 68-W8-0017
Case #_____
SAS #_____
SDG #_____
Page #_____

CYANIDE TITRATION LOG

Batch No.	Client	Date	YNC Sample ID	EPA Sample ID	Sample Volume or Weight	% Solids	Volume Distillate	Volume for Titration	mls AgNO3 used	Total Cyanide	Units, mg/Kg or ug/L	Initials	Comments

Standard Silver Nitrate Code ID_____

Normality of Silver Nitrate_____

_____ Date_____

Reviewed by Classical Chemistry Supervisor

U. S. EPA - CLP

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name: ______________________ Contract: __________

Lab Code: ______ Case No.: ______ SAS No.: ______ SDG No.: ______

SOW No. ______

EPA Sample No.	Lab Sample ID

Were ICP interelement corrections applied? Yes/No ___

Were ICP background corrections applied? Yes/No ___
If yes, were raw data generated before application of background corrections? Yes/No ___

Comments: ____

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Signature: ______________________ Name: ______________________

Date: ______________________ Title: ______________________

U.S. EPA - CLP

INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Lab Sample ID: _____

Level (low/med): _____ Date Received: _____

% Solids: _____

Concentration Units (ug/L or mg/kg dry weight): _____

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
	Cyanide				

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____

_____

_____

_____

U.S. EPA - CLP

2A

INITIAL AND CONTINUING CALIBRATION VERIFICATION

ıme: _____ Contract: _____

ɔde: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ıl Calibration Source: _____

ıuing Calibration Source: _____

Concentration Units: ug/L

/te	Initial Calibration True	Found	%R(1)	Continuing Calibration True	Found	%R(1)	Found	%R(1)	M
ınum									
ıony									
ıic									
ım									
um									
ıum									
.t									
ır									
esium									
ganese									
ry									
l									
assium									
ium									
r									
ım									
ium									
ium									
de									

ontrol Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

U.S. EPA - CLP

3
BLANKS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Preparation Blank Matrix (soil/water): _____

Preparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Prepa-ration Blank	C	M
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

FORM III - IN

7/88

301266

U. S. EPA - CLP

5A
SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

ab Name: _____ Contract: _____

ab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

atrix: _____ Level (low/med): _____

Solids for Sample: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

FORM V (Part 1) - IN

301267

U. S. EPA - CLP

5B

POST DIGEST SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix: _____ Level (low/med): _____

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									

Comments:

FORM V (Part 2) - IN

Rev. 2/89

301268

U.S. EPA - CLP

6
DUPLICATES

EPA SAMPLE NO.

me: _____ Contract: _____ | _____

de: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

(soil/water): _____ Level (low/med): _____

ds for Sample: _____ % Solids for Duplicate: _____

Concentration Units (ug/L or mg/kg dry weight): _____

alyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
uminum								
timony								
senic								
rium								
ryllium								
dmium								
lcium								
mium								
lt								
per								
con								
ad								
agnesium								
anganese								
ercury								
ickel								
Potassium								
elenium								
ilver								
Sodium								
hallium								
anadium								
inc								
yanide								

301269

FORM VI - IN

7/88

U.S. EPA - CLP

7

LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Solid LCS Source: _____

Aqueous LCS Source: _____

Analyte	Aqueous (ug/L) True	Found	%R	Solid (mg/kg) True	Found	C	Limits		%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
romium									
balt									
pper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

APPENDIX IV

Cyan-CLP Development - CN:051490:18.1 DTN 06/20/90

Standard	Acceptable Absorbance Range	Concentration Range
10 ug/L	+0.018 - +0.020	9.5 - 10.5 u/g/L
100 ug/L	+0.184 - +0.203	95.0 - 105 ug/L
300 ug/L	+0.552 - +0.610	285 - 315 ug/L
CCV (100 ug/L)	+0.165 - +0.223	85 - 115 ug/L
ICV6 (0789)	+0.062 - +0.084	79.9 - 115 ug/L
Distilled Standard:		
25 ug/250 mL	+0.165 - +0.223	85.0 - 115 ug/L
50 ug/500 mL	+0.329 - +0.446	85.0 - 115 ug/L
Spike:		
25 ug/250 mL	+0.145 - +0.242	75.0 - 125 ug/L
50 ug/500 mL	+0.291 - +0.484	75.0 - 125 ug/L

CN:051490:18:1 Slope: 0.0969 y-int = -0.0003 Cov. Coef. = 0.9994

Calculation: $ug = \frac{absorbance}{0.0969} - (-0.0003)$

For Liquids: $CN(mg/L) = \frac{ug \times vol.\ distilled\ (mL)}{Sample\ vol.\ (mL) \times vol.\ color\ (mL)}$

For Soils: $CN(mg/Kg) = \frac{ug \times vol.\ distilled\ (mL)}{Sample\ wt.\ (g) \times \%\ solids\ (decimal) \times vol.\ color\ (mL)}$

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

TRACKING METALS AND CYANIDE SAMPLE ANALYSIS

IN:050189:1

Whippany, New Jersey 201-428-8181
Schaumburg, Illinois 708-705-0740
Miramar, Florida 407-989-0928
Essex Junction, Vermont 802-878-5138
N. Billerica, Massachusetts 617-272-5212
Cary, North Carolina 919-677-0090

STANDARD OPERATING PROCEDURE
TRACKING METALS AND CYANIDE SAMPLE ANALYSIS

Abstract

This Standard Operating Procedure addresses the tracking of samples for metals and cyanide analysis from sample receipt through analysis and final reporting. It is meant to provide an analytical scheme of those forms required to adequately trace a sample's path through the laboratory.

SAMPLE TRACKING

As the process of sample tracking involves several different departments, this SOP is broken into subgroups based upon each department's responsibilities. Included in this outline are references to the most current SOP appropriate to each set of forms. The user is instructed to refer to these SOP's for further elaboration on the specific use and accurate generation of each form.

Sample Control - Sample Receipt and Log-In

Figure 1.0: IEA's log-in record - Upon receipt samples are logged-in and assigned an IEA-CT job and sample number.

Figure 2.0: pH log - The pH of all samples is checked and recorded.

Figure 3.0: Sample control record in-house custody sheet - A sample control chain of custody record is generated which includes the refrigerator location of the samples.

Figure 4.0: Sample disposal/archive request - A sample disposal request form is generated.

Figure 5.0: SDG TR - A sample delivery group traffic report cover sheet is generated and sent to all appropriate departments.

At this point sample tracking diverges based upon analysis required (i.e. metals and/or cyanide) and will be addressed separately.

Metals - Standard and Sample Preparation Logs

Figure 6.0: AS sample tracking sheet - The case is logged-in and ready for batch generation in the metals department.

Figure 7.0: Metals sample preparation log - ICP and/or furnace preparation is started for either:*

- 7a: Waters
- 7b: Soils

Figure 8.0: TCLP

Figure 9.0: Mercury sample preparation log - Mercury preparation is performed for either water or soil.*

Figure 10.0: Percent solids determination - If soils are being prepared, percent solids are determined.*

Figure 11.0: ICP analysis is performed:

- 11a: ICP analytical run log
- 11b: QC checklist - JA61 ICAP
- 11c: QC checklist - PE6500 ICP (if required)

Figure 12.0: Furnace analysis is performed:

- 12a: Analytical run log/raw data worksheet
- 12b: Furnace checklist

Figure 13.0: Mercury analysis is performed:

- 13a: Mercury run log
- 13b: Mercury data sheet
- 13c: Mercury analytical checklist

Figure 14.0: Metals chain of custody record - This chain of custody is signed as each task is completed with final approval of the supervisor upon job completion.

Figure 15.0: QCAR - A final quality control approval report is signed as each task is completed and placed in job folder. Supervisor performs the final review of this form when the job is completed.

Cyanide - Analysis of Environmental Standards for Total Cyanide

Figure 16.0: Preparation is set-up initially in the laboratory notebook as follows:

- 16a: Sequence of preparation
- 16b: Color, clarity and tests for oxidizing agents and sulfides are performed.*

Figure 17.0: A cyanide distillation log is generated.

Figure 18.0: Cyanide development log is generated during analysis.

Figure 19a: If required (i.e. cyanide levels >1.0 mg/L) a cyanide titration log is generated.

Figure 19b: Cyanide auto-analyzer development log is generated.

Figure 20.0: A quality control approval report is signed and dated as each task is completed. Supervisor performs final review and signs report upon job completion.

*Note: See Figure 3.0 - This chain of custody is signed when samples are removed/returned to the refrigerator.

CLP Deliverables

Figure 21.0: CLP review checklist - For jobs requiring CLP deliverables the data entry clerk generates and signs a CLP review checklist after deliverables generation.

Attachment 1: Final reports form required for CLP deliverables.

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[redacted]
an environmental testing company
iea 200 Monroe Turnpike
[redacted] Monroe, Connecticut 06468

FIELD CHAIN OF CUSTODY RECORD

IEA JOB # : _____

Client : _____

Project ID : _____

IEA Project Mgr : _____

Rush: Y N TAT: __ Due Date: _____

Page __ of

Protocols : _____

Deliverables : _____

Bottle Set #	Client Sample ID	Date/Time Sampled	*Matrix	Lab ID	TESTS: Bottle Type & Preservative / Field Filt?										QC Required Y/N	Comments
		/				Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	Y N	
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
		/														
AS CC EX GC BNA VO				Ref. #												

* Matrix Codes

A - Air
AQ - Aq
C
D
S - Soil
SL - Sludge
W - Wipe
O - Other

Bottles Prepped By : _____
Date/Time : _____ / _____
Signature : _____

Samples Collected By : _____
Date/Time : _____ / _____

Bottles Rec'd By : _____
Date/Time : _____ / _____
Signature : _____

Rec'd In Lab By : _____
Date/Time : _____ /

Shipper : _____
Air Bill #: _____
Outgoing Seal ID#:
Intact On Field Receipt? N
Incoming Seal ID#:
Intact On Lab Receipt N

IEA, INC. SAMPLE PRESERVATIVE RECORD

JOB NUMBER:
CLIENT:
CLIENT PROJECT:

LAB NUMBER	PRESERVATIVE	PH	KI	ADJUSTMENT	INITIALS	DATE

FIGURE 2.0

301280

IEA, INC.
IN-HOUSE CUSTODY SHEET

Client:

Custody Seal	present/absent	DATE:
	intact/not intact	SAMPLE CUSTODIAN SIGNATURE:
Chain of Custody	present/absent	JOB NUMBER:
Sample Tags	present/absent	CASE NUMBER:
	listed/not listed	AIRBILL NUMBER:
SMO Forms	present/absent	SAMPLE NUMBERS:

LABORATORY SAMPLE NUMBER	REMOVED BY	DATE AND TIME REMOVED	REASON	DATE AND TIME RETURNED	RETURNED TO REF. #
					REF#

FIGURE 3.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

SAMPLE DISPOSAL/ARCHIVE REQUEST

CLIENT: _____

JOB #: _____ DATE REQUESTED: _____

DATE OF INVOICE: _____ REQUESTED BY: _____

REF. # _____

ITEM	APPROXIMATE QUANTITY	ARCHIVE DISPOSAL CODE[1]	DISPOSED OF BY	DATE
Soils/Sediments				
Waters/Leachates				
Extracts				
Other - _____				
_____				
_____				

[1] Codes: 01 - dispose of in solid waste drums
02 - dispose of in aqueous waste drums
03 - archive for 60 days then dispose of in solvent waste drums
04 - archive for 180 days - John Cullt cills TRA fo instruction
05 - special instructions

FIGURE 4.0

301281

SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

Lab Name: _____ Contract No.: 68- _____

Lab Code: _____ Case No.: _____ SAS No.: _____-___

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ Sample Receipt Date: _____ (MM/DD/YY)
(Lowest EPA Sample Number in first shipment of samples received under SDG)

Last Sample in SDG: _____ Sample Receipt Date: _____ (MM/DD/YY)
(Highest EPA Sample Number in last shipment of samples received under SDG)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____
Sample Custodian Date

FIGURE 5.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

ATOMIC SPECTROSCOPY SECTION SAMPLE TRACKING SHEET								
Case/SAS #	YWC Job #	YWC/Contract Sample #	SDG #	Date Received	Date Complete	Date to Purge	Purged	Analysis

FIGURE 6.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

METALS SAMPLE PREP LOG - WATER

Calibration of eppendorf
Case/SAS_____ Low/Med/Std
ICAP/Furnace SDG #:_____

IEA ID	IFB ID	Vol i (ml)	Vol f (ml)	Phys. Desc.* before/after		Color Key*
				1- /	2- /	R = red
				1- /	2- /	B = blue
				1- /	2- /	Y = yellow
				1- /	2- /	G = green
				1- /	2- /	O = orange
				1- /	2- /	V = violet
				1- /	2- /	C = colorless
				1- /	2- /	Bn = brown
				1- /	2- /	Gy = gray
				1- /	2- /	Bl = black
				1- /	2- /	Clarity*
				1- /	2- /	CL = clear
				1- /	2- /	C = cloudy
				1- /	2- /	O = opaque
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	

*Each physical description involves 1-Color and 2-Clarity both before and after

QA/QC

IEA ID		IFB ID	Volume i (ml)	Volume f (ml)	QC Addition
PB					
LCS					
	D				
	S				

Prepared by:_____ Supervisor:_____
Date:_____ Date:_____
Accepted by:_____ Date:_____
ICP: HNO_3_____ HCl_____ H_2O_2_____ Furnace: HNO_3_____ H_2O_2_____
Page ___ of ___

FIGURE 7A

IEA Environmental Testing Company
300 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

METALS SAMPLE PREP LOG - SOIL

Case/SAS_____ Low/Med/Std SDG #:_____
Calibration:_____ ICAP/Furnace

IEA ID	IFB ID	Weight (g)	Volume (ml)	Physical Description* before/after				Color Key*
				1- /	2-	3-	4-	R = red
								B = blue
								Y = yellow
								G = green
								O = orange
								V = violet
								C = colorless
								Bn = brown
								Gy = gray
								Bl = black
								Clarity*
								Cl = clear
								C = cloudy
								O = opaque
								Texture*
								F = fine
								M = medium
								C = coarse

*Each physical description involves 1-Color (before and after), 2-Texture (before), 3-Clarity (after), and 4-Artifacts (yes or no description)

QA/QC

IEA ID		IFB ID	Weight (g)	Volume f (ml)	QC Addition
PB					
LCS					
	D			-	
	S				

Prepared by:_____ Supervisor:_____
Date:_____ Date:_____
Accepted by:_____ Date:_____
ICP: HNO_3_____ HCl_____ H_2O_2_____ Furnace: HNO_3_____ H_2O_2_____

FIGURE 7B

301285

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

METALS SAMPLE PREP LOG - TCLP

Calibration of eppendorf
Case/SAS_____ Low/Med/Std ICAP/Furnace SDG #:_____

IEA ID	IFB ID	Vol i (ml)	Vol f (ml)	Phys. Desc.* before/after		Color Key*
				1- /	2- /	R = red
				1- /	2- /	B = blue
				1- /	2- /	Y = yellow
				1- /	2- /	G = green
				1- /	2- /	O = orange
				1- /	2- /	V = violet
				1- /	2- /	C = colorless
				1- /	2- /	Bn = brown
				1- /	2- /	Gy = gray
				1- /	2- /	Bl = black
				1- /	2- /	Clarity*
				1- /	2- /	CL = clear
				1- /	2- /	C = cloudy
				1- /	2- /	O = opaque
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	
				1- /	2- /	

*Each physical description involves 1-Color and 2-Clarity both before and after

QA/QC

IEA ID		IFB ID	Volume i (ml)	Volume f (ml)	QC Addition
PB					
LCS					
	S				
	S				
	S				

Prepared by:_____ Supervisor:_____
Date:_____ Date:_____
Accepted by:_____ Date:_____
ICP: HNO_3_____ HCl_____ H_2O_2_____ Furnace: HNO_3_____ H_2O_2_____

FIGURE 8.0

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

METALS WATER/SOIL PREPERATION LOG - MERCURY

Top Loading Balance Calibration________________
Analytical Balance Calibration________________

Page ___ of ___

IEA ID	IFB ID	Wi(g) Vi(ml)	Vf(ml)	BOD ID	QA/QC				
					ID	Wi/Vi	QC add	Vf(ml)	BOD ID

QA/QC

Standard ID = ________________

ICVS ID = ________________

CCV ID = ________________

LCS ID = ________________

Spike ID = ________________

Prepared by:________________ Accepted by:________________
Date:________________ Date:________________
Lot #: Hydroxylamine Hydrochloride____ Stannous Chloride____ KMnO4____
H2SO4________ HNO3________ K2O8S2________ NaCl________

FIGURE 9.0

301287

% SOLIDS DETERMINATION

Case/SAS/Batch No:_____ SDG No:_____

Calibration:_____ Oven Temp (°C):IN

$$\% \text{ Solids} = \frac{\text{Dry Wt}}{\text{Wet Wt}} \times 100$$

IEA ID	IFB ID	Dish Wt (g) W_{ds}	Sample Wet Wt (g) W_w* (minus dish)	Sample Dry Wt (g) plus dish	Sample Dry Wt (g) minus dish	% Sol

Time in:_____ Time out:_____

*Notes: 1) A minimum of 5.00 g of wet sample should be taken for this analysis.
2) Do not exceed a 24 hour drying time.
3) One sample must be determined in duplicate (see ICP prep for duplicat selection).

Prepared by:_____ Witnessed by:_____

Date:_____ Date:_____

FIGURE 10.0

Rename 66 sample

AutoSamoler Report Table: A042889 Fri 04-28-89 04:28:27 PM Page 1

T[illegible]e Name: A042889 Autosamoler Tvoe: TYPE 222
S[illegible]ple Positions: 206/240 QC Positions: 0/14 # Sets: 1
Rinse Station location is rack -1. pos. -1.

--- Racks ---

Rack #	Tvpe	Usage	#Pos Left	Analvses/Pos
1	Small Tubes	Samples	38	1
2	Small Tubes	Samples	60	1
3	Small Tubes	Samples	60	1
4	Small Tubes	Samples	48	1
5	Large Tubes	STD/QC/BLANK	0	10

--- Sample Sets ---

Set #	Description	Method	#Pos	Rack #	Start Pos
1		IEA LAB	22	1	1

Rack #1

Pos	Row	Col	Sample Name	Set #	#Used	Tvpe
1	1	1	RINSE	1	-NA-	Sample
[illegible]	1	2	1.0001.000PBS	1	-NA-	Sample
	1	3	1.0200.200LCSS	1	-NA-	Sample
	1	4	1.0600.200747001	1	-NA-	Sample
5	2	1	1.0500.200747001D	1	-NA-	Sample
6	2	2	1.0600.200747001S	1	-NA-	Sample
7	2	3	1.0700.200747002	1	-NA-	Sample
8	2	4	1.0100.200747003	1	-NA-	Sample
9	3	1	1.1100.200756002	1	-NA-	Sample
10	3	2	1.0100.200756002D	1	-NA-	Sample
11	3	3	1.1200.200756002S	1	-NA-	Sample
12	3	4	1.0400.200756003	1	-NA-	Sample
13	4	1	1.0500.200756004	1	-NA-	Sample
14	4	2	1.0100.200756005	1	-NA-	Sample
15	4	3	1.0001.000PBW	1	-NA-	Sample
16	4	4	1.0001.000LCSW	1	-NA-	Sample
17	5	1	1.0001.000756001	1	-NA-	Sample
18	5	2	1.0600.200747001P Sb=120	1	-NA-	Sample
19	5	3	~~1.1100.200756002P~~ VOID 5/11/89	1	-NA-	Sample
20	5	4	1.0005.000747003 L	1	-NA-	Sample
21	6	1	1.0005.000756002 L	1	-NA-	Sample
22	6	2	Sample-22	1	-NA-	Sample
(23...60	Not Used)					

Rack #2

Pos	Row	Col	Sample Name	Set #	#Used	Tvpe
[illegible]..60	Not Used)					

FIGURE 11A

301239

Rack #3

Pos	Row	Col	Sample Name	Set #	#Used	Type
1...60	Not Used)					

Rack #4

Pos	Row	Col	Sample Name	Set #	#Used	Type
1...48	Not Used)					
49	13	1	CCB3	-NA-	1	QC Standard
50	13	2	CCV3	-NA-	1	QC Standard
51	13	3	CCV3A	-NA-	1	QC Standard
52	13	4	ICSABF	-NA-	1	QC Standard
53	14	1	ICSAF	-NA-	1	QC Standard
53	14	1	ICSAF	-NA-	1	QC Standard
55	14	3	CRIF	-NA-	1	QC Standard
56	14	4	CCB2	-NA-	1	QC Standard
57	15	1	CCV2	-NA-	1	QC Standard
58	15	2	CCV2A	-NA-	1	QC Standard
59	15	3	CCB1	-NA-	1	QC Standard
60	15	4	CCV1	-NA-	1	QC Standard

ack #5

Pos	Row	Col	Sample Name	Set #	#Used	Type
1	1	1	STD2	-NA-	1	Standard
2	1	2	STD3	-NA-	1	Standard
3	2	1	STD4	-NA-	1	Standard
4	2	2	STD5	-NA-	1	Standard
5	3	1	STD6	-NA-	1	Standard
6	3	2	STD7	-NA-	1	Standard
7	4	1	STD1	-NA-	1	Standard
8	4	2	ICV1	-NA-	1	QC Standard
9	5	1	ICV1A	-NA-	1	QC Standard
10	5	2	ICB	-NA-	1	QC Standard
11	6	1	CRI1	-NA-	1	QC Standard
12	6	2	ICSAI	-NA-	1	QC Standard
3	7	1	ICSABI	-NA-	1	QC Standard
4	7	2	CCV1A	-NA-	1	QC Standard

FIGURE 11A (continued)

301290

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4468
FAX (203) 268-5346

ICP APPROVAL CHECKLIST

Batch:_____ Analyst:_____

Autosampler Table #:_____ Date:_____

Approval of Senior Analyst:_____

Set-Up

1) Appropriate standards for all analytes? _____

2) Is ICV2 or ICV3 necessary? i.e. EPTOX? _____

3) Autosampler table in correct order? _____
STDS, ICV's, ICB, CRI, ICSA, ICSAB

4) Ten samples between CCV's? within 2 hours? _____

5) All analytical spikes made? enter prep code _____

6) For CLP protocol - room for postdigests? _____

7) Room for serial dilutions? _____

8) Room at end of run for analyte checks? _____

9) Print out (2) of table - dated and initialed? _____

Run

10) Initial QC pass? _____

11) Prep blanks and LCS pass? _____
If no, corrective action reported? _____

12) Spike recoveries calculated? _____

13) Post digests made? _____

14) Serial dilutions made? _____

15) Continuing calibrations pass? _____

16) Did saturation occur? _____

17) High analytes noted? _____
If yes, analyte check added to end of run? _____

18) Final QC pass? _____

FIGURE 11b

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

Reporting

19) CLP protocol - diskette made? _____

20) Renamed sample file? _____

21) IEC's calculated? _____

22) Regular work reported on sheets? _____

23) Chain of custodies signed and dated? _____

24) Any further scans necessary? _____

25) CLP - copies of raw data required? _____
If yes, copies made? _____

26) CLP - all runs labeled with regard to their applicability? _____

27) Were any problems noted requiring case narrative? _____

FIGURE 11b (continued)

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

6500 ANALYTICAL APPROVAL CHECKLIST

CASE/BATCH:_____ ANALYST:_____ DATE:_____

LOGBOOK PAGE:_____

1) Standardization acceptable? _____

2) Initial, continuing, final QC acceptable? _____

3) a) QA (PB, LCS) acceptable? _____
   b) If no, corrective action reported? _____

4) Weights, percent solids, volumes on data sheets? _____

5) Dilutions noted on data sheets? _____

6) IEC's

   a) Present - determined and calculated? _____
   b) Not required? _____

7) Data calculated? _____

8) Data reported? _____

9) Run recorded in logbook? _____

10) Copies of raw data required? _____
    If yes, copies made? _____

11) Were any problems noted requiring a case narrative? _____

FIGURE 11c

IEA
200 Monroe Turnpike
Monroe CT 06468 (203) 452-8200

Std Prepped Date:___/___/___
Start Run Time :_____

Date:___/___/___

FURNACE DATA SHEET

Element:___ I.D. Wt. File:_____ Instrument:_____ Analyst:_____

Cup	Element	Sample ID	% Solids	Wt. Vol/ Vol	Dil. Factor	Sample Recovery	% Recovery	Flags	Value ug/L, mg/L
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									
31									
32									
33									
34									
35									
36									
37									
38									
39									
40									

FIGURE 12A

ID/WT FILE: _____ BATCH: _____

INSTRUMENT: _____ DATE: _____

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

ANALYST: _____

ANALYTES: _____

	FURNACE ANALYTICAL APPROVAL CHECKLIST	Analyst
1)	Run log generated, signed, dated & filed	_____
2)	Standardization acceptable	_____
3)	Initial continuing & final QC acceptable	_____
4)	Analytical spike criteria met	_____
5)	PB LCS pass: a) yes b) no, corrective action report submitted	_____ _____
6)	Reruns noted	_____
7)	RSD's less than 20% (sample ≥CRDL)	_____
8)	Initial volume/wt, final volumes and percent solids recorded on calculated sheets	_____
9)	Results calculated	_____
10)	Sensitivity check recorded in logbook	_____
11)	Results reported	_____
12)	Analytical run times recorded on raw data	_____
13)	Appropriate copies made of data	_____
14)	Were any problems noted requiring a case narrative	_____

FIGURE 12B

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

MERCURY RUN LOG

Matrix: water/soil
Job #:_____

Analyst:_____
Date:_____
Page ___ of ___

Sample ID	Bottle ID	Time	Sample ID	Bottle ID	Time

FIGURE 13A

301296

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

Parameter: Mercury Matrix:_____ Job #s':_____

Sample/ Standard	Pk. Ht. (in mm)	Calculated (ug/l)	Initial Vol(ml)	Final Vol(ml)	Value Rptd. (ug/l)	QA Concentration units (ug/l)
						Sample Number_____
						Concentration _____
						_____
						RPD _____
						Spike
						True Conc._____
						Found Conc._____
						Percent Recovery_____
						ICV
						True Conc._____
						Found Conc._____
						Percent Recovery_____
						C of C_____
						Analyst_____
						Date_____
						Reviewed By:_____
						Page ___ of ___

FIGURE 13B

301297

CASE/BATCH: _____ ANALYST: _____ DATE: _____

MERCURY ANALYSIS APPROVAL CHECKLIST

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

1) Coefficient of correlation ≥ 0.995 _____

2) ICV, ICB pass? _____

3) CCVs, CCBs pass? _____
   If no corrective action reported? _____

4) QC frequency met?
   a) CCVs, CCBs every 10 samples _____
   b) Ending CCV, CCB present _____

5) PBs, PCSS pass?
   a) PB, LCSS run for each batch/SDG? _____
   b) If fail, corrective action reported? _____

6) Spike recoveries pass? _____
   a) Spike frequency met per batch/ SDG/Matrix? _____
   b) If fail, corrective action reported? _____
   c) Analytical spikes performed for E.P. toxicity/EPDW/TCLP? _____

7) V_I/W_I, V_f and percent solids recorded? _____

8) All data calculated? _____

9) Data entered on final report sheets? (non-CLP) _____

10) Appropriate number of copies made? _____

11) Original preplog and analysis log placed in log books? _____

12) Copy of run log attached? _____

13) Were any problems noted requiring a Case Narrative? _____

FIGURE 13C

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

CHAIN OF CUSTODY
ATOMIC SPECTROSCOPY DEPARTMENT

Job Number _____ Sample Numbers _____

WATER - SOIL - SLUDGE - EPTOX/TCLP

I confirm that I have performed the preparation below following SOP guidelines and authorize the release of this preparation:

Sample Prep _____ _____

_____ _____ ICP/FLME

_____ _____ FURN

_____ _____ MERCURY

Chemist Date(s)

I confirm that I have performed the analysis below following SOP guidelines and authorize the release of all associated data:

Analysis _____ _____ ICP

_____ _____ FLAME

_____ _____ FURN

_____ _____ MERCURY

Chemist Date(s)

I have reviewed and authorize the release of this job:

Complete _____ _____

Supervisor Date

Batch Assignment _____

FIGURE 14.0

301299

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

JOB/SDG:_____ CASE/BATCH #:_____

REPORT LEVEL_____

QCAR - METALS

		Initials/Date	Initials/Date
1.	Chain of custody signed		_____
	a) ICP prep released	_____	
	b) Furn prep released	_____	
	c) Mercury prep released	_____	
2.	Data reviewed and reported		_____
	a) ICP checklist completed	_____	
	b) Furn checklist completed	_____	
	c) Mercury checklist completed	_____	
3.	All forms complete		_____
4.	Batch QC reviewed and reported		_____
	a) ICP	_____	
	b) Furnace	_____	
	c) Mercury	_____	
5.	All problems noted in case narrative		_____
	a) ICP	_____	
	b) Furnace	_____	
	c) Mercury	_____	
6.	Case narrative complete		_____
7.	Release of data approved		_____
8.	Chain of custody complete		_____

FIGURE 15.0

301300

TITLE STAIN - CLP

Project No.

Book No.

From Page No.

	COLOR		CLARITY			
	BEFORE DISTILLATION	AFTER	BEFORE DISTILLATION	AFTER	KI PANEL AS IS	AFTER
	SPECKLESS	SPECKLESS	CLEAR SLIGHT		-	-
	"	"	CLEAR	CLEAR	-	-
	TAN	TAN	OPAQUE	OPAQUE	-	-
	YELLOW	YELLOW	"	"	-	-
105	BLACK	YELLOW	"		-	-
	"	"	"	"	-	-
	"	"	"	"	-	-
	"	"	"		-	-

To Page No.

Read & Understood by me.	Date 7/19	Invented by	Date
		Recorded by	

FIGURE 16A

301301

TITLE Cyan. Calc

Project No._____
Book No._____

COPY

rom Page No.___

Sample #	Sample wt/vol	% Solids	Vol dil	Vol for color	Abs @ 579 nm	µg	mg/L
RECOVERY - 25µg				250 ml			
ICV-C							
[illegible]	4.99 g						
[illegible]	5.13 g						
-005	4.99 g						
-006	5.00 g						
-007	5.08 g						
-008	5.01 g						

Sample #	Pan wt	Pan wt + wet Sample	Pan wt + Dry Sample	% Solids TS
[illegible]	1.27 g	6.69 g	5.64 g	.8564
[illegible] D	1.30 g	5.97 g	~~4.72 g~~	.7357
[illegible]	1.29 g	6.64 g	4.99 g	.6916
[illegible]	1.27 g	6.04 g	2.64 g	.2914
[illegible]	1.29 g	7.11 g	6.24 g	.8505
[illegible]	1.29 g	6.95 g	5.52	74.3%
[illegible]	1.33 g	7.85 g	6.23 g	.7945

To Page No. ...

nessed & Understood by me.	Date 4/14	Invented by	Date
		Recorded by bbn	

FIGURE 16A

301302

IEA
200 Monroe Turnpike
Monroe, CT 06468
(203) 452-8200
Lab Code IEA

CYANIDE DISTILLATION LOG

Case #_____
SAS #_____
SDG #_____
Page#_____

Batch No.	Client	Date	IEA Sample ID	EPA Sample ID	Dist. App. No.	Sample Volume or Weight	pH >12.0	Cl2 Present?	Volume Distillate	Sulfide Present?	Initials	Comments

FORM I V O

301303

_____
Reviewed by Classical Chemistry Supervisor

Date_____

IEA
200 Monroe Turnpike
Monroe, CT 06468
(203) 452-8200
Lab Code IEA

CYANIDE DEVELOPMENT LOG

Case #_____
SAS #_____
SDG #_____
Page#_____

Batch No.	Client	Date	IEA Sample ID	EPA Sample ID	Sample Volume or Weight	% Solids	Volume Distillate	Volume for Color Development	ABS at 578 nm	ug Cyanide	Total Cyanide	Units, mg/Kg or ug/L	Initials	Comments

Standard Cyanide Code ID_____

Cyanide Curve Slope_____

Cyanide Curve y Intercept_____

Cyanide Curve Correlation_____

Time Analysis Start_____

Time Analysis End_____

_____ Date

Reviewed by Classical Chemistry Supervisor

IEA
200 Monroe Turnpike
Monroe, CT 06468
(203) 452-8200
Lab Code IEA

CYANIDE AUTO-ANALYZER DEVELOPMENT LOG

Case #_____
SAS #_____
SDG #_____
Page #_____

Batch NO.	Client	Date	IEA Sample ID	EPA Sample ID	Sample Volume or Weight	Percent Solids	Volume Distillate	Cyanide Distillate Conc. (ug/L)	Total Cyanide	units mg/Kg or ug/L	Initials	Comments

Standard Cyanide Code ID_____
Cyanide Curve Slope_____
Cyanide Curve y Intercept_____

Cyanide Curve Correlation_____
Time Analysis Start_____
Time Analysis End_____

_____
Reviewed by Classical Chemistry Supervisor

Date_____

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5348

Contract # 68-W8-0017
Case #_____
SAS #_____
SDG #_____
Page #_____

CYANIDE TITRATION LOG

Batch No.	Client	Date	YMC Sample ID	EPA Sample ID	Sample Volume or Weight	% Solids	Volume Distillate	Volume for Titration	mls AgNO3 used	Total Cyanide	Units, mg/Kg or ug/L	Initials	Comments

Standard Silver Nitrate Code ID_____

Normality of Silver Nitrate_____

_____ Date_____

Reviewed by Classical Chemistry Supervisor

EPA LABORATORIES

CLASSICAL CHEMISTRY
QUALITY CONTROL APPROVAL REPORT

JOB # : 3089-0790
CLIENT : EPA

REPORT LEVEL : CLP

I. ANALYTICAL CHECKS

ANALYTE	SAMPLE NUMBERS	METHOD QC ACCEPTABLE	DATE	SAMPLE QC ACCEPTABLE	DATE
CYAN-CLP	001\010				

	APPROVAL	DATE
II. CHAIN OF CUSTODY COMPLETE :	_____	_____
CASE NARRATIVE COMPLETE :	_____	_____
QC DOCUMENTATION COMPLETE :	_____	_____

III. FINAL APPROVAL : _____ _____
GROUP LEADER DATE

PAGE ___ of ___

FIGURE 20.0

301307

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

CLP REVIEW CHECKLIST

Case/Batch #:_____ Date:_____

SDG/Job #:_____ Analyst:_____

All ICAP data has been calculated and copied by analyst _____

All Furnace data has been calculated and copied by analyst _____

All Mercury data has been calculated and copied by analyst _____

All Cyanide data has been calculated and copied by analyst _____

Raw data in job folder is as follows: ICAP analysis with autosampler table _____

Furnace analysis with run log _____

Mercury analysis with run log _____

Cyanide analysis _____

ICAP prep sheets _____

Furnace prep sheets _____

Mercury prep sheets _____

Cyanide prep sheets _____

Percent solids _____

Chain of custodies have been signed _____

All final report forms are present with no entries missing _____

Form I's are checked for calculation errors and all manual calculations match form results _____

Form IIA calibration verification - all data is checked to verify that ICV's and CCV's fall within limits _____

FIGURE 21.0

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

Form III calibration blanks - all data is less than the CRDL _____

Forms VA, VI, IX are checked for flags and are reported on Form I _____

Form VB post digest spikes are reported, if any _____

Form VII LCS all LCSW and LCSS values are within control for all analytes _____

Form VIII MSA all MSA's are reported, if any _____

Complete data package given to supervisor for final review _____

Case narrative is written _____

Completed case is given to CLP project officer for reporting _____

301309

FIGURE 21.0 (continued)

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

ATTACHMENT 1

COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

SOW No.: _____

EPA Sample No.	Lab Sample ID.

Were ICP interelement corrections applied? Yes/No ___

Were ICP background corrections applied? Yes/No ___

If yes-were raw data generated before application of background corrections? Yes/No ___

Comments:

_____

_____

_____

_____

Release of the data contained in this hardcopy data package and in the computer-readable data submitted on floppy diskette has been authorized by the Laboratory Manager or the Manager's designee, as verified by the following signature.

Lab Manager: _____

Date: _____

U.S. EPA - CLP

1

INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

a. me: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Lab Sample ID: _____

Level (low/med): _____ Date Received: _____

Solids: _____

Concentration Units (ug/L or mg/kg dry weight): _____

CAS No.	Analyte	Concentration	C	M	Q
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
	Cyanide				

Color Before: _____ Clarity Before: _____ Texture: _____

Color After: _____ Clarity After: _____ Artifacts: _____

Comments:

_____

_____

_____

_____

2A
INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Initial Calibration Source: _____

Continuing Calibration Source: _____

Concentration Units: ug/L

Analyte	Initial Calibration True	Initial Calibration Found	Initial Calibration %R(1)	Continuing Calibration True	Continuing Calibration Found	Continuing Calibration %R(1)	Continuing Calibration Found	Continuing Calibration %R(1)	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
romium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

2B
CRDL STANDARD FOR AA AND ICP

ıb Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ı CRDL Standard Source: _____

ICP CRDL Standard Source: _____

Concentration Units: ug/L

	CRDL Standard for AA			CRDL Standard for ICP				
					Initial		Final	
Analyte	True	Found	%R	True	Found	%R	Found	%R
Aluminum								
ntimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
C[illegible]ium								
[illegible]omium								
oalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

3
BLANKS

ab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

eparation Blank Matrix (soil/water): _____

reparation Blank Concentration Units (ug/L or mg/kg): _____

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L) 1	C	2	C	3	C	Prep-aration Blank	C	M
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
lcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											

U.S. EPA - CLP

4

ICP INTERFERENCE CHECK SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ ICS Source: _____

Concentration Units: ug/L

Analyte	True Sol. A	True Sol. AB	Initial Found Sol. A	Initial Found Sol. AB	Initial Found %R	Final Found Sol. A	Final Found Sol. AB	Final Found %R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
lcium								
.romium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

301316

U.S. EPA - CLP

5A
SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

L Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

_____

_____

_____

_____

U.S. EPA - CLP

5B

POST DIGEST SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

_____

_____

_____

_____

6
DUPLICATES

EPA SAMPLE NO.

Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Matrix (soil/water): _____ Level (low/med): _____

Solids for Sample: _____ % Solids for Duplicate: _____

Concentration Units (ug/L or mg/kg dry weight): _____

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

LABORATORY CONTROL SAMPLE

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Solid LCS Source: _____

Aqueous LCS Source: _____

Analyte	Aqueous (ug/L) True	Found	%R	Solid (mg/kg) True	Found	C	Limits		%R
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

8

STANDARD ADDITION RESULTS

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

Concentration Units: ug/L

EPA Sample No.	An	Dil	0 ADD ABS	1 ADD CON	1 ADD ABS	2 ADD CON	2 ADD ABS	3 ADD CON	3 ADD ABS	Final Conc.	r	Q

9

ICP SERIAL DILUTIONS

EPA SAMPLE NO

me: _____ Contract: _____

o Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

atrix (soil/water): _____ Level (low/med): _____

Concentration Units: ug/L

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Difference	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

10
HOLDING TIMES

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

EPA Sample No.	Matrix	Date Received	Mercury Prep Date	Mercury Holding Time	Cyanide Prep Date	Cyanide Holding Time

Form XI

INSTRUMENT DETECTION LIMITS

LAB NAME _____ DATE _____

ICP/Flame AA (Circle One) Model Number _____ Furnace AA Number _____

Element	Wavelength (nm)	IDL (ug/L)	Element	Wavelength (nm)	IDL (ug/L)
1. Aluminum			13. Magnesium		
2. Antimony			14. Manganese		
3. Arsenic			15. Mercury		
4. Barium			16. Nickel		
5. Beryllium			17. Potassium		
6. Cadmium			18. Selenium		
7. Calcium			19. Silver		
8. Chromium			20. Sodium		
9. Cobalt			21. Thallium		
10. Copper			22. Vanadium		
11. Iron			23. Zinc		
12. Lead					

Footnotes:
- Indicate the instrument for which the IDL applies with a "P" (for ICP an "A" (for Flame AA), or an "F" (for Furnace AA) behind the IDL valu
- Indicate elements commonly run with background correction (AA) with a "B" behind the analytical wavelength.
- If more than one ICP/Flame or Furnace AA is used, submit separate Forms XI-XIII for each instrument.

COMMENTS: _____

Lab Manager _____

12A

ICP INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Wavelength (nm)	Interelement Correction Factors for: Al	Ca	Fe	Mg	
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

_____

_____

_____

_____

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12B

ICP INTERELEMENT CORRECTION FACTORS (QUARTERLY)

ab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

CP ID Number: _____ Date: _____

Analyte	Wave-length (nm)	Interelement Correction Factors for:				
		___	___	___	___	___
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

_____

_____

_____

_____

U.S. - CLP

ICP LINEAR RANGES (QUARTERLY)

Lab Name: _____ Contract: _____

Lab Code: _____ Case No.: _____ SAS No.: _____ SDG No.: _____

ICP ID Number: _____ Date: _____

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Mercury			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			

Comments:

_____

_____

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STANDARD OPERATING PROCEDURE
TRACKING METALS AND CYANIDE SAMPLE ANALYSIS

Abstract

This Standard Operating Procedure addresses the tracking of samples for metals and cyanide analysis from sample receipt through analysis and final reporting. It is meant to provide an analytical scheme of those forms required to adequately trace a sample's path through the laboratory.

STANDARD OPERATING PROCEDURE

MEASUREMENT OF CONDUCTIVITY

IN WATER SAMPLES

WC:082190:0

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1.0 SCOPE AND APPLICATION

1.1 This method is applicable to the measurement of conductivity in drinking, surface, and saline waters, and industrial wastes.

2.0 SUMMARY OF METHOD

2.1 The conductivity or specific conductance of a sample is measured by use of a conductivity meter (Wheatstone bridge type).

2.2 Samples are to be analyzed at 25 C. A temperature conversion can be made in order that results may be reported at 25 C when temperature control is not possible.

3.0 INTERFERENCES

3.1 Temperature fluctuations represent the largest source of errors.

4.0 APPARATUS AND MATERIALS

4.1 Conductivity Meter

4.2 Conductivity cell, cell constant 1.0

4.3 Beakers, 50 mL

4.4 Thermometer

5.0 REAGENTS

5.1 Reagent Water (ASTM Type II)

5.2 Standard potassium chloride, 0.01 M: Dissolve 0.7456 grams of pre-dried (2 hrs at 105 C) KCl in reagent water and dilute to 1 liter at 25 C.

5.3 Working standards are prepared by making a 10 and 100 fold dilution of the potassium chloride stock (5.2).

6.0 **SAMPLE PRESERVATION AND STORAGE**

6.1 Samples must be unpreserved and analyzed as soon as possible.

7.0 **PROCEDURE**

7.1 All samples to be analyzed must be signed out of sample control on the chain-of-custody for each job.

7.2 Measurement of Conductivity

7.5.1 Calibrate the conductivity meter with two standards that bracket the expected conductivity of the samples. Follow the manufacturer's instructions to calibrate.

7.5.2 Allow the samples to come to room temp. (23 to 27 C) Determine the temperature of the sample and correct to 25 C if necessary (mathematically, section 10.0).

7.5.3 Place the sample or standard solution in a clean glass beaker using a sufficient volume to cover the sensing element of the conductivity cell. Stir gently at a constant rate so as to insure homogeneity. Record the conductivity.

7.5.4 Thoroughly rinse the cell with reagent water and wipe between measurements.

7.5.5 After the completion of all conductivity measurements, store the cell in reagent water.

8.0 **QUALITY CONTROL**

8.1 All chemicals should conform to minimum criteria set by the Reagent Chemical Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

8.2 All stock preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, date and initials of the chemist preparing the stock.

8.3 A reagent water blank is run with every batch of samples analyzed, however, the nature of the test is such that there is no blank criteria.

8.4 After the initial calibration of the meter at least one known standard will be measured for conductivity and recorded.

8.5 A sample duplicate will be analyzed for every batch of twenty or fewer samples. The maximum acceptable criteria for conductivity is a relative percent difference (RPD) of 20.0 .

9.0 METHOD PERFORMANCE

9.1 No data submitted.

10.0 DATA HANDLING AND REDUCTION

10.1 All raw data is recorded in the analyst's notebook. The conductivity results are entered into LIMS for sample tracking and reporting purposes.

10.2 If the sample temperature is below 25 C, add 2% of the reading per degree. If the temperature is above 25 C, subtract 2% of the reading per degree. Report results as Specific Conductance, umho/cm at 25 C.

10.3 The sample QA/QC is reported on the inorganic QA/QC summary form (see appendix I).

10.4 Technical Data Review: includes verifying that batch QC is within criteria and the raw data entered into LIMS is checked for transcription errors.

11.0 DOCUMENTATION OF PROBLEMS

11.1 Corrective Action Reports: must be filed if any problems are associated with the analysis (see appendix I).

11.2 Case Narratives: should include any deviation from this SOP or any difficulties encountered in the procedure.

12.0 REFERENCES

12.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, 1983.

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APPENDIX I

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QA: 111086
Rev 3

Reported By: _____
Group
Date: _____ Time: _____

CORRECTIVE ACTION REPORT / CASE NARRATIVE REPOF

Person Who Reported Problem: _____

Person Who Is Requested To Provide Corrective Actions: _____

Deadline To Complete Corrective Actions: _____

Category of Problem: _____
Poor Communication With ___. Sample Matrix; Instrument

Client: _____ Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____
Document Corrective Actions On This

_____
Form and Place a Copy of Corrective Actions In Job Folder

_____

_____

_____

_____

Is this a recurring Problem? _____ Should SOP be Modified or Updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Distribution: (circle) Lab Director, Lab manager, Marketing Manager, Branch Manager
Group/Section Leader, QA Officer: Organic[ ], Inorganic[ ]
Client Services, Job Folder, Other: _____

Note Problem and Corrective Action(s) In Case Narrative: [ ]

Attachments: Telephone Record Log, Services Change Request, Memo, ____

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IEA
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CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte:_____ Date:_____ Ref:_____

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

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STANDARD OPERATING PROCEDURE

ANALYSIS OF TOTAL ALKALINITY

IN WATER SAMPLES

WC:040491:0

301338

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

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Total Alkalinity

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.2 The method has a practical quantitation limit (PQL) of 1.0 mg/L (as $CaCO_3$) and is suitable for all higher concentration ranges of alkalinity. However, aliquots should be used to avoid a titration volume of greater than 50mL.

1.3 Automated titrametric analysis is equivalent.

2.0 SUMMARY OF METHOD

2.1 A sample is titrated with a standardized acid to an electrometrically determined end point of pH 4.5. The sample must not be filtered, or altered in any way.

3.0 INTERFERENCES

3.1 Organic material, soaps, oily matter, suspended soils, or precipitates may coat the glass electrode and cause a sluggish response.

3.2 Allow sufficient time between titrant additions to let the electrode response to come to equilibruim.

4.0 APPARATUS AND MATERIALS

4.1 A pH meter or electrically operated titrator that uses a glass electrode and can be read to 0.05 pH units. Standardize and calibrate according to manufacturer's instructions. If automatic temperature compensation is not provided, perform total alkalinity analysis at 25 $\pm$ 2°c.

4.2 Titration rack and electrode support

4.3 Magnetic stirrer plate and bar magnets.

4.4 Pipets (Class A); 3, 5, 10, and 20mL.

4.5 Erlenmeyer flasks (wide mouth); 100 and 250mL.

4.6 Volumetric flasks; 200 and 1000mL.

4.7 Burets (Class A); 50, 25, and 10mL.

4.8 Graduated Cylinders; 25 and 100mL.

5.0 REAGENTS

NOTE: See 10.2 for acid normality calculation.

5.1 Sodium carbonate solution (approx. 0.05N):
Place 2.5 ± 0.2 grams (to the nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1 liter volumetric flask and brought to volume.

5.2 Standard sulfuric acid (0.1 N):
Dilute 3.0 mL of concentrated H_2SO_4 to 1 liter with reagent grade water. Standardize versus 40.0 mL of 0.05 N Na_2CO_3 solution with 60 mL of reagent grade water by titrating potentiometrically to a pH of approx. 5. Lift the electrode and rinse with reagent grade water into the beaker. Boil the solution gently for 3 to 5 minutes under a watch glass. Cool to room temperature. Rinse the watch glass into the beaker and continue the titration to the inflection point.

5.3 Standard sulfuric acid (0.02 N):
Dilute 200.0 mL of 0.1000 N H_2SO_4 to 1 liter with regent grade water. Standardize by the potentiometric titration of 15.0 mL 0.05 N Na_2CO_3 solution as in 5.2.

CAUTION: Sulfuric acid solutions are corrosive and cause burns. Avoid all contact. Wear gloves and appropriate eye protection at all times!

6.0 SAMPLE STORAGE AND HANDLING

6.1 Samples are to be stored unpreserved at 4°C.

6.2 Samples are to be analyzed within 14 days of sampling or within 12 days of receipt (NYSDEC protocol).

7.0 PROCEDURE

7.1 All samples to be analyzed must be signed out of sample control on the chain-of-custody (COC) for each job. All alkalinity data collected is recorded in the alkalinity log (see appendix I).

7.2 Place 100 mL or an appropriate smaller sample volume into a flask, measure the pH of the sample. Record the sample volume used.

NOTE: Use a sufficiently large sample size to require a volume of titrant (>10 mL in 50 ml buret) which will obtain good precision while keeping the volume low enough to permit a sharp end point.

7.3 Add 0.0200 N H_2SO_4, being careful to stir thoroughly but gently to allow for equilibrium. Titrate to pH 4.5 and record the volume of titrant and exact normality.

NOTE: For samples containing >1000 mg/L as $CaCO_3$ of alkalinity, use 0.1000 N H_2SO_4 as the titrant.

8.0 QUALITY CONTROL

8.1 All stock solutions and standard preparations are logged and coded. All solutions are labeled with the following: analyte, concentration, analyst's initials, date prepared, and expiration date.

8.2 All chemicals will conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first-in, first-out basis.

8.3 A prep blank (reagent water) is to be analyzed with every batch of ten or fewer samples.

8.4 One laboratory control sample (LCS) is to be analyzed with each batch of ten or fewer samples. LCS control limits are ± 15% of the known concentration.

NOTE: Should the LCS fail, the source of the problem must be corrected and an acceptable LCS run before continuing with the analysis of samples.

8.5 One sample duplicate must be analyzed from each batch of ten or fewer samples. Samples identified as field blanks are not to be used for duplicate analysis. The relative percent difference (RPD) between the sample and duplicate must be less than or equal to 20.0 (see section 10.4 for the RPD calculation). No RPD criteria is applied if both results are less than five times the PQL. No RPD criteria is applied if one result is lower than five times the PQL and one above, if the difference between the two results is no more than the PQL.

9.0 METHOD PERFORMANCE

9.1 The average relative percent difference over the last 25 positive duplicates was 2.36% as of 3/28/91.

9.2 The last ten LCS percent recoveries averaged 105.6% as of 3/28/91.

10.0 DATA HANDLING AND REDUCTION

10.1 All raw data including sample volume, titrant volume, and acid normality are entered into LIMS. The LIMS system calculates the results.

10.2 The normality of the H_2SO_4 titrant is calculated:

$$N = \frac{A \times B}{53. \times C}$$

where: A = grams Na_2CO_3 into 1 liter
B = mL Na_2CO_3 solution
C = mL acid used to inflection point

10.3 Manual alkalinity calculation (titration to pH 4.5).

$$\text{Alkalinity (mg/L as } CaCO_3) = \frac{A \times N \times 50{,}000}{\text{mL of sample}}$$

where: A = mL of acid titrated
N = normality of acid

10.4 The relative percent difference is calculated:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

where: S = Original sample result
D = Duplicate sample result

10.5 The sample quality control and LCS recovery results are reported on the QA/QC summary form (see appendix I).

10.6 The technical data review includes verifying that samples analyzed were unpreserved and that holding times were met. All quality control measures are checked to insure that all QC criteria were met. Results entered into LIMS are checked for transcription error against the raw data.

11.0 DOCUMENTATION OF PROBLEMS

11.1 Corrective Action Reports - must be filed if any problems are encountered during analysis.

11.2 Case Narratives - should include information collected from corrective action reports and any deviations from this SOP.

12.0 REFERENCES

12.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, Method 310.1, 1983.

12.2 STANDARD METHODS: For the Examination of Water and Wastewater, 15th ed., Method 403, 1980.

APPENDIX I

IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____

_____

_____

_____

_____

_____

_____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

301346

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CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte:_____ Date:_____ Ref:_____

LABORATORY CONTROL SAMPLE

Reference	True Value	Value Found	% Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

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Alkalinity

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QC #	Sample ID	Initial pH	Sample Volume	Buret Measurement		mls Titrated	Alkalinity mg/L as CaCO3
				Initial	Final		

Normality of
H2SO4 _____

Analyst _____

Date _____

Page _____

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STANDARD OPERATING PROCEDURE

ANALYSIS OF TOTAL DISSOLVED SOLIDS

IN WATER

WC:081290:0

301349

TABLE OF CONTENTS

1.0 SCOPE AND APPLICATION

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

1.2 The practical range of the determination is 1.0 mg/L to 20,000 mg/L.

2.0 SUMMARY OF METHOD

2.1 A well-mixed sample is filtered through a glass fiber filter, and the filtrate is evaporated to dryness in a weighed beaker and dried to a constant weight at 180 C. The increase in beaker weight represents the total dissolved solids (TDS).

2.2 If total suspended solids is being determined, the filtrate may be utilized in the TDS determination.

3.0 INTERFERENCES

3.1 Highly mineralized waters containing significant concentrations of calcium, magnesium, chloride, and sulfate may be hygroscopic and will require prolonged drying, desiccation and rapid weighing.

3.2 Samples containing high concentrations of bicarbonate will require prolonged drying at 180 C to insure that all of the bicarbonate is converted to carbonate.

3.3 Too much residue in the beaker will crust over and entrap water that will not be driven off during drying. Total residue should be limited to 0.25 g.

4.0 APPARATUS AND MATERIALS

4.1 Glass Fiber Filters, 2.1 cm
4.2 Gooch Crucible Adapter
4.3 Side Arm Filtration Flask, 1 L
4.4 Beakers, 150mL or 250mL
4.5 Drying Oven, 180 C + 2 C
4.6 Desiccator
4.7 Gooch Crucibles
4.8 Graduated Cylinder, 100mL
4.9 Analytical Balance

5.0 **REAGENTS**

5.1 ASTM Type II Reagent Water (Nano)

6.0 **SAMPLE PRESERVATION, AND STORAGE**

6.1 Samples are to be unpreserved and stored at 4 C. The holding time for TDS is 48 hours from the time of sampling, however, the NYSDEC 89 protocol holding time is 24 hours from the time of receipt in the lab.

7.0 **PROCEDURE**

7.1 Gooch Preparation: Insert a glass fiber filter disc into the bottom of a gooch crucible. While vacuum is applied, wash the disc with three successive 20 mL volumes of nano water.

7.2 Beaker Preparation: Heat clean beakers to 180 + 2 C for one hour. Cool in desiccator and store until needed. Weigh immediately before use.

7.3 All samples to be analyzed must be signed out of sample control on the chain-of-custody for each job.

7.4 Assemble the filtering apparatus and begin suction. Shake the sample vigorously and rapidly transfer 100 mL to the gooch, and filter.

NOTE: The use of Millipore setup and glass fiber filter is an acceptable alternative to using a gooch.

7.5 After the sample has passed through the the glass fiber filter, rinse with three 10 mL portions of nano water and continue to apply vacuum for three minutes to insure that as much water has been removed as possible.

7.6 Transfer the filtrate to a weighed beaker and evaporate to dryness in a drying oven set at 100 - 105 C.

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7.7 Dry the evaporated sample for at least four hours at 180 C. Cool in a desiccator and record the weight after a constant weight has been obtained.

NOTE: Choose a sample size to yield no mre than 250 mg of dried residue. If more than five min. are required to complete filtration decrease the sample volume.

8.0 **QUALITY CONTROL**

8.1 A reagent blank will be analyzed with every batch of twenty or fewer samples. If the blank has a positive total dissolved solids value the sample results are acceptable if the blank is no more than 10% of the sample result.

8.2 Duplicates

One duplicate analysis should be analyzed with every batch of twenty or fewer samples. Samples identified as field blanks should not be used for duplicate analysis. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{(S - D)}{(S + D)/2} \times 100$$

A control limit of 20 percent for RPD shall be used for sample and duplicate values equal to or greater than five times the lower detection limit.

8.3 Laboratory Control Samples

One independent standard (LCS) is to be analyzed with each batch of twenty or fewer samples if available. The control limits for the LCS is $\pm$ 15 percent of the true value.

8.4 A class S weight of approx. the same mass of the beakers used should weighed when the beakers are tared and weighed back.

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9.0 **METHOD PERFORMANCE**

9.1 The average RPD over the last 25 duplicates was 5.8% as of 08/03/90.

10.0 **DATA HANDLING AND REDUCTION**

10.1 All raw data including weights and sample volumes are entered into LIMS. The LIMS system will calculate the result automatically.

10.2 Manual TDS Calculation:

$$\text{Filterable Residue (mg/L)} = \frac{(A - B) \times 1000}{C}$$

where:

A = Weight of dried residue and beaker in mg

B = Weight of beaker in mg

C = Sample Volume in mL

10.3 The sample QA/QC and LCS are reported on the inorganics QA/QC summary form (see appendix I).

10.4 Technical Data Review: includes verifying that sample holding times were met, that batch QC is within criteria and the raw data entered into LIMS is checked for transcription errors.

11.0 **DOCUMENTATION OF PROBLEMS**

11.1 Corrective Action Reports: must be filed if any problems are associated with the analysis (see appendix I).

11.2 Case Narratives: should include any deviation from this SOP or any difficulties encountered in the procedure.

12.0 REFERENCES

12.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, 1983, Method 160.1.

12.2 STANDARD METHODS For the Examination of Water and Wastewater, 15th edition, 1980.

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APPENDIX I

301356

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CLASSICAL CHEMISTRY QA/QC SUMMARY

ANALYTE: _____ DATE: _____ REF.: _____

LABORATORY CONTROL SAMPLE

REFERENCE	TRUE VALUE	VALUE FOUND	% RECOVERY

DUPLICATES

LAB SAMPLE ID	RESULT 1	RESULT 2	MEAN	RPD

PREP BLANK

BLANK ID	RESULT

SAMPLES IN BATCH:	COMMENTS:

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IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____

_____

_____

_____

_____

_____

_____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

301358

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Standard Operating Procedure
Analysis of Ammonia-Nitrogen

WC:021690:0

301359

1.0 SCOPE AND APPLICATION

1.1 The distillation method covers the determination of ammonia-nitrogen in drinking, surface and saline waters, domestic and industrial wastes.

1.2 The latest version of the automated phenate method should be utilized (after the preliminary distillation step) when sample load warrants.

1.3 This method covers the range from 0.05 to 4.0 mg NH_3-N/L for the colorimetric procedure, and from 4.0 mg/l to 25 mg/l for the titrametric procedure.

2.0 SUMMARY OF METHOD

2.1 The sample is buffered at a pH of 9.5 with a borate buffer in order to decrease hydrolysis of cyanates and organic nitrogen compounds, and is then distilled into a solution of boric acid. The ammonia in the distillate can be determined colorimetrically by nesslerization, or titrametrically with standard sulfuric acid with the use of a mixed indicator.

3.0 INTERFERENCES

3.1 Aromatic and aliphatic amines, as well as other compounds, both organic and inorganic, will cause turbidity upon the addition of Nessler reagent, these interferences can be removed via distillation of the sample.

3.2 Cyanates will hydrolyze to some extent even at the pH of 9.5 at which the distillation is carried out. Volatile alkaline compounds, such as certain ketones, aldehydes, and alcohols, may cause an off-color upon nesserlization in the distillation method.

3.3 Residual chlorine must be removed by pretreatment of the sample with sodium thiosulfate before distillation.

4.0 APPARATUS AND MATERIALS

4.1 Heating mantles with temperature controllers

4.2 Florence Flasks, 500 mL

4.3 Erlenmeyer Flasks, 250 mL

4.4 Volumetrics, 250 mL

4.5 Graduated Cylinders, 50 mL

4.6 Pipets

4.7 Nessler Tubes

4.8 Volumetrics, 1 Liter

5.0 REAGENTS

5.1 Ammonium chloride, stock solution: 1.0 mL = 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in Nano water and bring to volume in a 1 liter volumetric flask.

5.2 Ammonium chloride, standard solution: 1.0 mL = 0.01 mg NH_3-N. Dilute 10 mL of stock solution (5.1) to 1 liter.

5.3 Boric acid solution: Dissolve 20 grams of boric acid in Nano water and dilute to 1 liter.

5.4 Mixed indicator: Mix 20 mL of 0.2% methyl red in 95% ethanol with 10 mL of 0.2% methylene blue is 95% ethanol. This solution should be prepared fresh every 30 days.

5.5 Nessler reagent: Dissolve 100 g of mercuric iodide and 70 g of potassium iodide in a small amount of Nano water. Add this mixture slowly, with stirring, to a cooled solution of 160 g of NaOH in 500 mL of water. Dilute to 1 liter. If stored in an amber bottle the solution will be stable for a period of up to 1 year.

5.6 Borate buffer: Add 88 mL of 0.1N NaOH solution to 500 mL of 0.025M sodium tetraborate solution (50 g anhydrous $Na_2B_4O_7$ on 95 g $Na_2B_4O_7$ per liter) and dilute to 1 liter.

5.7 Sulfuric acid, standard solution: (0.02N, 1 mL = 0.28 mg NH3-N). Dilute 200 mL 0.1N H_2SO_4 (standardized against 0.100N Na_2CO_3 solution) to 1 liter with Nano water.

5.8 Sodium carbonate, standard solution: Dissolve 1.060 g anhydrous Na_2CO_3, oven dried at 140°C to 1 liter in CO_2 free distilled water.

6.0 SAMPLE HANDLING AND PRESERVATION

6.1 Water and soils are to be distilled within 28 days of sampling.

6.2 Water samples should be preserved to a pH <2 with sulfuric acid and stored at 4 C.

7.0 PROCEDURE

7.1 All samples to be analyzed must be signed out of sample control on the chain-of-custody for each job.

7.2 Distillation

7.2.1 Each distillation apparatus is to be steamed out between samples. To steam out; add approximately 250 mL of nano water, a drop of phenolphthalein, and sufficient 6N NaOH to turn the solution pink to each florence flask. Add boiling stones to the flask, and turn on the condenser water. Turn on the power to the heating mantle. Continue to distill until no trace of ammonia is observed (no reaction upon the addition of nessler reagent).

NOTE: To minimize contamination, leave the distillation apparatus assembled until ready to distill samples.

7.2.1 Add 250 mL of aqueous or 250 mL nano water and 5 grams of soil to a florence flask. Add a drop of phenolphthalein and sufficient 6N NaOH dropwise buffer to the flask.

NOTE: Percent solids must be determined for soil sample analysis.

7.2.3 Add 50 mL of boric acid solution to a 250 mL erlenmeyer receiving flask. Raise the receiving flask so that the tip of the condenser is below the surface of the boric acid, but not touching the bottom of the flask.

7.2.4 Turn on the heating mantles and condenser water, distill over a volume of 200 mL.

NOTE: When the volume of distillate has been received turn off the heat and lower the receiving flask.

7.2.5 Transfer the distillate into 250 mL volumetrics.

NOTE: If the sample requires TKN or TON analysis, save the sample in the florence flask.

7.3 Development of Nesslerization

7.3.1 Place 50 mL or an aliquot diluted to 50 mL with nano water in a clean nessler tube.

7.3.2 Prepare a 10 ug and a 200 ug standard from the standard ammonia solution.

7.3.3 Add 2 mL of nessler reagent to each nessler tube, allow 10 minutes for color development and read at 425 nm.

7.3.4 See Appendix II for spectrophotometer instructions.

NOTE: If an aliquot of 0.5 mL of distillate has an absorbance higher than the high standard the distillate will have to be titrated (7.4).

7.4 Analysis by Titration

7.4.1 To 50 mL of distillate, add 3.5 drops of mixed indicator and titrate with 0.02 N H_2SO_4 until the solution turns a pale lavender. A 50 mL nano water blank must be titrated for calculation purposes.

8.0 QUALITY CONTROL

8.1 For the nesslerization method the 10 ug and 200 ug must agree with the established curve ±15%, if the control limits are not met a new curve should be prepared.

8.2 A new curve requires the preparation and nessler development of a preparation blank, 0.05, 0.20, 0.60, 1.00, 2.00, and 4.00 mg/L standards.

8.3 For the titration analysis method the 0.02 N H_2SO_4 should be standardized against a 0.0200 Na_2CO_3 solution.

8.4 A preparation blank is distilled each day that samples are distilled.

8.5 A sample duplicate is distilled per batch (20 samples max.).

$$RPD = \frac{(S-D)}{(S+D)/2} \times 100\%$$

where S = sample result
D = duplicate result

The RPD value must be ≤20 percent.

8.6 A sample spike is distilled per batch (20 sample maximum).

$$\% \text{ Recovery} = \frac{(SSR-SR)}{SA} \times 100\%$$

The acceptable spike recovery limits are 75-125 percent.

8.7 Batch QA/QC is reported on Inorganic QA/QC summary forms (See Appendix I).

9.0 METHOD PERFORMANCE

9.1 The detection limit for waters is established by the low standard on the curve (0.05 mg/L). Soil detection limits must be calculated taking into account sample mass and percent solids.

9.2 Precision and Accuracy

Twenty-four analysts in sixteen laboratories analyzed natural water samples containing exact increments of an ammonium salt, with the following results:

Increment as Nitrogen, Ammonia mg N/liter	Precision as Standard Deviation mg N/liter	Accuracy as Bias, %	Accuracy as Bias, mg N/liter
0.21	0.122	-5.54	-0.01
0.26	0.070	-18.12	-0.05
1.71	0.244	+0.46	+0.01
1.92	0.279	-2.01	-0.04

10.0 DATA REDUCTION AND DATA HANDLING

10.1 All distillation data including sample ID, sample volume, and volume distilled are recorded in each analysts notebook. Development data is recorded with the distillation information. If titration is required, the data is recorded in the analyst's notebook performing the titration.

10.2 Raw data and results are entered onto LIMS, the final concentrations are calculated by the system (for the colorimetric method).

10.3 Titration Calculation

$$\text{mg } NH_3\text{-N/L} = \frac{(A-B) \times 280}{\text{mL Sample}}$$

INORGANICS QA/QC SUMMARY

Analyte:_____ Date:_____

Notebook Ref:_____

SPIKES

Lab Sample ID	Spike Result	Sample Result	Amount Added	Percent Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

BLANKS

Blank ID	Result

Samples in Batch:	Comments:

301365

IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____

_____

_____

_____

_____

_____

_____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

301366

APPENDIX II

INSTRUCTIONS FOR THE MILTON ROY SPECTROPHOTOMETER

1. Turn the power on, and allow the instrument to warm up for 30 minutes.
2. Enter the desired wavelength and press "Go to λ"
3. Set the test mode to ABS.
4. Insert the sample cell with calibration blank into the spectrophotometer, close the sample door and press "Auto Zero".
5. Rinse the cuvette with sample twice and dry sides with kimwipes. Fill the cell with sample, place the cell back into the spec, close the sample door and record the absorbance.

INSTRUCTIONS FOR THE PERKIN-ELMER 0B SPECTROPHOTOMETER

1. Preparation

Power W damp - on (not D_2 damp)
Pull out Vis lamp knob
Slit - set at 2.0 nm
Response - Med
Suppression - Off
Scan - Off
NM - ignore (used with scan)
Wavelength - put at 500 - reads in absorbance or % T
Push % T (= % Transmittance)
w/o cells - adjust digital readout to 100% T with the knob on top (always reads ± when it's +)

Block front (sample) cell path with board - should read 0% T if not, adjust to 0% T with 0% T knob.
Remove board.
Back Cell = Blank
Front Cell = Sample - Lettering on cells should always point to the right when in the spec.

2. Operation

Abs - depress 0-2 scale
Clean cells; rinse out 1 time with blank and dry sides with Kimwipes
Put blank cells in front and back
Adjust wavelength to that required for the analysis.
With the blanks in, adjust 0 abs to read 0.

Now take the front cell out; rinse twice with the sample, fill with sample, put in the spec and take down the absorbance reading.
When done - just take out cells, clean them and leave on top to dry.

3. Shut Down

Switch off the W lamp and main power.

an environmental testing company
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Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

FOR CHLORIDE

WC:031189:0

301370

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

ABSTRACT

Chloride is one of the major inorganic anions in potable and wastewater supplies. When sodium is present in drinking water, chloride concentrations in excess of 250 mg/L give a salty taste. Chloride is essential in the diet and passes through the digestive system unchanged to become one of the major components of raw sewage. The use of zeolite in water softeners also contributes a large amount of chloride to sewage and wastewaters. Chlorides may be present in high concentrations along coastal areas, due to the seepage of seawater into the sewage system.

High chloride concentrations in water are not known to have toxic effects on man, however, large amounts may act corrosively on metal pipes and be harmful to plant life. The maximum contaminant level for chloride in drinking water has been established at 250 mg/L for reasons of taste rather than as a safeguard against a physical hazard.

1.0 SCOPE AND APPLICATION

There are four methods available for the determination of chloride. The Argentometric method is suitable for use in relatively clear waters when 0.15 to 10 mg Cl^- are present in the portion titrated. The same can be said for the mercuric nitrate method, except that its endpoint is easier to detect. The potentiometric method is suitable for colored or turbid samples in which color-indicated endpoints might be difficult to observe. The ferricyanide method is an automated technique.

2.0 SUMMARY OF ARGENTOMETRIC METHOD

In a neutral or slightly alkaline solution, potassium chromate can indicate the endpoint of the silver nitrate titration of chloride. Silver chloride is precipitated quantitatively before red silver chromate is formed.

3.0 INTERFERENCES

Substances in amounts normally found in potable waters will not interfere. Bromide, iodide and cyanide register as equivalent chloride concentrations. Sulfide, thiosulfate and sulfite ions interfere but can be removed by treatment with hydrogen peroxide. Orthophosphate in excess of 25 mg/L interferes by precipitating as silver phosphate. More than 10 mg/L of iron will mask the end-point.

4.0 APPARATUS

50 ml buret
250 ml erlenmeyer flasks

5.0 REAGENTS

5.1 - Potassium chromate indicator solution: Dissolve 50 g K_2CRO_4 in approximately 50 ml nano water. Add $AgNO_3$ solution until a definite red precipitate is formed. Let stand 12 hours, filter, and dilute to 1L with nanopure water.

where A = mLs of titrant in the sample
B = mLs of titrant in the sample

11.0 USE OF OTHER SOP'S

11.1 The latest version of the automated phenate method may be utilized for developing distillates if sample quantities warrant.

12.0 DOCUMENTATION OF PROBLEMS

12.1 Corrective Action Reports - must be filed if any problems were associated with the analysis (See Appendix I).

12.2 Case Narratives - should include any deviation from this SOP or any difficulties encountered in the procedure.

13.0 REFERENCES

13.1 EPA-600/4-79-020, Methods for Chemical Analysis of Water and Wastes, Method 350.2.

APPENDIX I

5.2 - Standard silver nitrate titrate, 0.141 N: Dissolve 2.395 g $AgNO_3$ in nanopure water and dilute to 1L. Standardize against 0.0141 N NaCl by the procedure described below. 1.00 ml = 500 ug Cl^-. Store in a brown bottle.

5.3 - Standard sodium chloride, 0.014 N: Dissolve 824.0 mg NaCl (dried at 140°C) in nanopure water and dilute to 1L. 1.00 ml = 500 ug Cl^-.

5.4 Phenolphthalein indicator solution
5.5 Sodium hydroxide, NaOH, 1N
5.6 Sulfuric acid, H_2SO_4, 1N

Note: When making a new reagent or standard record it in the prep log book!

Note: All reagents should be of A.C.S. reagent grade quality.

.0 PROCEDURE

6.1 Sample Preparation - Acid wash erlenmeyer flasks and buret with 1:1 HNO_3 and rinse three times with nano water. Use 50 ml of an unpreserved sample or a suitable portion diluted to 50 ml with nanopure water. For a soil matrix, weigh out 10.00 g of the sample and place in a beaker. Add two drops of concentrated HNO_3 and 100 ml nanowater. Stir and heat on hot plate until warm to touch. Filter through 45 um filter and use 50 ml filtrate.

Adjust pH to neutralize sample by adding a drop of phenolphthalein indicator solution. Then drop wise add 1N NaOH until a pink color appears. Add 1N H_2SO_4 just until the sample turns colorless.

6.2 Titration - Add 1.0 ml of K_2CrO_4 indicator solution. Titrate with standard $AgNO_3$ titrant to a pinkish yellow endpoint. Be consistent in end-point recognition.

Standardize $AgNO_3$ titrant by adding 5 ml of standard NaCl to 45 ml of nano water. Titrate as described above. Establish a reagent blank of 0.3 ml is normal. Titrate two NaCl standards and two blanks to average and use for calculations.

6.3 Dilutions: Normally, when the first few drops of $AgNO_3$ titrant go into the sample, a reddish color will appear from the red silver chromate. If this color does not appear, a dilution is in order. Ideally, 1.0 - 10.0 ml of titrant should be used on each sample.

7.0 QUALITY CONTROL

For every twenty chloride samples titrated, a sample duplicate and matrix spike should be performed. To spike, pipet 5 ml of standard NaCl to the 50 ml of sample try not to spike a dilution) and titrate.

Calculation:

To find the normality of the standard $AgNo_3$:

$$N_{NaCl} \times \underset{(0.0141)}{\text{mls NaCl used}} = N_{AgNO3} \times \underset{5\ \text{mls}}{\text{mls } AgNO_3 \text{ used}} - \text{avg. blank}$$

$$mg\ Cl^-/L = \frac{(A-B) \times N \times 35{,}450}{\text{ml sample}}$$

where A = ml titrant sample
B = ml titrant blank
N = Normality $AgNO_3$

Spikes

Spikes are used to calculate the recovery of an analyte:

$$\% \text{ Recovery} = \frac{(SSR - SR)}{SA} \times 100$$

Where: SSR = spiked sample result (mg/L)
SR = sample result (mg/L)
SA = spike added (mg/L)

The acceptable range is 75 - 125%

Duplicates

Relative Percent Difference (RPD) is used to determine the precision of results.

$$RPD = \frac{[S - D]}{(S + D)/2} \times 100$$

Where S = sample result (mg/Kg)
D = duplicate result (mg/L)

The RPD value must be $\leq$ 20%

Significant Figures: <10;2 >10;3
LDL (Lower Detectable Limit) 1.0 mg/L

Reporting of Results: .

Results are computed for samples on LIMS and may have to be modified to take into account LDL's and significant figures.

Documentation of Problems:

Corrective action reports must be filed if any problems were associated with the analysis.

Case Narratives should include any departure from normal procedure or any difficulties encountered in the analysis.

References:

Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985, APHA-AWWA-WPCF.

APPENDIX I:

BENCH SHEETS, REAGENT LOGS

AND CORRECTIVE ACTION FORM

STANDARD OPERATING PROCEDURE

ANALYSIS OF SULFATE

IN WATER

WC:030489:0

301380

200 MONROE TURNPIKE • MONROE, CONNECTICUT 06468 • (203) 452-8200

Abstract

Sulfate (SO_4^{2-}) occurs in natural waters in a wide range of concentrations. Mine drainage wastes and industrial effluents may contribute large amounts of sulfate from pyrite oxidation and the use of sulfuric acid. Secondary maximum contaminant levels for drinking water call for no more than 250 mg/L of sulfate because of its cathartic action. In domestic waters, sulfates do not cause any increased corrosion on brass fittings, but concentrations above 250 mg/L do increase the amount of lead dissolved from lead pipes or joints.

1.0 SCOPE AND APPLICATION

The gravimetric method with ignition of residue and the gravimetric method with drying of residue are suitable for sulfate concentrations above 10.0 mg/L; use only one of these methods for accurate results. The turbidimetric method is applicable in the range of 1.0 to 40.0 mg SO_4^{2-}/L.

2.0 SUMMARY OF GRAVIMETRIC METHOD WITH DRYING OF RESIDUE

Sulfate is precipitated in a hydrochloric acid (HCl) solution as barium sulfate ($BaSO_4$) by the addition of barium chloride ($BaCl_2$). The precipitation is carried out near the boiling temperature, and after a period of digestion the precipitate is filtered, washed with water until free of Cl^-, dried, and weighed as $BaSO_4$.

3.0 INTERFERENCES

The interferences leading to high results are suspended matter, silica, $BaCl_2$ precipitant, nitrate, and sulfite. Alkali metal sulfates frequently yield low results, as do heavy metals such as chromium and iron. The latter two interfere with the complete precipitation of SO_4^{2-} and by the formation of heavy metal sulfates, $BaSO_4$ has a small but significant solubility, which is increased in the presence of acid. Although an acid medium is necessary to prevent precipitation of barium carbonate and phosphate, it is important to limit its concentration to minimize the solution effect.

4.0 APPARATUS

a) Drying oven, equipped with thermostatic control
b) Desiccator
c) Analytical balance
d) Gooch crucibles
e) Filter pads 934 AH, unsterile, 45 filter membranes
f) Filtering apparatus
g) Beakers (100 or 150 mL)
h) Hot plate
i) Grad. cylinder 50 mL
j) 10 mL class A pipet

5.0 REAGENTS

a) Hydrochloric acid (HCl) conc.
b) Barium chloride solution: Dissolve 100 g $BaCl_2 \times 2H_2O$ in 1L nanopure water. Filter through a membrane filter before use. 1 mL is capable of precipitating approximately 40 mg SO_4^{2-}. Record in prep logbook.
c) Standard sulfate solution: Dissolve 0.1479 g anhydrous Na_2SO_4 in nano water and dilute to 1L. 1.00 mL = 100 ug SO_4^{2-}

Note: All chemicals should conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society (A.C.S. reagent).

6.0 SAMPLE ANALYSIS

Prepare crucibles by washing, fitting with two 934 AH filter pads and filtering through a half crucible of nano water to make sure the pads are snug. Place in drying oven for at least one hour, desiccate for one hour, and weigh on analytical balance. (Be sure the Gooch I.D. number is recorded.)

Prep the same amount of beakers with 1:1 HNO_3 and nano water. Clean millipore filter apparatus in the same way.

Filter a quantity of unpreserved sample through a 0.45 um millipore and from the filtrate, measure ~~50~~ 100 mL in a graduated cylinder. Pour into a labeled beaker. Repeat for each sample and blank, duplicate and matrix spike.

Place beakers on a hot plate until they are hot but not boiling! Add 1-2 drops concentrated HCl and approximately 5 mL $BaCl_2$ to each. A white precipitate will form in the presence of SO_4^{2-}. Cool samples and cover to prevent contamination.

Filter samples through a weighed Gooch crucible. Add another 5 mL $BaCl_2$ to the filtrate to ensure all the SO_4^{2-} has been precipitated. If the filtrate turns white, filter it through the crucible again. Put the crucibles in the drying oven (at 105°C) for at least one hour, desiccate for an hour and weigh back on the same analytical balance.

7.0 QUALITY CONTROL

Every 20 samples a duplicate and matrix spike are required. Spike with ~~1,000~~ 500 ug SO_4^{2-} (~~10~~ 5 mLs of sulfate standard where 1 mL = ~~100~~ 1000 ug) to ~~50~~ 100 mL of sample.

a) Calculation

$$\text{mg } SO_4^{2-}/L = \frac{\text{mg } BaSO_4 \times 411.6}{\text{mL sample}}$$

b) Spike recovery

$$\% \text{ recovery} = \frac{(SSR - SR)}{SA} \times 100$$

where: SSR = spiked sample result (mg/L)
SR = sample result (mg/L)
SA = spike added (mg/L)

The acceptable range is 75-125%.

c) Duplicates - relative percent difference (rpd) is used to determine the precision of results.

$$RPD = \frac{[S-D]}{(S+D)/2} \times 100$$

where S = sample result (mg/L)
D = duplicate result (mg/L)

The RPD value must be ≤20%.

Significant figures = 3
LDL (Lower Detectable Limit) = 10.0 mg/L

8.0 REPORTING OF RESULTS

Results are computed for samples on LIMS and may have to be modified to take LDL's and significant figures into account.

9.0 DOCUMENTATION OF PROBLEMS

Corrective action reports must be filed if any problems were associated with the analysis.

Case narratives should include any departure from normal procedure or any difficulties encountered in the analysis.

REFERENCES

Standard Methods for the Examination of Water and Wastewater, 16th Edition, 1985 (and revisions).

APPENDIX I:

BENCH SHEETS, REAGENT LOGS AND CORRECTIVE ACTION FORM

Sulfates

IEA
200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

Sample ID	Batch ID	Sample vol. mls.	Gooch ID	Tare weight(g)	Final weight(g)	Sulfate mg/l
	PB					
	LCS					
	DUP					
	SPIKE					
______ Class S Weight		______	______			______

Stock Code ID ____________________

Spike Amount Added ______________

Analyst ________________

Date ________________

Page ______

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200 Monroe Turnpike
Monroe, CT 06468 (203) 452-8200

CLASSICAL CHEMISTRY QA/QC SUMMARY

Analyte:_____ Date:_____ Ref:_____

LABORATORY CONTROL SAMPLE

Reference	True Value	Value Found	% Recovery

SAMPLE SPIKE

Lab Sample ID	Spike Result	Sample Result	Amount Added	% Recovery

DUPLICATES

Lab Sample ID	Result 1	Result 2	Mean	RPD

PREP BLANK

Blank ID	Result

Samples in Batch:	Comments:

301388

IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____

_____

_____

_____

_____

_____

_____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

301389

an environmental testing company
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Monroe, Connecticut 06468
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Standard Operating Procedure
Analysis of Hardness in Water

WC:081390:0

301390

Miramar, Florida 305-989-0928
Schaumburg, Illinois 708-705-0740
N. Billerica, Massachusetts 617-272-5212
Whippany, New Jersey 201-428-8181
Cary, North Carolina 919-677-0090
Essex Junction, Vermont 802-878-5138

TABLE OF CONTENTS

Hardness

1.0 Scope and Application

1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial waters.

1.2 The method is suitable for all concentration ranges of hardness; however, in order to avoid large titration volumes, use a sample aliquot containing not more than 25 mg CaCO3.

2.0 Summary of Method

Ethylenediaminetetraacetic acid and its sodium salts (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of dye such as Eriochrome Black T of Calmagite is added to an ageous solution containing calcium and magnesium ions at a pH of 10.0 $\pm$ 0.1, the solution becomes red wine, If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the magnesium and calcium has been compexed the solution turns from wine red to blue, marking the end point of the titration. Magnesium ion must be present to yield a satisfactory end point. To insure this, a small amount of complexometrically neutral magnesium salt of EDTA is added to the buffer; this automatically introduces sufficient magnesium and obviates the need for a blank correction.

The sharpness of the endpoint increases with increasing pH. However, the pH cannot be increased indefinitely because of the danger of precipitating calcium carbonate, CaCO3, or magnesium hydroxide, Mg(HO)2, and because the dye changes color at high values. The specified pH of 10.0 $\pm$ 0.1 is a satisfactory compromise. A limit of 5 min. is set for the duration of the titration to minimize the tendency toward CaCO3 precipitation.

3.0 Interferences

Some metal ions interfere by causing fading or indistinct endpoints of by stoichiometric consumption of EDTA. Reduce this interference by adding certain inhibitors, such as mg CDTA, before titration. When higher concentrations of heavy metals are present, determine calcium and magnesium by a non-EDTA method and obtain hardness by calculation.

4.0 Apparatus

4.1 Standard laboratory titrimetric equipment.

5.0 Reagents

5.1 Buffer solution

5.1.1 If magnesium EDTA ia available: Dissolve; 16.9g NH4CL in 143ml conc. NH4OH in a 250ml volumetric, add 125g of magnesium salt of EDTA and dilute to the mark with distilled water. THen go to 5.1.3.

5.1.2 If magnesium EDTA ia unavailable: Dissolve 1.179g disodium EDTA (analytical reagent grade) and 780mg $MgSO4 \cdot 7H2O$ (or 644mg $MgCL2 \cdot 6H2O$) in 50ml distilled water. Add this solution to a 250 ml volumetric flask containing 16.9g NH4CL and 143 ml conc. NH4OH with mixing and dilute to the mark with distilled water.

5.1.3 Store in tightly stoppered plastic bottle; stable for approximately one month. Dispense with bulb operated piped. Discard when 1 or 2ml added to sample fails to produce a pH of 10.0 $\pm$ 0.1 at end point of titration.

5.1.4 Commercially available "odorless buffers" which are nor stable, may be used.

5.2 Inhibitors: For most waters inhibitors are not necessary, If interfering ions are present use one of the following:

5.2.1 Inhibitor I: NaCN power. (Caution: extremely poisonous). Flush solutions on sample containing this down drain using large quantities of water. Make sure no acids are present which might liberate HCN gas.

5.2.2 Inhibitor II: Dissolve 5.0 g $Na2S \cdot H2O$ or 3.7 g $Na2S \cdot 5H2O$ in 100 ml distilled water. Exclude air with tightly filled rubber stopper. This gives sulfide precipitates which may obscure the end point if large quantities of heavy metals are present. Deteriorates rapidly through air oxidation.

5.2.3 Inhibitor III: Dissolve 4.5 g hydroxylamine hydrochloride in 100ml of 95% ethanol or isopropanol.

5.3 Indicator: Use a commercially available indicator such as Calmagite indicator (Mallinckrodt) or one of the formulations described below (5.3.1 -5.3.3)

5.3.1 Mix 0.5 g Eriochrome Black T with 4.5 g hydroxylamine hydrochloride. Dissolve in 100 ml of 95% ethanol or isopropanol.

5.3.2 Dissolve 0.5 to 1.0 g Eriochrome Black T in an appropriate solvent such as triethanolamine or 2-methoxyethanol. Stable approximately one week.

5.3.3 Mix together 0.5g Eriochrome Black T and 100g NaCI.

5.4 Standard EDTA titrant, 0.02 N: Place 3.723 g analytical reagent grade disodium ethylenediamine tetraacetate dihydrate, $Na_2H_2C_{10}H_{12}O_8N_2 \cdot H_2O$ In 1 liter volumetric flask and dilute to the mark with distilled water. Check with standard calcium solution (5.4.1) by titration (5.4.5). Store in polyethylene. Check periodically because of gradual deterioration.

5.4.1 Standard calcium solution 0.02 N: Place 1.000g anhydrous calcium carbonate (primary standard low in metals) in a 500 ml flask. Add a little at a time, 1+1 HCL (5.4.2) until all of the $CaCO_3$ has dissolved. Add 200 ml distilled water. Boil for a few minutes to expel CO_2. Cool. Add a few drops of methyl red indicator (5.4.3) and adjust to intermediate orange color by adding 3NNH4OH (5.4.4) or 1+1 HCL (5.4.2) as required. Quantitatively transfer to a 1 liter volumetric flask and dilute to mark with distilled water.

5.4.2 Hydrochloric acid solution, 1+1.

5.4.3 Methyl red indicator: Dissolve 0.10g methyl red in distilled water in a 100 ml volumetric flask and dilute to the mark.

5.4.4 Ammonium hydroxide solution, 3n: Dilute 210 ml of conc. NH4OH to 1 liter with distilled water.

5.4.5 Standardization titration procedure: Place 10.0 ml standard calcium solution (5.4.1) in vessel containing about 50ml distilled water. Add 1 ml buffer solution (5.1). Add 1-2 drops indicator(5.3) or small scoop of dry indicator (5.3.3). Titrate

slowly with continuous stirring until the last reddish tinge disappears, adding last few drops at 3-5 second intervals. At end point the color is blue. Total titration duration should be 5 minutes from the time of buffer addition.

$$N \text{ of EDTA} = \frac{0.2}{\text{ml of EDTA}}$$

5.5 Ammonium Hydroxide, 1N: Dilute 70ml of conc. NH_4OH to 1 liter with distilled water.

6.0 Sample Collection, Preservation and storage

6.1 Cool to 4 c, preserve with nitric acid (HNO_3) to pH <2.

7.0 Procedure

.1 Pretreatment

7.1.1 For drinking waters, surface waters, saline waters, and dilutions thereof, no pretreatment steps are necessary. Proceed to 7.2.

7.1.2 For most wastewaters, and highly polluted waters, the sample must be digested as given in the Atomic Absorption Methods section of this manual, paragraphs 4.1.3 and 4.1.4. Following this digestion, proceed to 7.2.

7.2 Titration of sample-normal to high hardness:

7.2.1 Sample should require <15 ml EDTA titrant (5.4) and titration should be completed within 5 minutes of buffer addition.

7.2.2 Place 25.0 ml sample in titration vessels, neutralize with 1N ammonium hydroxide (5.5) and dilute to about 50 ml.

7.2.3 Add 1 to 2 ml buffer solution (5.1).

7.2.4 If end point is not sharp (as determined by practice run) add inhibitor at this point (see 7.4).

7.2.5 Add 1 to 2 drops indicator solution (5.3.1.or 5.3.2) or small scoop of dried powder indicator formulation (5.3.3).

7.2.6 Titrate slow with continuous stirring with standard EDTA titrant (5.4) until last reddish tint disappears. Solution is normally blue at end point.

7.3 Titration of sample-low hardness (less than 5mg/1)

7.3.1 Use a larger sample (100ml)

7.3.2 Use proportionately larger amounts of buffer, inhibitor and indicator.

7.3.3 Use a microburet and run a blank amount using redistilled, distilled or deionized water.

7.4 To correct for interferences:

7.4.1 Some metal ions interfere by causing fading or indistinct end points. Inhibitors reduce this accord with the scheme below for 25.0 ml samples diluted to 50ml.

8.0 Quality Control

8.10 All stock standard preparations are logged and coded. All standards are labeled with the following: analyte, concentration, analyst's initials, and date prepared.

8.20 All chemicals should conform to minimum specifications set by the Reagent Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

8.30 A sample prep blank is analyzed with every batch of twenty or fewer samples.

8.40 Laboratory Control Sample (LCS) Analysis

One independent standard (LCS) is to be analyzed with each batch of twenty or fewer samples. The control limits fir the LCS are + or - 15 percent of the true value.

8.50 Duplicates

One duplicate must be analyzes from each of twenty or fewer samples. Samples identified as field blanks cannot be used for duplicate analysis. The relative percent differences (RPD) are calculated as follows:

$$RPD = \frac{(S - D)}{(S - D) / 2} \times 100$$

Where RPD = Relative Percent Difference
S = Original Sample Value
D = Duplicate Sample Value

A control limit of 20 percent for RPD shall be used for original and duplicate sample values greater that or equal to five times the reporting detection limit (RPD). If one result is aboye five times the RDL level and the other below, the difference between the two values should be no more than the RDL. If both sample values are less than the RDL, the RPD is not calculated.

8.6 Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the sample matrix and measurement methodology. At least one spike sample analysis must be performed on each batch of twenty or fewer samples. Samples identified as field blanks cannot be used for spoke sample analysis.

If the spike recovery is not within the control limits 75 to 125 percent and the sample concentration does not exceed four times the spiking level, a fresh aliquot of the sample is to be spike and analyze.

Spike recoveries are calculated as follows:]

$$\% \text{ Recovery} = \frac{(SSR-SR)}{SA}$$

Where sample concentration is less than the instrument detection limit, use SR = 0 for calculating percent recovery.

9.0 Method Performance

9.1 The average RPD over the last 25 duplicates was 2.18% as of 7/27/90. The average spike recovery over the sample period was 96.9%.

10.0 Data Handling and Reduction

10.1 All raw data including sample volume, titrant volume, and normality are entered into LIMS. The LIMS system calculates the results.

10.2 Manual Hardness Calculation :

$$\text{Hardness (EDTA) as mg CaCO3/L} = \frac{A \times B \times 100}{\text{ml sample}}$$

Where: A = ml titration for sample
B = mg CaCO3 equivalent to 1.00ml EDTA titration

10.3 The sample QA/QC and LCS are reported on the inorganics QA/QC summary form (see Appendix I).

10.4 Technical Data Review

The technical data review includes verifying that samples were preserved and holding times were met. All standards and batch quality control are checked to insure that they were within criteria. All results entered into LIMS are checked for transcription error against the raw data.

11.0 Documentation of Problems

11.1 Corrective Action Reports - must be filed if any problems are associated with the analysis (see Appendix II).

11.2 Case Narratives - should include any deviation from this SOP or any difficulties encountered in the procedure.

12.0 References

12.1 Methods of Chemical Analysis of Water and Wastes, EPA 600, 1983, Method 130.2.

APPENDIX I

301400

200 MONROE TURNPIKE • MONROE CONNECTICUT 06468 • (203) 452-5200

CLASSICAL CHEMISTRY QA/QC SUMMARY

E:_____ DATE:_____ REF.:_____

LABORATORY CONTROL SAMPLE

REFERENCE	TRUE VALUE	VALUE FOUND	% RECOVERY

SAMPLE SPIKE

LAB SAMPLE ID	SPIKE RESULT	SAMPLE RESULT	AMOUNT ADDED	% RECOVERY

DUPLICATES

LAB SAMPLE ID	RESULT 1	RESULT 2	MEAN	RPD

PREP BLANK

BLANK ID	RESULT

SAMPLES IN BATCH:	COMMENTS:

301401

IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____

_____

_____

_____

_____

_____

_____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

301402

an environmental testing company
200 Monroe Turnpike
Monroe, Connecticut 06468
(203) 261-4458
FAX (203) 268-5346

STANDARD OPERATING PROCEDURE

ANALYSIS OF NITRATE AND NITRITE

FOR WATER SAMPLES

WC:050390:0

301403

TABLE OF CONTENTS

Nitrate (as NO3- + NO2-)

1.0 SCOPE AND APPLICATION

1.1 The automated cadmium reduction method can determine nitrate and nitrite singly or together in potable, surface, and saline waters and domestic and industrial wastewaters. The range is 0.02-2.0 mg N/L, for soil and water samples.

2.0 SUMMARY OF METHOD

2.1 Nitrate is quantatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus initial nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl) ethylenediamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite alone also can be determined by removing the cadmium column.

3.0 INTERFERENCES

3.1 Build up of suspended matter in the cadmium reduction column will restrict sample flow. Since nitrate-nitrogen is soluble, the sample may be prefiltered.

3.2 Low results would be obtained for samples that contain high concentrations of iron, copper, or other metals. In this method, EDTA is added to the buffer to reduce this interference.

3.3 Samples that contain high concentrations of oil and grease should be pre-extracted with an organic solvent so not to coat the surface of the cadmium.

4.0 APPARATUS AND MATERIALS

4.1 Lachat autoanalyzer system

5.0 REAGENTS

5.1 15 M sodium hydroxide Add 150 g NaOH slowly to 250 mL of nanopure water. CAUTION: The solution will get very hot! Swirl until dissolved, cool and store in a plastic bottle.

5.2 Ammonium chloride buffer, pH = 8.5 - In a 1 L volumetric flask, dissolve 85.0 g ammonium chloride (NH4Cl) and 1.0 g disodium ethylenediamine tetraacetic acid dihydrate (Na2 EDTA. 2H20) in about 800 ml. Adjust the pH up to 8.5 with 15 M sodium hydroxide. Dilute to volume and invert three times.

5.3 Sulfanilamide color reagent - To a 1 L volumetric flask add about 600 mL of nanopure water. Then add 100mL of 85% phosphoric acid (H3P04), 40.0 g sulfanilamide, and 1.0 g N-1-naphthyl-ethylenediamine dihydrochloride (NED). Shake to wet and stir to dissolve (about 20 mins). Dilute to volume and invert three times. Store in a dark bottle. This solution is stable for one month.

5.4 Cadmium - Copper Reduction Column - refer to QuickChem method # 10-107-04-1-C.

5.5 Stock Nitrate solution - (2000 mg N/L as NO3-). In a 1L volumetric flask dissolve 14.44 g potassium nitrate (KNO3) in about 600 mL nano water. Add 2.0 mL chloroform. Dilute to volume and invert three times. This solution is stable for 6 months.

5.6 Standard Nitrate solution (10 mg N/L NO3-) In a 1L volumetric flask add about 600 ml nano water. With a class A pipet, transfer 5.0 ml of stock nitrate solution (2000 mg/L) to volumetric and dilute to mark. Invert three times.

5.7 Nitrate working standards - Dilute as follows:

	MLS of 10 mg/L Std		Concentration of Standards
A	50.0	*Note:*	2.0 mg/L
B	20.0		0.8 mg/L
C	10.0	Taken to volume with	0.4 mg/L *
D	2.5	nano water in 250 mL	0.1 mg/L
E	1.0	volumetric flasks.	0.04 mg/L
F	0.5		0.02 mg/L

* Where standard C (0.4 mg/L) is used as the check standard.

5.8 Stock Nitrite Solution (2000 mg N/L as NO2-). In a 1 L volumetric flask dissolve 9.86 g of sodium nitrite (NaNO2) or 12.14 g of potassium nitrite (KNO2) in approximately 800 mL water. Add 2 mL chloroform and dilute to volume. KEEP REFRIGERATED!

5.9 Standard Nitrite Solution (10 mg N/L NO2-). In a 1 L volumetric flask add about 600 mL nano water. Using a class A 5.0 mL pipet, transfer 5 mL of stock nitrite solution (2000 mg/L) and take to volume.

5.10 Standard Nitrite Solution (1.0 mg N/L NO2-). In a 100 mL volumetric flask pipet, add 10.0 mL of nitrite standard solution (10 mg N/L) and dilute to volume.

5.11 Nitrite working standards - Dilute as follows:

	MLS of 10 mg/L Std	MLS of 1.0 mg/L Std		Concentration of Standards
A	50.0	-----	Taken to volume	2.0 mg/L
B	20.0	-----	with nano water	0.8 mg/L
C	10.0	-----	in 250 mL	0.4 mg/L
D	2.5	-----	volumetric flasks	0.1 mg/L
E	1.0	-----		0.04 mg/L
F	-----	1.0	<----Taken to volume in 200 mL volumetric	0.005 mg/L

6.0 SAMPLE HANDLING AND PRESERVATION

6.1 Sample to be analyzed for Nitrite should be analyzed as soon as possible. Samples may be stored up to 48 hours at 4° C.

6.2 Samples to be analyzed for Nitrate-Nitrate should be preserved with H2SO4 (pH <2) and stored at 4° C. The Nitrate-Nitrite holding time is 28 days from the time of sampling.

7.0 PROCEDURE

Nitrate + Nitrite:
Inject to start of peak period: 22 s
Inject to end of peak period: 58 s

Cycle period: 40 s
Load period: 20 s
Inject period 20 s

Nitrite (no column):
Inject to start of peak period: 15 s
Inject to end of peak period: 51 s

GAIN:

Gain = 260 x 1

7.10 System Operation

7.11 Inspect modules for proper connections.

7.12 Turn on power and all modules, except sampler.

7.13 Place reagent feedlines into proper containers. Raise tension levers on pump tube cassettes.

7.14 Stop the pump and place reduction column in the manifold for NO3-. Start the pump. For NO2-, do not connect the cadmium column.

7.15 Establish a stable baseline. Set zero on colorimeter. Manually inject a high standard to check gain.

7.16 Place calibration standards and blank in sample tray in descending order of concentration followed by unknowns and check standards.

7.17 At end of run turn off the pump and remove reduction column from manifold and connect ends with a union, taking care to avoid introduction of air bubbles.

7.18 Turn on the pump and place all feedlines in water. Flush system and pump dry.

7.19 Turn off pump, all modules and release tension levers on pump tube cassettes.

System Notes: The Cd column efficiency should be above 90% if working properly. To check this, inject one or several different nitrite standards followed by one or several different nitrate standards. Fit the nitrite standards' responses to a line. The ratio of the slope of the nitrate/nitrite lines x 100% is the efficiency of the column. If the column is less than 80% efficient, it should be repacked with freshly copperized Cd (Reagents, 5.).

8.0 QUALITY CONTROL

8.10 All stock standard preparations are logged and coded. All standards are labeled with the following: analyte, concentration, analyst's initials, and date prepared.

8.20 All chemical should conform to minimum specifications set by the Regeant Chemicals Committee of the American Chemical Society. All chemical inventories are used on a first in first out basis.

8.30 Tuning and Calibration of Instrument

8.31 The baseline is adjusted to a colorimeter LED output of approximately -0.200.

8.32 The high standard is manually injected and the gain set in such a manner that the peak height is the size of the background analytical window. The high standard manual injection is repeated to verify the peak height criteria.

8.33 The instrument is then calibrated with the standards in section 5.7 or 5.11 depending on whether nitrate-nitrites or nitrites are being analyzed.

8.40 Initial Calibration Verification (ICV)

Immediately after the instrument has been calibrated, the accuracy of the initial calibration shall be verified by the analyst of the ICV. The control limit for the ICV is + or - 15 percent of the true value.

8.50 Continuing Calibration Verification (CCV)

To ensure calibration accuracy during each analysis run, a mid-range standard must be analyzed at a frequency of 10 percent of samples. The standard must be analyzed at the beginning of the run, after ten samples, and after the last analytical sample. The same continuing calibration standard must be used throughout the analysis run. If the deviation of the CCV is greater than the control limit + or - 15 percent of the true value, the instrument must be recalibrated and all analytical samples analyzed since the last acceptable calibration verification must be re-analyzed.

8.60 Initial Calibration Blank (ICB), Continuing Calibration Blank (CCB), and Preparation Blank (PB) Analyses

A calibration blank must be analyzed after every initial and continuing calibration verification. If the absolute value blank result exceeds the contract required detection limit (CRDL), terminate analysis, correct problem, recalibrate and

reanalyze all analytical samples run after the last acceptable blank.

At least one preparation blank (or reagent blank) must be analyzed with every batch of twenty samples or less.

8.70 Spike Sample Analysis (S)

The spike sample analysis is designed to provide information about the effect of the sample matrix and measurement methodology. At least one spike sample analysis must be performed on each batch of twenty or fewer samples. Samples identified as field blanks cannot be used for spike sample analysis.

If the spike recovery is not within the control limits 75 to 125 percent and the sample concentration does not exceed four times the spiking level, a fresh aliquot of the sample is to be spike and analyzed.

Spike recoveries are calculated as follows:

$$\% \text{ Recovery} = \frac{(SSR-SR)}{SA} \times 100$$

where SSR = Spiked Sample Result
SR = Sample Result
SA = Spike Added

When sample concentration is less than the instrument detection limit, use SR = 0 for calculating percent recovery.

8.80 DUPLICATES

One duplicate must be analyzed from each batch of twenty or fewer samples. Samples identified as field blanks cannot be used for duplicate analysis. The relative percent differences (RPD) are calculated as follows:

$$RPD = \frac{(S-D)}{(S+D)/2} \times 100$$

where RPD = Relative Percent Difference
S = Original Sample Value
D = Duplicate Sample Value

A control limit of 20 percent for RPD shall be used for original and duplicate sample values greater than or equal to five times the CRDL. If one result is above 5x the CRDL level and the other is below, the difference between the two values should be no more than the CRDL. If both sample values are less than the IDL, the RPD is not calculated.

8.90 LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

One independent standard (LCS) is to be analyzed with each time the instrument is

calibrated. The control limits established for the LCS are + or - 15 percent of the true value.

8.10 INSTRUMENT DETECTION LIMIT (IDL) DETERMINATION

Before any field samples are analyzed, the instrument detection limits must be determined quarterly.

The instrument detection limits shall be determined by multiplying by three, the standard deviations obtained from the analysis of a standard solution at a concentration 3x-5x the CRDL, with seven consecutive measurements.

8.11 INSTRUMENT MAINTENANCE

Separate maintenance logs are kept for each instrument.

9.0 METHOD PERFORMANCE

9.1 An instrument detection limit study performed on 03/04/90 yielded a 0.02 mg/L as N detection limit fir nitrate-nitrite.

9.2 The last 25 sample matrix spikes (0.400 mg/L as N) average recovery was 96.1 percent as of 01/12/90. The average RPD over the same period was 6.12 percent.

10.0 DATA REDUCTION AND DATA HANDLING

10.1 All sample ID's and dilution factors are entered into instrument controlling PC and are printed out with the tabular results.

10.2 All results are entered into LIMS for reporting and sample tracking purposes. The tabular results are filed by analyte and in chronological order in the raw data file.

10.3 The sample QA/QC is reported on the Inorganics QA/QC Summary form (see Appendix II).

10.4 Technical Data Review

The technical data review includes verifying that samples were preserved and holding times were met. All standards and batch quality control are checked to insure that they were within criteria. All results entered into LIMS are checked for transcription error against the raw data.

11.0 DOCUMENTATION OF PROBLEMS

11.1 Corrective Action Reports - must be filed if any problems are associated with the analysis (see Appendix II).

11.2 Case Narratives - should include any deviation from this SOP or any difficulties encountered in the procedure.

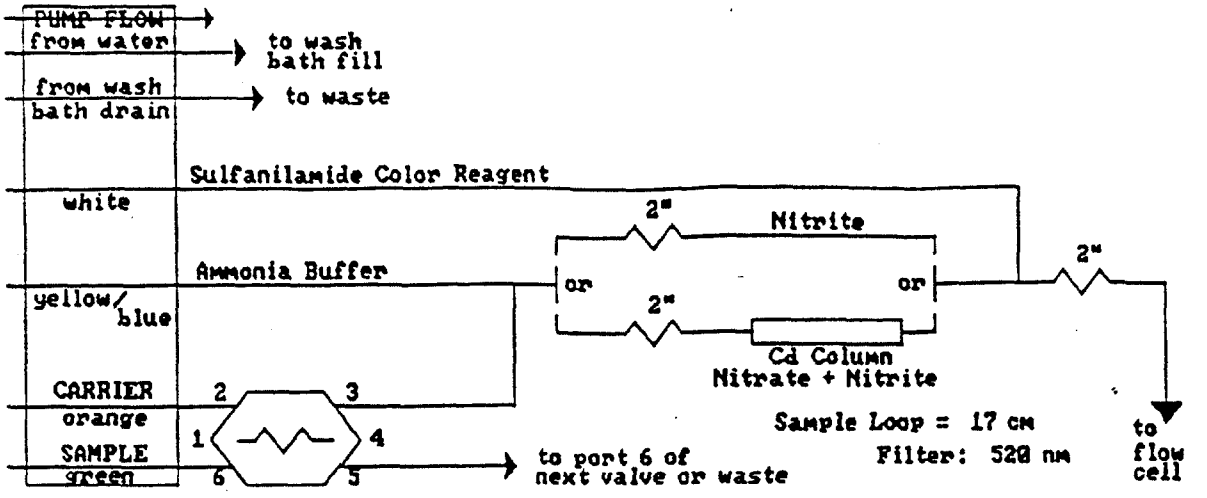
12.0 REFERENCES

12.1 Methods for QUIKCHEM Automated Ion Analyzer, Lachat Instruments, Method 10-107-04-1-C, 1986.

12.2 Methods for Chemical Analysis of Water and Wastes, EPA 600, 1986.

Appendix I

Manifold Diagram:



CARRIER is water.

1"	is	70.0	cm of tubing on a 1 in coil support
2"	is	135	cm of tubing on a 2 in coil support
2.5"	is	168	cm of tubing on a 2.5 in coil support
3"	is	202	cm of tubing on a 3 in coil support
4"	is	255	cm of tubing on a 4 in coil support
8"	is	550	cm of tubing on a 8 in coil support

Heated tubing is shown inside a box with the temperature next to the box. Heated tubing is **650** cm unless otherwise specified.

All manifold tubing is **0.8** mm (0.032 in) i.d. This is 5.2 uL/cm.

Notes: A new manifold includes two packed cadmium columns.

MANIFOLD DIAGRAM REVISION DATE: 30 December 1986

Appendix II

IEA/CT

Group(s): _____

Date: _____

CORRECTIVE ACTION REPORT

Problem/Question reported by: _____

Job/Case Number: _____ Sample Number(s): _____

Description of Problem: _____

_____

_____

_____

_____

_____

_____

Summary of Corrective Action(s): _____

_____

_____

_____

_____

_____

_____

Is this a recurring Problem? _____

Should SOP be modified or updated? _____

Cooperative help of the following groups/people is requested: _____

_____

Approval of Group/Section leader: _____

Distribution: Section/Group Leader
Lab Manager
QA Officer
Other: _____

301415

INORGANICS QA/QC SUMMARY

Analyte:_____ Date:_____

Notebook Ref:_____

SPIKES

Lab Sample ID	Spike Result	Sample Result	Amount Added	Percent Recovery

DUPLICATES

ab Sample ID	Result 1	Result 2	Mean	RPD

BLANKS

Blank ID	Result

Samples in Batch:	Comments:

APPENDIX B

SECTION III: QUALITY ASSURANCE PROJECT PLAN

APPENDIX B

ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

APPENDIX B

ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

Volatile Organic Compounds	CAS Number	Estimated Quantitation Limit Water* (ug/L)
Chloromethane	74-87-3	1.0
Bromomethane	74-83-9	1.0
Vinyl chloride	75-01-4	1.0
Chloroethane	75-00-3	1.0
Methylene chloride	75-09-2	2.0
Acetone	67-64-1	5.0
Carbon disulfide	75-15-0	1.0
1,1-Dichloroethene	75-35-4	1.0
1,1-Dichloroethane	75-34-3	1.0
1,2-Dichloroethene (total)	540-59-0	1.0
Chloroform	67-66-3	1.0
1,2-Dichloroethane	107-06-2	1.0
2-Butanone	78-93-3	5.0
1,1,1-Trichloroethane	71-55-6	1.0
Carbon tetrachloroethane	56-23-5	1.0
Vinyl acetate	108-05-4	1.0
Bromodichloromethane	75-27-4	1.0
1,2-Dichloropropane	78-87-5	1.0
cis-1,3-Dichloropropene	10061-01-5	1.0
Trichloroethene	79-01-6	1.0
Dibromochloromethane	124-48-1	1.0
1,1,2-Trichloroethane	79-00-5	1.0
Benzene	71-43-2	1.0
trans-1,3-Dichloropropene	10061-02-6	1.0
Bromoform	75-25-2	1.0
4-Methyl-2-pentanone	108-10-1	5.0
2-Hexanone	591-78-6	5.0
Tetrachloroethene	127-18-4	1.0
Toluene	108-88-3	1.0
1,1,2,2-Tetrachloroethane	79-34-5	1.0
Chlorobenzene	108-90-7	1.0
Ethyl benzene	100-41-4	1.0
Styrene	100-42-5	1.0
Xylenes (total)	1330-20-7	1.0

CAS Chemical abstract service.
* Analysis by USEPA Contract Laboratory Program Statement of Work for Low Concentration Water for Organic Analysis, 4/90.
** Analysis by USEPA Contract Laboratory Program Statement of Work for Organic Analysis, 6/91.
*** Analysis by USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, 3/90.
ug/L Micrograms per liter.
mg/L Milligrams per liter.

301419

APPENDIX B

ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

Semivolatile Organic Compounds	CAS Number	Estimated Quantitation Limit Water** (ug/L)
Phenol	108-95-2	10
bis(2-Chloroethyl)ether	111-44-4	10
2-Chlorophenol	95-57-8	10
1,3-Dichlorobenzene	541-73-1	10
1,4-Dichlorobenzene	106-46-7	10
Benzyl alcohol	100-51-6	10
1,2-Dichlorobenzene	95-50-1	10
2-Methylphenol	95-48-7	10
bis(2-Chloroisopropyl)ether	108-60-1	10
4-Methylphenol	106-44-5	10
N-Nitroso-di-n-dipropylamine	621-64-7	10
Hexachloroethane	67-72-1	10
Nitrobenzene	98-95-3	10
Isophorone	78-59-1	10
2-Nitrophenol	88-75-5	10
2,4-Dimethylphenol	105-67-9	10
Benzoic acid	65-85-0	50
bis(2-Chloroethoxy)methane	111-91-1	10
2,4-Dichlorophenol	120-83-2	10
1,2,4-Trichlorobenzene	120-82-1	10
Naphthalene	91-20-3	10
4-Chloroaniline	106-47-8	10
Hexachlorobutadiene	87-68-3	10
4-Chloro-3-methylphenol	59-50-7	10
2-Methylnapthalene	91-57-6	10
Hexachlorocyclopentadiene	77-47-4	10
2,4,6-Trichlorophenol	88-06-2	10
2,4,5-Trichlorophenol	95-95-4	50
2-Chloronaphthalene	91-58-7	10
2-Nitroaniline	88-74-4	50
Dimethylphthalate	131-11-3	10
Acenaphthylene	208-96-8	10
2,6-Dinitrotoluene	606-20-2	10
3-Nitroaniline	99-09-2	50
Acenaphthene	83-32-9	10
2,4-Dinitrophenol	51-28-5	50
4-Nitrophenol	100-02-7	50

CAS Chemical abstract service.
* Analysis by USEPA Contract Laboratory Program Statement of Work for Low Concentration Water for Organic Analysis, 4/90.
** Analysis by USEPA Contract Laboratory Program Statement of Work for Organic Analysis, 6/91.
*** Analysis by USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, 3/90.
ug/L Micrograms per liter.
mg/L Milligrams per liter.

APPENDIX B

ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

Semivolatile Organic Compounds	CAS Number	Estimated Quantitation Limit Water** (ug/L)
Dibenzofuran	132-64-9	10
2,4-Dinitrotoluene	121-14-2	10
Diethylphthalate	84-66-2	10
4-Chlorophenyl-phenylether	7005-72-3	10
Fluorene	86-73-7	10
4-Nitroaniline	100-01-6	50
4,6-Dinitro-2-methylphenol	534-52-1	50
N-nitrosodiphenylamine	86-30-6	10
4-Bromophenyl-phenylether	101-55-3	10
Hexachlorobenzene	118-74-1	10
Pentachlorophenol	87-86-5	50
Phenanthrene	85-01-8	10
Anthracene	120-12-7	10
Di-n-butylphalate	84-74-2	10
Fluoranthene	206-44-0	10
Pyrene	129-00-0	10
Butylbenzylphthalate	85-68-7	10
3,3'-Dichlorobenzidine	91-94-1	20
Benzo(a)anthracene	56-55-3	10
Chrysene	218-01-9	10
bis(2-Ethylhexyl)phthalate	117-81-7	10
Di-n-octylphthalate	117-84-0	10
Benzo(b)fluoranthene	205-99-2	10
Benzo(k)fluoranthene	207-08-9	10
Benzo(a)pyrene	50-32-8	10
Indeno(1,2,3-cd)pyrene	193-39-5	10
Dibenz(a,h)anthracene	53-70-3	10
Benzo(g,h,i)perylene	191-24-2	10
PCB Compounds		
Aroclor-1016	12674-11-2	0.5
Aroclor-1221	11104-28-2	0.5
Aroclor-1232	11141-16-5	0.5
Aroclor-1242	53469-21-9	0.5
Aroclor-1248	12672-29-6	0.5
Aroclor-1254	11097-69-1	1.0
Aroclor-1260	11096-82-5	1.0

CAS Chemical abstract service.
* Analysis by USEPA Contract Laboratory Program Statement of Work for Low Concentration Water for Organic Analysis, 4/90.
** Analysis by USEPA Contract Laboratory Program Statement of Work for Organic Analysis, 6/91.
*** Analysis by USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, 3/90.
ug/L Micrograms per liter.
mg/L Milligrams per liter.

APPENDIX B

ANALYTICAL PARAMETERS AND QUANTITATION LIMITS

Inorganic Parameters	Estimated Quantitation Limit*** (ug/L)
Antimony	60
Arsenic	10
Beryllium	5
Cadmium	5
Chromium	10
Copper	25
Lead	3
Mercury	0.2
Nickel	40
Selenium	5
Silver	10
Thallium	10
Zinc	20
Cyanide	10

Leachate Indicator Parameters	USEPA Method Reference Number	Estimated Quantitation Limit
Total dissolved solids	160.1	5 mg/L
Ammonia	350.2	0.04 mg/L
Chloride	325.2	3 mg/L
Total hardness	130.2	1 mg/L
Nitrate	353.2	0.1 mg/L
Sulfate	375.3	10 mg/L
Alkalinity	310.1	1 mg/L
Iron	CLP	100 ug/L
Potassium	CLP	5000 ug/L
Sodium	CLP	5000 ug/L
pH (field)	–	0.1 unit
Specific conductance (field)	–	0.1 umhos/cm
Temperature (field)	–	0.1 degrees C

CAS Chemical abstract service.
* Analysis by USEPA Contract Laboratory Program Statement of Work for Low Concentration Water for Organic Analysis, 4/90.
** Analysis by USEPA Contract Laboratory Program Statement of Work for Organic Analysis, 6/91.
*** Analysis by USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, 3/90.
ug/L Micrograms per liter.
mg/L Milligrams per liter.

SECTION 4

SECTION IV

HEALTH AND SAFETY PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

CONTENTS

SECTION IV: HEALTH AND SAFETY PLAN

301425

APPENDICES

A. Site Visitors Log.

B. Tailgate Safety Meeting Form.

C. Accident Reporting Form, OSHA 101.

D. Utilities and Structures Checklist.

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SECTION IV

HEALTH AND SAFETY PLAN
SECOND OPERABLE UNIT
REMEDIAL INVESTIGATION
SYOSSET LANDFILL, SYOSSET, NEW YORK

1.0 INTRODUCTION

This Health and Safety Plan (HASP) has been developed to address the potential physical and chemical hazards that field personnel may face while performing the Second Operable Unit (OU-2) Remedial Investigation (RI) of the Syosset Landfill. This HASP has been developed to meet the requirements of the Occupational Safety and Health Administration (OSHA) regulation, Title 29, Code of Federal Regulations, Part 1910.120 (29 CFR 1910.120), "Hazardous Waste Operations and Emergency Response." It is intended for the protection of the workers and establishes procedures to minimize workers' exposures through the use of personal protective equipment and safe work practices.

2.0 RESPONSIBILITIES

The field hydrogeologist is designated as the site safety officer (SSO) who will be responsible for implementing the safe work practices and procedures that are established in this HASP. Whenever the SSO must leave the site while the work is in progress, an alternate SSO will be designated to ensure that the HASP will continue to be followed. The SSO will report all health and safety matters to the project manager who has responsibility for overseeing the planned field activities. The project health and safety coordinator (Geraghty & Miller Northeast Regional Health and Safety Manager) will be available on an as-needed basis. The project director for this investigation has overall responsibility for assuring that the HASP is implemented properly.

3.0 SITE DESCRIPTION

The Syosset Landfill encompasses approximately 38 acres and is located in the Town of Oyster Bay, Syosset, New York. The topography of the landfill is relatively flat and is similar in elevation to the surrounding area. The landfill is surrounded by a 6-foot high cyclone fence, which limits access to it. A residential area and the South Grove Elementary School border the site to the north and northeast. The proposed ground-water monitoring wells are located in this area and the proposed gas monitoring wells are located on Great Eastern Printing Company property located southwest of the landfill (Figure 1). The OU-2 RI will also present and evaluate gas monitoring results obtained from on-site gas Cluster Wells CW-2, 4, 5, 6, 7, and 8 which will be installed and monitored during the OU-1 Remedial Design Program (see Figure 1). Additional details on the site description and its history are provided in the OU-1 (on-site) RI report (Geraghty & Miller, Inc. 1989).

4.0 PLANNED FIELD ACTIVITIES

The following field tasks will be performed during the OU-2 RI.

- Installation of on-site and off-site ground-water monitoring wells.
- Installation of off-site gas monitoring wells.
- Sampling of new and existing ground-water monitoring wells.
- Monitoring of new and existing gas monitoring wells.

Detailed descriptions of the RI field program are contained in the Sampling and Analysis Plan (SAP) (Section II).

5.0 PERSONNEL TRAINING

Anyone entering the work zone during the RI field program will be required to have completed an OSHA-required initial health and safety training course (24 or 40 hours). In addition, the project manager will have completed the 8-hour required supervisor and manager's class. Each worker must also have completed the 8-hour refresher course, as required.

6.0 HAZARD EVALUATION

The potential physical and chemical hazards associated with the OU-2 RI field program were evaluated based on the existing data base primarily from the OU-1 RI and the field tasks that will be performed during the OU-2 RI.

6.1 PHYSICAL HAZARDS

The physical hazards associated with the planned field activities include the potential for workers being struck by falling objects while working near the drill rig; being splashed with liquids during well installation and well sampling; slips or falls; and noise exposure during the installation of monitoring wells. Heat stress is another potential physical hazard that may occur during the summer months. Finally, underground utilities will be marked out before drilling begins and measures will also be taken to monitor for the potential presence of landfill gas.

6.2 CHEMICAL HAZARDS

The potential chemical hazard of greatest concern with respect to the planned field activities is the possible inhalation of vapors containing volatile organic compounds (VOCs). VOCs were detected in the low parts per billion (ppb) range in some of the ground-water monitoring wells and/ or gas monitoring wells during the OU-1 RI but not at consistent concentrations or distributions and may not be related to the landfill. Notwithstanding the low detection of VOCs, protective measures will be followed to ensure worker and community health and safety during the OU-2 RI field program.

7.0 AIR MONITORING

Air monitoring for VOCs and the Lower Explosive Limit (LEL) will be conducted at this site during the ground-water and gas well installation programs to ensure that the workers are adequately protected from the potential inhalation of vapors containing VOCs and explosion hazard. A photoionization detector (PID) such as a TIP or HNU meter, will be used for monitoring VOCs. These instruments are designed to measure VOCs in air in the low parts per million (ppm) range. The PID will be calibrated each morning before use (see Appendix E of the SAP [Section II]). Calibration records will be maintained. In order to quantify specific compounds, Draeger tubes will be used (see Section 8.0 [Action Levels] of this HASP). A combustible gas indicator will be used to monitor the air inside each boring as drilling proceeds (cased borings and auger borings only) to determine the explosion potential in each boring.

8.0 ACTION LEVELS

Although VOCs were detected in some of the on-site ground-water monitoring wells, the concentrations were similar to background water-quality conditions for the area (Geraghty & Miller, Inc. 1989). Similarly, VOCs that were detected in some of the on-site gas monitoring wells were not detected at a consistent concentration or distribution. As stated in the Section 9.0 of this HASP, work will be conducted under Level D protection. However, protective measures will be followed to minimize the exposure of field personnel to the potential presence of these compounds in the work zone. This will be accomplished by monitoring the air in the breathing zone for VOCs using a PID. A summary of the VOCs that were found at this site and their current occupational exposure limits is presented in Table 5.

The following procedure for air monitoring has been established for off-site ground water study and subsurface gas study to evaluate whether actual field conditions necessitate an upgrade in the level of protection. This procedure entails the establishment of action levels for the VOCs detected at the site having the lowest occupational exposure limits (see Table 5). These compounds are vinyl chloride and benzene. An action level of 1 ppm PID needle deflection for a sustained period of 5 minutes in the worker's breathing zone has been established for the presence of vinyl chloride and benzene. If the action level is exceeded, Draeger tubes for vinyl chloride and benzene will be used to determine whether either compound is present. If the action level is exceeded for either compound, then the work will be discontinued. The level of protection will be upgraded to Level C before work resumes. This level requires that a full-face air purifying respirator equipped with organic vapor cartridges be used. Draeger tubes for vinyl chloride and benzene tubes will be used every 30 minutes to measure the concentrations of these compounds. If the concentration of both compounds are below their respective action levels, then downgrading to Level D will be considered.

Measurements will be taken during the drilling of each well boring for the presence of explosive vapors using a combustible gas indicator. Work will be discontinued if the meter readings are 20 percent of the LEL or greater at the borehole opening. Work will not resume until the meter reading drops to below 20 percent of the LEL. If the reading does not drop to below 20 percent within a reasonable period of time (e.g., 1/2 hour); then water will be added to the borehole to suppress the explosive vapors.

9.0 LEVELS OF PROTECTION

Based upon the hazard evaluation results, all tasks will be performed in Level D protection. In the event that the established action level for vinyl chloride and/or benzene is exceeded, the level of protection will be upgraded to Level C (see Section 7.0 [Air Monitoring] of this HASP). The following is a description of the personal protective equipment required for each level:

Level D

- Hard hat.
- Disposable coveralls (optional).
- Safety glasses, goggles, or faceshield.
- Steel-toe and shank, chemical-resistant boots.
- Chemical-resistant gloves (optional except when handling soil, sediment or surface water).
- Hearing protection, NRR of 35 decibels (mandatory during drilling).

Level C

- Hard hat
- Disposable coveralls (optional).
- Safety glasses, goggles, or faceshield.
- Steel-toe and shank, chemical-resistant boots.
- Chemical-resistant gloves (optional except when handling soil, sediment or surface water).
- Hearing protection, NRR of 35 decibels (mandatory during drilling).
- Full face air purifying respirator equipped with organic vapor cartridges

If the noise levels are found to be disrupting to the community, noise reduction measures will implemented.

10.0 SITE CONTROL

Prior to the start of the field activities, the SSO will be responsible for the designation of the work, support, and clean zones. The work zone will be an area surrounding the immediate work being performed where the greatest potential hazards exist. Only the necessary workers required to perform the work will be permitted in this zone. If there is more than one work zone at any time, then a communication network (walkie-talkies) will be established. A support zone will be established for equipment storage and personnel decontamination. A clean zone will be established for site control of visitors, equipment deliveries, and communications.

For the off-site work activities, the SSO will take additional measures to secure the work area by barricading the area and/or sign posting, in order to deter unauthorized personnel from entering the work area.

11.0 PERSONNEL DECONTAMINATION

A personnel decontamination station will be established in the support zone. Personnel decontamination will consist of washing potentially contaminated items, such as boots and gloves with a mild soap and water solution and a water rinse. Decontamination water will be disposed at a designated location at the landfill. All personnel leaving the work zone will go though the decontamination process before leaving the work area.

12.0 SAFE WORK PRACTICES

An itemized summary of the safe work practices that will be followed during the RI field program is provided below:

- Anyone entering the work zone must have completed an OSHA-required initial health and safety training course. This includes 24 or 40 hours of initial training and an 8-hour annual refresher training course.

- All Geraghty & Miller site personnel are participants of the company's medical surveillance program.

- A copy of the HASP will be available for reference at the site during the planned field activities.

- Dust suppression, using a water spray, will be used when needed to reduce airborne particulates during the field activities.

- A pre-entry, tailgate safety meeting will be conducted prior to the start of each task to discuss the associated hazards.

- The SSO will inform all subcontractors of the potential hazards associated with the site and the planned field activities. A copy of the HASP will be made available for their review.

- No eating, drinking, or smoking will be permitted in the work and support zones.

- No sources of ignition, such as matches or lighters, will be permitted in the work and support zones.

- Before the field program begins, the Police Department, Fire Department, and hospital will be notified when and where the work will be conducted.

- The buddy system will be used in all work areas.

- During hazardous weather conditions, such as lightning and thunder storms, work will cease immediately.

12.1 HEAT STRESS

As portions of this job may be conducted during the summer months when ambient temperatures may be high, the following procedures will be followed to recognize and prevent heat stress:

- The SSO will monitor the activities of the workers and the conditions of the work environment.

- The SSO will monitor the workers for initial signs of heat stress, which include dizziness, nausea, inability to concentrate, impaired performance, and loss of coordination.

- The number of rest periods will be increased. The SSO will designate rest areas in shaded areas and when possible in air conditioned environments, such as trailers and cars.

- Potable water will be available and all workers will be encouraged to drink throughout the day.

- Adjustment of the work schedule will be made, when possible, to conduct labor-intensive tasks during the early morning or later afternoon hours.

13.0 EMERGENCY PLAN

Verbal communications may be difficult at times due to personal protective equipment and noise. A universal set of hand signals will then be used. They are as follows:

Hand gripping throat:	Can't breathe.
Grip partner's wrist or place hands around waist:	Leave work area immediately.
Hand on top of head:	Need assistance.
Thumbs up:	OK. I'm all right.
Thumbs down:	No. Negative.

All job-related injuries and illnesses will be reported to the SSO. If medical attention is needed, the injured worker will be decontaminated, if possible, prior to leaving the site. The SSO will investigate the cause of the accident and corrective measures will be taken before the work can resume. It will be the responsibility of the SSO to complete the accident reporting form, OSHA 101, attached to this section for all injuries. The completed OSHA 101 form will be forwarded to the office health and safety manager within 6 days for recording into the OSHA 200 log. In the event of a fatality or five or if more workers are hospitalized as a result of a single incident, the SSO will contact the office health and safety manager immediately for OSHA reporting purposes.

13.1 EMERGENCY TELEPHONE NUMBERS

Police	-	911
Fire	-	911
Syosset Hospital	-	496-6500

13.2 DIRECTIONS TO THE HOSPITAL

From the work areas, on-site and off-site, proceed to South Oyster Bay Road. Proceed north on South Oyster Bay Road to Jericho Turnpike. Proceed west on Jericho Turnpike, approximately 1/2 mile and Syosset Hospital will be on the right-hand side of the road. These directions will be outlined on a local street map with a copy of this map posted at each drilling location.

14.0 REFERENCE

Geraghty & Miller, Inc. 1989. Interim Remedial Investigation Report, Syosset Landfill, Syosset, New York. August 1989.

APPENDIX A

SECTION IV: HEALTH AND SAFETY PLAN

APPENDIX A

SITE VISITORS LOG

SITE VISITORS LOG

THE UNDERSIGNED VISITORS REQUIRE ENTRANCE TO THE EXCLUSION ZONE AND HAVE THOROUGHLY READ THE HEALTH AND SAFETY PLANS, UNDERSTAND THE POTENTIAL HAZARDS AT THE SITE AND THE PROCEDURES TO MINIMIZE EXPOSURE TO THE HAZARDS, WILL FOLLOW THE DIRECTION OF THE SITE HEALTH AND SAFETY MANAGER, AND WILL ABIDE BY THE HEALTH AND SAFETY PLAN.

NAME (print)	COMPANY	DATE	SIGNATURE

3A:SVL.FRM

APPENDIX B

SECTION IV: HEALTH AND SAFETY PLAN

APPENDIX B

TAILGATE SAFETY MEETING FORM

TAILGATE SAFETY MEETING

Prepared by _____

Client _____ Project _____

Date _____ Project Number _____

Work Location _____

Type of Work to be Done _____

SAFETY TOPICS PRESENTED

Chemical Hazards _____

_____

Physical Hazards/Underground Utilities _____

_____

Protective Clothing/Equipment _____

_____

Special Equipment _____

Emergency Procedures _____

_____

Hospital/Clinic _____ Phone ( )_____

Paramedic Phone ( )_____

Hospital Address _____

Other _____

ATTENDEES

NAME PRINTED	SIGNATURE

Meeting Conducted By _____ _____

Name Printed Signature

Note: This tailgate safety form must be completed daily.

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APPENDIX C

SECTION IV: HEALTH AND SAFETY PLAN

APPENDIX C

ACCIDENT REPORTING FORM, OSHA 101

OSHA No. 101
Case or File No. .....

Form approved
OMB No. 44R 1453

Supplementary Record of Occupational Injuries and Illnesses

EMPLOYER

1. Name .....
2. Mail address ..... (No. and street) (City or town) (State)
3. Location, if different from mail address .....

INJURED OR ILL EMPLOYEE

4. Name ..... (First name) (Middle name) (Last name) Social Security No. .....
5. Home address ..... (No. and street) (City or town) (State)
6. Age ..... 7. Sex: Male..... Female..... (Check one)
8. Occupation ..... (Enter regular job title, *not* the specific activity he was performing at time of injury.)
9. Department ..... (Enter name of department or division in which the injured person is regularly employed, even though he may have been temporarily working in another department at the time of injury.)

THE ACCIDENT OR EXPOSURE TO OCCUPATIONAL ILLNESS

10. Place of accident or exposure ..... (No. and street) (City or town) (State)
    If accident or exposure occurred on employer's premises, give address of plant or establishment in which it occurred. Do not indicate department or division within the plant or establishment. If accident occurred outside employer's premises at an identifiable address, give that address. If it occurred on a public highway or at any other place which cannot be identified by number and street, please provide place references locating the place of injury as accurately as possible.
11. Was place of accident or exposure on employer's premises? ..... (Yes or No)
12. What was the employee doing when injured? ..... (Be specific. If he was using tools or equipment or handling material, name them and tell what he was doing with them.)
13. How did the accident occur? ..... (Describe fully the events which resulted in the injury or occupational illness. Tell what happened and how it happened. Name any objects or substances involved and tell how they were involved. Give full details on all factors which led or contributed to the accident. Use separate sheet for additional space.)

OCCUPATIONAL INJURY OR OCCUPATIONAL ILLNESS

14. Describe the injury or illness in detail and indicate the part of body affected. ..... (e.g.: amputation of right index finger at second joint; fracture of ribs; lead poisoning; dermatitis of left hand, etc.)
15. Name the object or substance which directly injured the employee. (For example, the machine or thing he struck against or which struck him; the vapor or poison he inhaled or swallowed; the chemical or radiation which irritated his skin; or in cases of strains, hernias, etc., the thing he was lifting, pulling, etc.)
16. Date of injury or initial diagnosis of occupational illness ..... (Date)
17. Did employee die? ..... (Yes or No)

OTHER

18. Name and address of physician .....
19. If hospitalized, name and address of hospital .....

Date of report ..... Prepared by .....
Official position .....

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APPENDIX D

SECTION IV: HEALTH AND SAFETY PLAN

APPENDIX D

UTILITIES AND STRUCTURES CHECKLIST

GERAGHTY & MILLER, INC.
Ground-Water Consultants

UTILITIES AND STRUCTURES CHECKLIST

Project: _____ **Prepared by:** _____

Location: _____ **Date:** _____

Instructions. This checklist has to be completed by a G&M staff member as a safety measure to insure that all underground utility lines, other underground structures as well as above-ground power lines are clearly marked out in the area selected for boring or excavation. **DRILLING OR EXCAVATION WORK MAY NOT PROCEED UNTIL LINES ARE MARKED AND THIS CHECKLIST HAS BEEN COMPLETED.** Arrangements for underground utility markouts are best made at the time of the preliminary site visit to allow client and/or utility company sufficient time. Keep completed checklist and maps onsite; send copy to Project Manager.

Assignment of Responsibility. Client is responsible for having underground utilities and structures located and marked. Preferably, the utilities themselves should mark out the lines.

Drilling or Excavation Sites. Attach a map of the property showing the proposed drilling or excavation site (or if sites are widely separated, several maps) clearly indicating the area(s) checked for underground utilities or underground structures and the location of above-ground power lines.

Utilities and Structures

Type	Not Present	Present	How Marked?[1)]
Petroleum products line			
Natural gas line			
Steam line			
Water line			
Sewer line			
Storm drain			
Telephone cable			
Electric power line			
Product tank			
Septic tank/drain field			
Overhead power line			

1) Flags, paint on pavement, wooden stakes, etc.

Name and affiliation of person who marked out underground lines or structures.

_____ NAME _____ ORGANIZATION _____ PHONE

Emergency Procedures

Persons at site or facility to contact in case of emergency

1. _____ Phone _____

2. _____ Phone _____

Fire Dept.: Phone _____ Ambulance: Phone _____

Utility: Phone _____ Utility: Phone _____

Utility: Phone _____ Utility: Phone _____

Directions to nearest hospital (describe or attach map).

TABLES

Table 1. Sum the Sampling and Analytical Program for the Second Operable Unit Remedial Investigation, Syosset Lan sset, New York.

Data Collection Activities	Media	Sample Location	Number of Samples/Data Points*	Analyses	Analytical Level**	Analytical Method	Objectives/Rationale
Monitoring Well Installation	Ground Water	See SAP	11	Not applicable	Not applicable	Not applicable	Construction of monitoring wells capable of providing water-level measurements and ground-water samples
Monitoring Well Installation with Ground-Water Sampling	Ground Water	See SAP	See SAP	VOC Screening Field Leachate Parameters	Level I	Foxboro OVA Hach Test Kits Ammonia Meter	Characterize ground-water quality conditions for evaluation of vertical and lateral extent of potential contamination and for remedial alternatives
Monitoring Well Installation with Ground-Water Sampling	Ground Water	See SAP	See SAP	Field Leachate Parameters	Level III	Standard USEPA Methods	Confirm ground-water leachate field testing
Ground-Water Sampling	Ground Water	See SAP	52	TCL Organics/PPL Inorganics Except Pesticides	Level IV	USEPA CLP	Characterize ground-water quality conditions for evaluation of remedial alternatives and engineering design
Ground-Water Sampling	Ground Water	See SAP	52	Leachate Parameters	Level III	Standard USEPA Methods	Characterize ground-water quality conditions for evaluation of remedial alternatives and engineering design
Ground-Water Sampling	Ground Water	See SAP	7	Leachate Parameters	Level III	Standard USEPA Methods	Characterize background ground-water quality conditions
Landfill Gas Measurements	Landfill Gas	See SAP	18	VOC Screening/ Combustible Gas Screening	Level I	Foxboro OVA/ Combustible Gas Indicator	Determine extent of off-site gas migration
Water-Level Measurements	Ground Water	See SAP	See SAP	Water-table and piezometric surface elevations	Not applicable	M-scope/Steel tape and chalk, depth to water measurements	Characterize ground-water flow patterns and identify pathways and migration routes to determine appropriate remedial action
Geophysical Logging	Unconsolidated Sediments	Deep Monitoring Wells	5	Natural Gamma log	Not applicable	EG&G Mount Sopris Model II Logging System; HLP-2375 probe	Characterize lithology of the unconsolidated sediments

TCL Target compound list.
PPL Priority pollutant list.
* Not including QA/QC
** The analytical levels are defined as follows:

Level I - Field screening or analysis using portable instruments. Results are often not compound-specific and not quantitative but results are available in real-time. It is the least costly of the analytical options.

Level II - Field analyses using more sophisticated portable analytical instruments; in some cases, the instruments may be set up in a mobile laboratory on-site. There is a wide range in the quality of data that can be generated, which depends on the use of suitable calibration standards, reference materials, and sample preparation equipment and the training of the operator. Results are available in real-time or several hours.

Level III - All analyses performed in an off-site analytical laboratory. Level III analyses may or may not use CLP procedures, but do not usually utilize the validation or documentation procedures required of Level IV analysis (New York Certified). The laboratory may or may not be a CLP laboratory.

Level IV - Routine analytical services (RAS). All analyses are performed in an off-site (New York Certified) analytical laboratory following NYSDEC ASP protocols. Level IV is characterized by rigorous QA/QC protocols and documentation.

Table 2. S y of Construction Details for On-Site Monitoring Wells Installed a Syosset Landfill, Syosset, New York.

Well Designation	Completion Date	Well Diameter (inches)	Total Depth (feet below land surface)	Screen Setting (feet below land surface)	Interval Gravel Packed (feet below land surface)	Interval Sealed with Bentonite Pellets (feet below land surface)	Interval Sealed with Bentonite Slurry (feet below land surface)	Height of Measuring Point a (relative to land surface)	Elevation of Measuring Point (feet above mean sea level) d	Well Casing & Screen Material
SY-1 *	10/19/82	2	135	125 - 135	35 - 135 b	34 - 35	8 - 34 c	-0.15	194.52	Black Steel
SY-1D	2/2/88	4	218	182 - 192	179 - 218	177 - 179	2 - 177	+2.31	197.36	PVC
SY-2R	2/12/88	4	150	115 - 125	112 - 150	110 - 112	2 - 110	+1.95	187.48	PVC
SY-2D	2/9/88	4	215	190 - 200	187 - 215	185 - 187	2 - 185	+2.18	186.57	PVC
SY-3 *	10/20/82	2	145	135 - 145	47 - 145 b	45 - 47	4 - 45 c	-0.50	191.38	Black Steel
SY-3D	2/25/88	3	240	189 - 199	184 - 240	181 - 184	2 - 181		194.74	PVC
SY-4 *	10/20/82	2	153	143 - 153	57 - 153 b	54 - 57	4 - 54 c	-0.20	193.32	Black Steel
SY-5 *	10/20/82	2.5	135	125 - 135	46 - 135 b	44 - 46	5 - 44 c	+4.20	188.07	Galvanized Steel
SY-6 *	10/19/82	2	145	135 - 145	31 - 145 b	28 - 31	5 - 28 c	-0.10	185.85	Black Steel
SY-6D	3/9/88	4	215	195 - 205	192 - 215	190 - 192	3 - 192	-0.30	185.60	PVC
SY-7 *	10/21/82	2	145	135 - 145	52 - 145 b	49 - 52	5 - 49 c	-0.25	199.63	Black Steel
SY-8	12/19/87	4	142	127 - 137	125 - 142	122 - 125	2 - 122	+2.25	195.84	PVC
SY-9	1/29/88	4	140	110 - 120	107 - 140	105 - 107	2 - 105	-0.70	199.41	PVC
W-3	11/10/87	2	120	105 - 115	102 - 120	100 - 102	2 - 100	+2.63	191.18	PVC
W-4	11/18/87	2	120	104 - 114	102 - 120	100 - 102	2 - 100	+2.56	192.82	PVC

a The measuring point of each well is the top of the well casing.

b Information not available as to whether this interval was gravel-packed or formation was allowed to collapse.

c Information not available as to whether grout or backfill (drill cuttings) was used to fill the annular space in this interval.

d Survey performed to USGS datum.

* Well installed during the ERM-Northeast site investigation.

Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Inorganic Compounds	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
Antimony	–	–	–	–
Arsenic	0.05	0.05	0.05	0.025
Barium	1.0	1.0	1.0	1.0
Beryllium	–	–	–	–
Cadmium	0.01	0.01	0.01	0.01
Chromium	0.05	0.05	–	–
Copper	1.0	1.0	<0.2	1.0
Iron	0.3	0.3 (a)	–	0.3 (a)
Lead	0.05	0.05	0.05	0.025
Mercury	0.002	0.002	0.005	0.002
Nickel	–	–	–	–
Potassium	–	–	–	–
Selenium	0.01	0.01	0.01	0.02
Silver	0.05	0.05	0.05	0.05
Sodium	–	–	<20	–
Thallium	–	–	–	–
Zinc	5.0	5.0	<0.3	5
Cyanide	–	–	<0.1	0.2

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	Applicable or Relevant and Appropriate Requirements.
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

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Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Volatile Organic Compounds	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
Chloromethane	–	0.005	–	–
Bromomethane	–	0.005	–	–
Vinyl Chloride	0.002	0.002	–	0.005
Chloroethane	–	0.005	–	–
Methylene Chloride	–	0.005	–	–
Acetone	–	0.05	–	–
Carbon Disulfide	–	0.05	–	–
1,1-Dichloroethene	0.007	0.005	–	–
1,1-Dichloroethane	–	0.005	–	–
1,2-Dichloroethene	–	0.005	–	–
Chloroform	0.1 (b)	0.1 (b)	–	0.1
1,2-Dichloroethane	0.005	0.005	–	–
2-Butanone	–	0.05	–	–
1,1,1-Trichloroethane	0.2	0.005	–	–
Carbon Tetrachloride	0.005	0.005	–	0.005
Vinyl Acetate	–	0.05	–	–
Bromodichloromethane	0.1 (b)	0.1 (b)	–	–
1,1,2,2-Tetrachloroethane	–	0.005	–	–
1,2-Dichloropropane	–	0.005	–	–
trans-1,2-Dichloropropene	–	0.005	–	–
Trichloroethene	0.005	0.005	–	0.010
Dibromochloromethane	0.1 (b)	0.1 (b)	–	–

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	Applicable or Relevant and Appropriate Requirements.
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

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Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Volatile Organic Compounds (continued)	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
1,1,2-Trichloroethane	–	0.005	–	–
Benzene	0.005	0.005	–	ND
cis-1,3-Dichloropropene	–	0.005	–	–
Bromoform	0.1 (b)	0.1 (b)	–	–
2-Hexanone	–	0.05	–	–
4-Methyl-2-pentanone	–	0.05	–	–
Tetrachloroethene	–	0.005	–	–
Toluene	–	0.005	–	–
Chlorobenzene	–	0.005	–	–
Ethylbenzene	–	0.005	–	–
Styrene(g)	–	0.005	–	0.931
Total Xylenes	–	0.005	–	–

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	Applicable or Relevant and Appropriate Requirements.
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

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Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

PCB Compounds	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
Arochlor-1016	–	0.001	–	0.0001
Arochlor-1221	–	0.001	–	0.0001
Arochlor-1232	–	0.001	–	0.0001
Arochlor-1242	–	0.001	–	0.0001
Arochlor-1248	–	0.001	–	0.0001
Arochlor-1254	–	0.001	–	0.0001
Arochlor-1260	–	0.001	–	0.0001

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	Applicable or Relevant and Appropriate Requirements.
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

301463

Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Semivolatile Organic Compounds	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
Phenol(s)	–	0.05	0.001	0.001 (c)
bis(-2-Chloroethyl)ether	–	0.005	–	0.001
2-Chlorophenol	–	0.005	–	0.001 (c)
1,3-Dichlorobenzene	–	0.005	–	–
1,4-Dichlorobenzene	0.075	0.005	–	4.7 (d)
Benzyl Alcohol	–	0.05	–	–
1,2-Dichlorobenzene	–	0.005	–	4.7 (d)
2-Methylphenol	–	0.05	–	0.001 (c)
bis(2-Chloroisopropyl)ether	–	0.005	–	–
4-Methylphenol	–	0.05	–	0.001 (c)
n-Nitroso-di-propylamine	–	0.05	–	–
Hexachloroethane	–	0.005	–	–
Nitrobenzene	–	0.005	–	–
Isophorone	–	0.05	–	–
2-Nitrophenol	–	0.005	–	0.001 (c)
2,4-Dimethylphenol	–	0.05	–	0.001 (c)
Benzoic Acid	–	0.05	–	–
bis(-2-Chloroethoxy)methane	–	0.005	–	–
2,4-Dichlorophenol	–	0.005	–	0.001 (c)
1,2,4-Trichlorobenzene	–	0.005	–	–
Naphthalene	–	0.05	–	–
4-Chloroaniline	–	0.005	–	–

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	*Applicable or Relevant and Appropriate Requirements.*
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

301464

Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Semivolatile Organic Compounds (continued)	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
Hexachlorobutadiene	–	0.005	–	–
4-Chloro-3-methylphenol	–	0.005	–	0.001 (c)
2-Methylnaphthalene	–	0.05	–	–
Hexachlorocyclopentadiene	–	0.005	–	–
2,4,6-Trichlorophenol	–	0.005	–	0.001 (c)
2,4,5-Trichlorophenol	–	0.005	–	0.001 (c)
2-Chloronaphthalene	–	0.005	–	–
2-Nitroaniline	–	0.005	–	–
Dimethylphthalate	–	0.05	–	–
Acenaphthylene	–	0.05	–	–
3-Nitroaniline	–	0.005	–	–
Acenaphthene	–	0.05	–	–
2,4-Dinitrophenol	–	0.005	–	0.001 (c)
4-Nitrophenol	–	0.005	–	0.001 (c)
Dibenzofuran	–	0.05	–	–
2,4-Dinitrotoluene	–	0.005	–	–
2,6-Dinitrotoluene	–	0.005	–	–
Diethylphthalate	–	0.05	–	–
4-Chlorophenyl-phenylether	–	0.005	–	–
Fluorene	–	0.05	–	–
4-Nitroaniline	–	0.005	–	–
4,6-Dinitro-2-methylphenol	–	0.005	–	0.001 (c)

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	Applicable or Relevant and Appropriate Requirements.
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

301465

Table 3. Potential Ground-Water Applicable or Relevant and Appropriate Requirements Identified for Ground Water, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Semivolatile Organic Compounds (continued)	USEPA MCL	Revised 10NYCRR Subpt 5-1	10NYCRR Part 170	6NYCRR Part 703.6
N-Nitrosodiphenylamine	–	0.005	–	–
4-Bromophenyl-phenylether	–	0.005	–	–
Hexachlorobenzene	–	0.005	–	0.00035
Penta-chlorophenol	–	0.005	–	0.021
Phenanthrene	–	0.05	–	–
Anthracene	–	0.05	–	–
Di-n-butylphthalate	–	0.05	–	0.77
Fluoranthene	–	0.05	–	–
Pyrene	–	0.05	–	–
Butylbenzylphthalate	–	0.05	–	–
3,3'-Dichlorobenzidine	–	0.005	–	–
Benzo(a)anthracene	–	0.05	–	–
bis(2-Ethylhexyl)phthalate	–	0.05	–	4.2
Chrysene	–	0.05	–	–
Di-n-octyl phthalate	–	0.05	–	–
Benzo(b)fluoranthene	–	0.05	–	–
Benzo(k)fluoranthene	–	0.05	–	–
Benzo(a)pyrene	–	0.05	–	ND
Indeno(1,2,3-c d)pyrene	–	0.05	–	–
Dibenz(g,h)anthracene	–	0.05	–	–
Benzo(g,h,i)perylene	–	0.05	–	–

All ARARs are given in Milligrams per Liter unless indicated (mg/L).

All ARARs are given in milligrams per liter unless indicated (mg/L).

ARARs	Applicable or Relevant and Appropriate Requirements.
MCL	Maximum Contaminant Levels.
PCBs	Polychlorinated biphenyls.
ND	Not detected.
–	No standard available.
(a)	Combined concentration of iron and manganese shall not exceed 0.5 mg/L.
(b)	Total trihalomethanes shall not exceed 0.1 mg/L.
(c)	Total phenolic compounds.
(d)	Applies to total of para (i.e., 1,4-) and ortho (i.e., 1,2-) isomes only.
(e)	MCL for styrene will be set after public comment period.
(f)	Flow limit is not applicable to storm-water runoff conditions.
(g)	Sum of concentrations of parameters given above.

Table 4. Summary of Water Sample Preservation and Container Specifications, Second Operable Unit Remedial Investigation, Syosset Landfill, Syosset, New York.

Parameter	Sample Container(s) (a)	Chemical Preservative (b)	Holding Time (c)
Volatile Organic Compounds	(3) 40 ml glass vials.	Hydrochloric acid, pH <2	10 days
Base Neutrals, PCBs	(3) 1 liter glass bottle.	Unpreserved.	Extraction-5 days Analysis-40 days following extraction
Metals	(1) 1 liter plastic bottle.	Nitric acid, pH<2	Mercury-26 days Metals (other than Mercury) 180 days
Cyanide	(1) 1 liter plastic bottle.	Sodium hydroxide, pH>12	12 days
Total Dissolved Solids	(1) 1 liter plastic bottle.	--	7 days from time of collection
Ammonia	(1) 1 liter plastic bottle.	Sulfuric acid, pH <2	28 days from time of collection
Chloride	(1) 1 liter plastic bottle.	--	28 days from time of collection
Total Hardness	(1) 1 liter plastic bottle.	Nitric acid, pH <2	6 months from time of collection
Nitrate	(1) 1 liter plastic bottle.	--	48 hours from the time of collection
Sulfate	(1) 1 liter plastic bottle.	--	28 days from time of collection
Alkalinity	(1) 1 liter plastic bottle.	--	14 days from time of collection

(a) Sample containers will be of demonstrated cleanliness as described in Appendix A of the Qapp (Section III).
(b) Samples will be cooled to approximately 4 C.
(c) Starts from verified time of sample receipt, except where noted.

301467

Table 5. Current Occupational Airborne Contaminants Standards and Guidelines for Selected Volatile Organic Compounds Detected During the First Operable Unit Remedial Investigation of the Syosset Landfill, Syosset, New York.

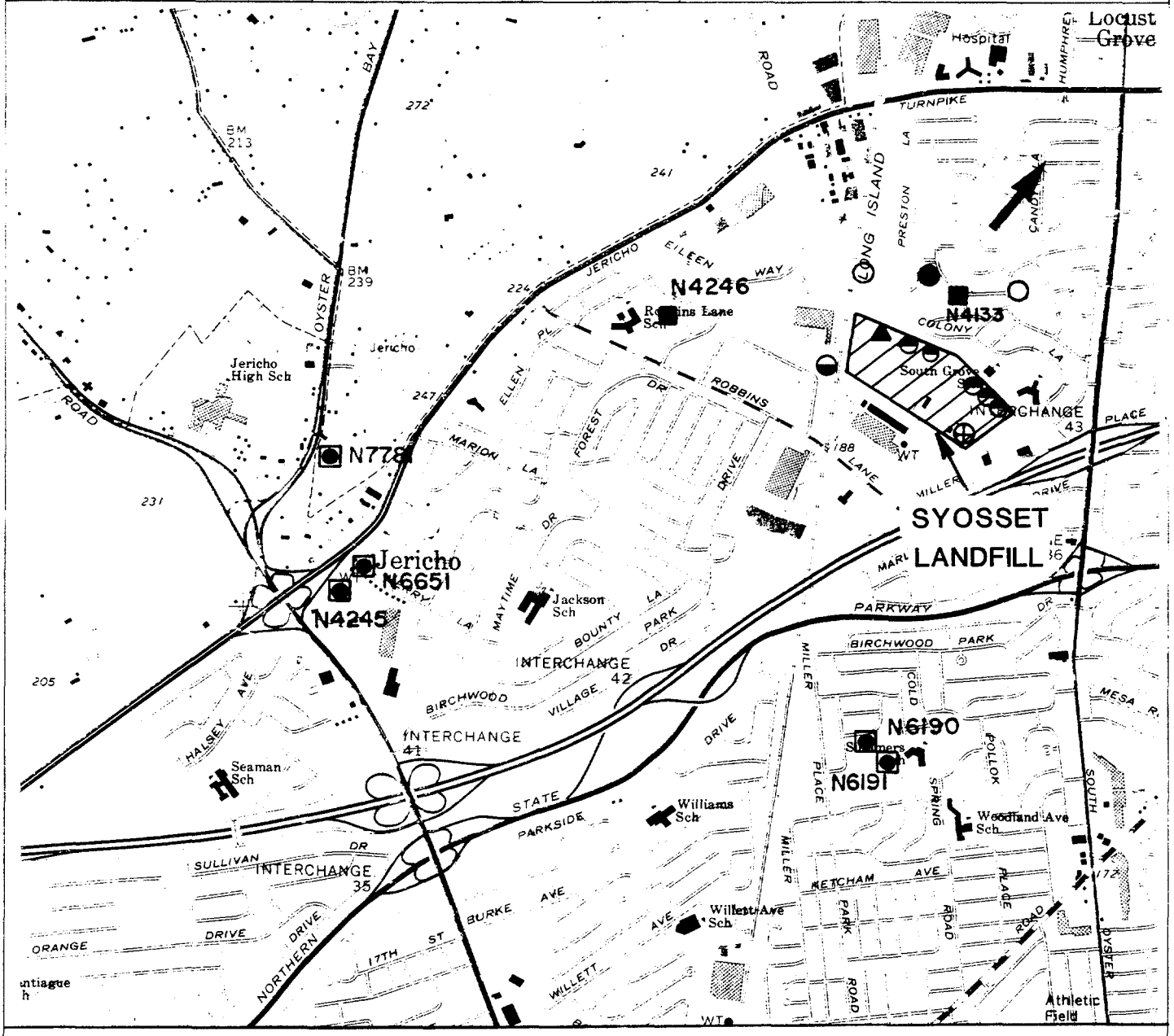
Compound	ACGIH-TLV (ppm) TWA	ACGIH-TLV (ppm) STEL	OSHA-PEL (ppm) TWA	OSHA-PEL (ppm) STEL
Benzene	10		1	
Chloroform	10		2	
Ethylbenzene	100	125	100	125
Tetrachloroethylene	50	200	25	-
Toluene	100	150	100	125
Vinyl chloride	5	-	1	-
Xylenes	100	150	100	150

ACGIH	American Conference of Governmental Industrial Hygienists, 1990-1991.
OSHA	Occupational Safety and Health Administration, 1989.
TLV	Threshold limit value.
PEL	Permissible exposure limit.
TWA	8-hour time-weighted average.
STEL	15-minute short-term exposure limit.
ppm	Parts per million.

FIGURES

GERAGHTY & MILLER, INC.
Ground-Water Consultants

COMPILED BY: V.J. GLASSER	DATE: 2/89	SCALE: SHOWN	PREPARED FOR: LOCKWOOD, KESSLER & BARTLETT Syosset, New York
PREPARED BY: E. DeLUCA	FILE NO: NY0340SL4-1115		
PROJECT MGR.: V.J. GLASSER			



EXPLANATION

- N7781 — LOCATION AND DESIGNATION OF FUNCTIONING PUBLIC SUPPLY WELL
- LOCATION OF PROPOSED OFF-SITE EXPLORATORY BORING/MONITORING WELL CLUSTER
- LOCATION OF PROPOSED ON-SITE EXPLORATORY BORING/MONITORING WELL
- LOCATION OF PROPOSED OFF-SITE CLUSTER BORINGS/MONITORING WELLS
- N4133 — LOCATION AND DESIGNATION OF ABANDONED PUBLIC SUPPLY WELL
- LOCATION OF THE THREE PROPOSED OFF-SITE GAS MONITORING WELLS
- LOCATION OF PROPOSED ON-SITE (OU-1 RD)* GAS MONITORING WELLS
- LOCATION OF PROPOSED DEEP ON-SITE UPGRADIENT MONITORING WELL
- GROUND-WATER FLOW DIRECTION

NORTH

* RD= Remedial Design Program

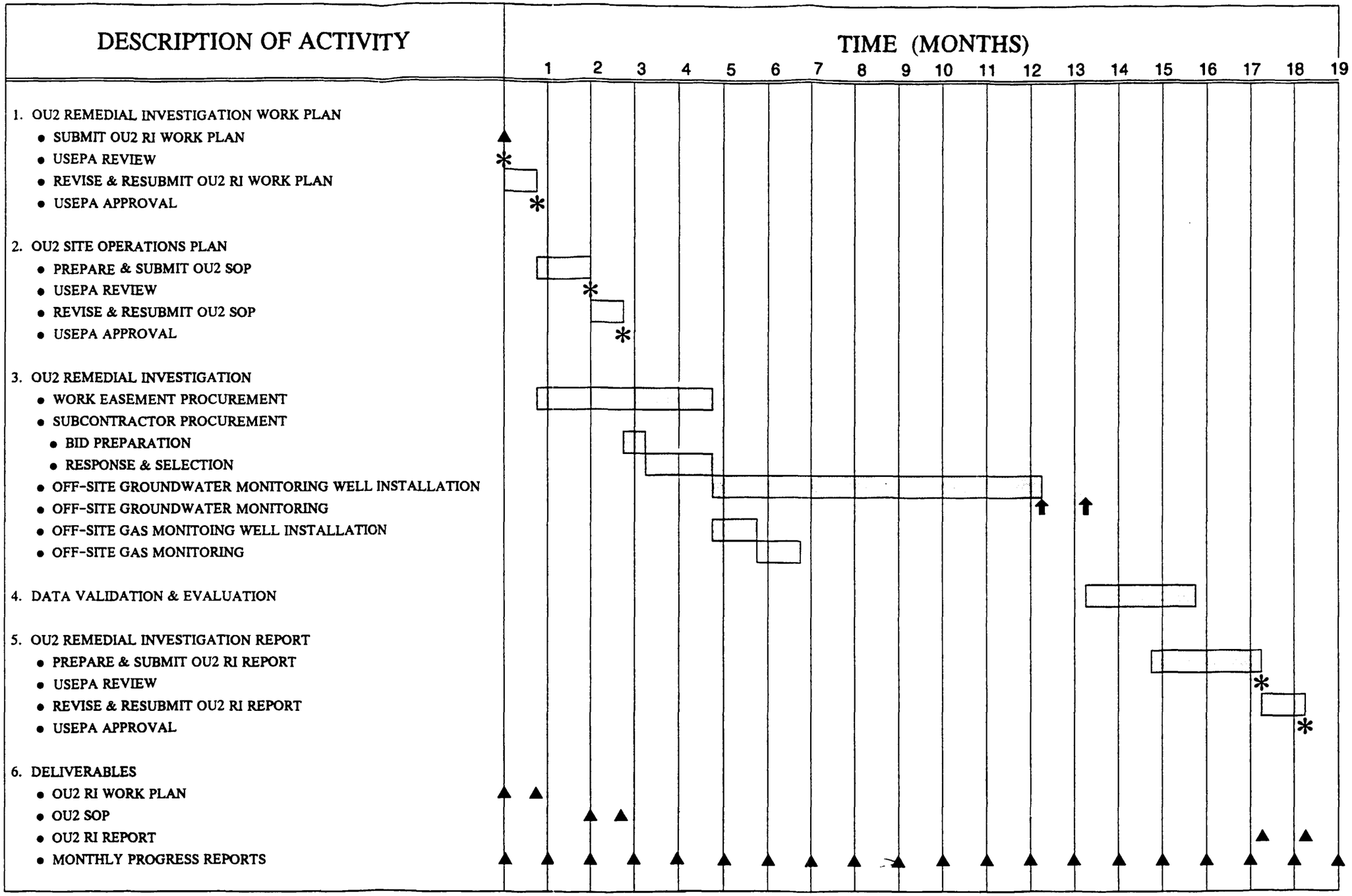
SUBJECT: LOCATIONS OF PROPOSED GROUND-WATER AND GAS MONITORING WELLS, Syosset Landfill, Syosset, New York

FIGURE 1

141991

301469A

SYOSSET LANDFILL OU2 REMEDIAL INVESTIGATION



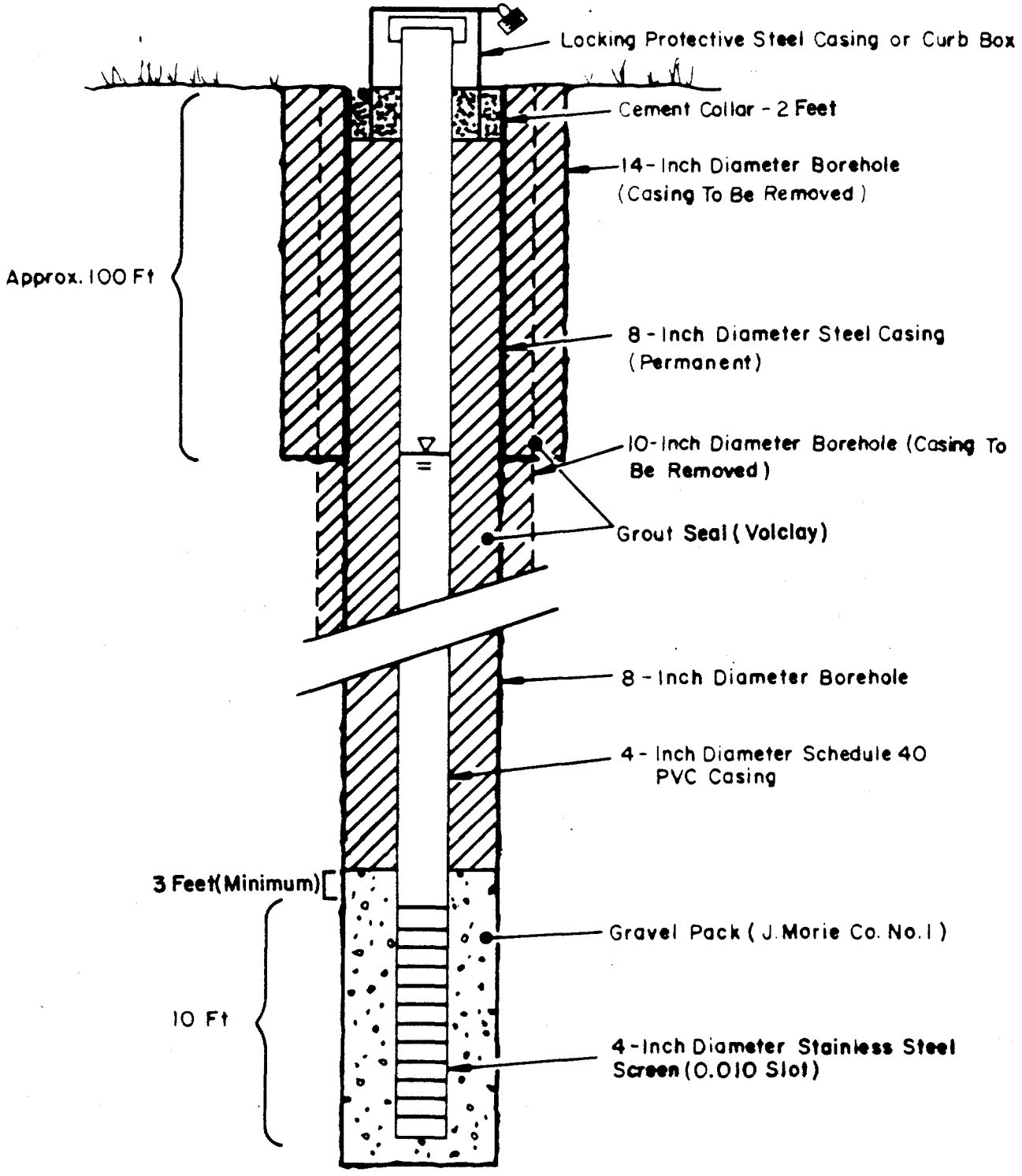
301470

FIGURE 2

LKB

OU2 RI PROJECT SCHEDULE

GERAGHTY & MILLER, INC. Ground-Water Consultants	COMPILED BY: V.J. GLASSER	DATE: 2/89	SCALE: NONE	PREPARED FOR: LOCKWOOD, KESSLER & BARTLETT
	PREPARED BY: E. DeLUCA	FILE NO: NY02908-1115		Syosset, New York
	PROJECT MGR.: V.J. GLASSER			



301471

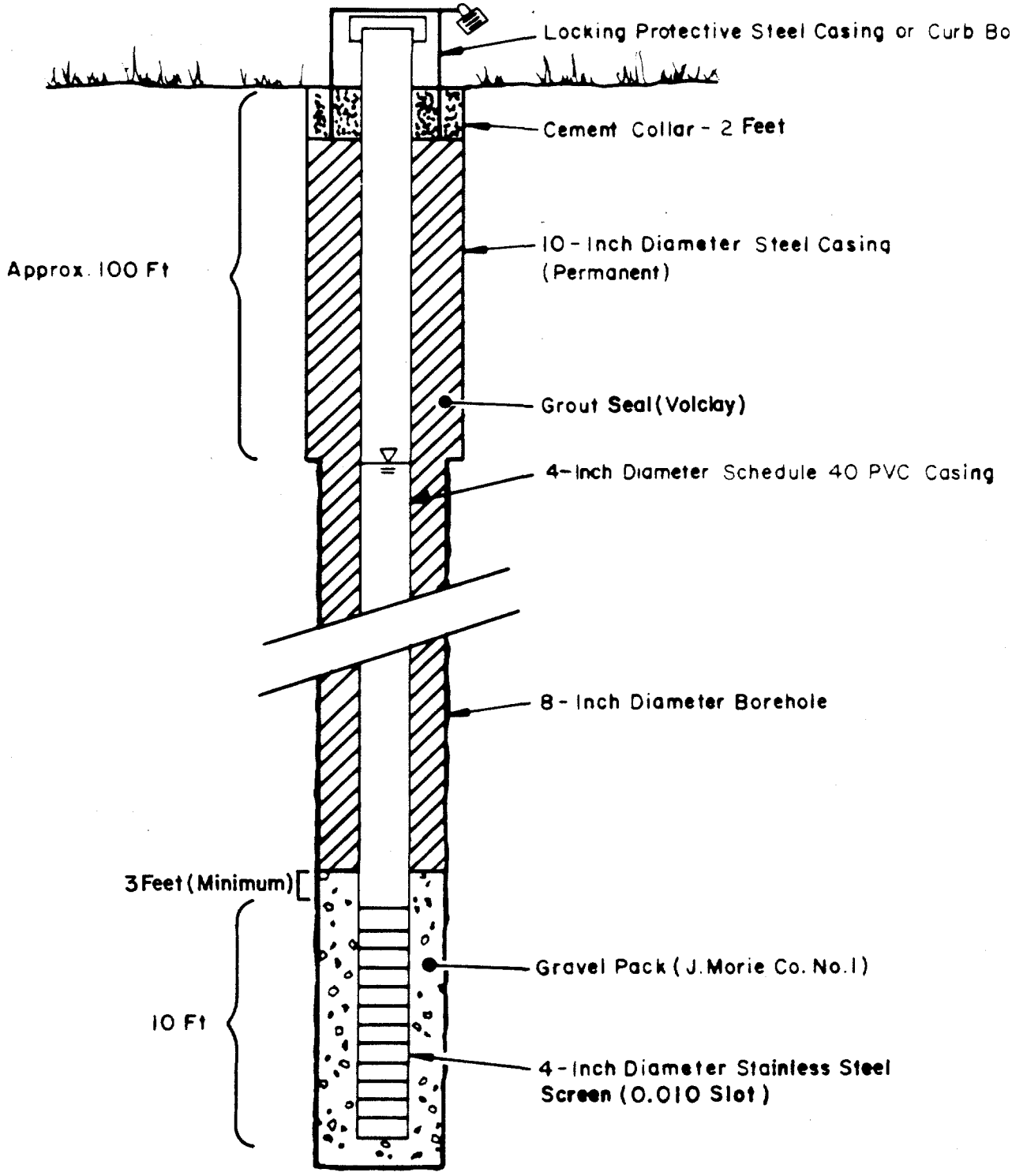
SUBJECT:

GENERALIZED CONSTRUCTION OF MONITORING WELL INSTALLED IN EXPLORATORY BORING, Syosset Landfill, Syosset, New York

FIGURE 3

141991

GERAGHTY & MILLER, INC. Ground-Water Consultants	COMPILED BY: V.J. GLASSER	DATE: 2/89	SCALE: NONE	PREPARED FOR: LOCKWOOD, KESSLER & BARTLETT Syosset, New York
	PREPARED BY: E. DeLUCA	FILE NO: NY02908-1115		
	PROJECT MGR.: V.J. GLASSER			



301472

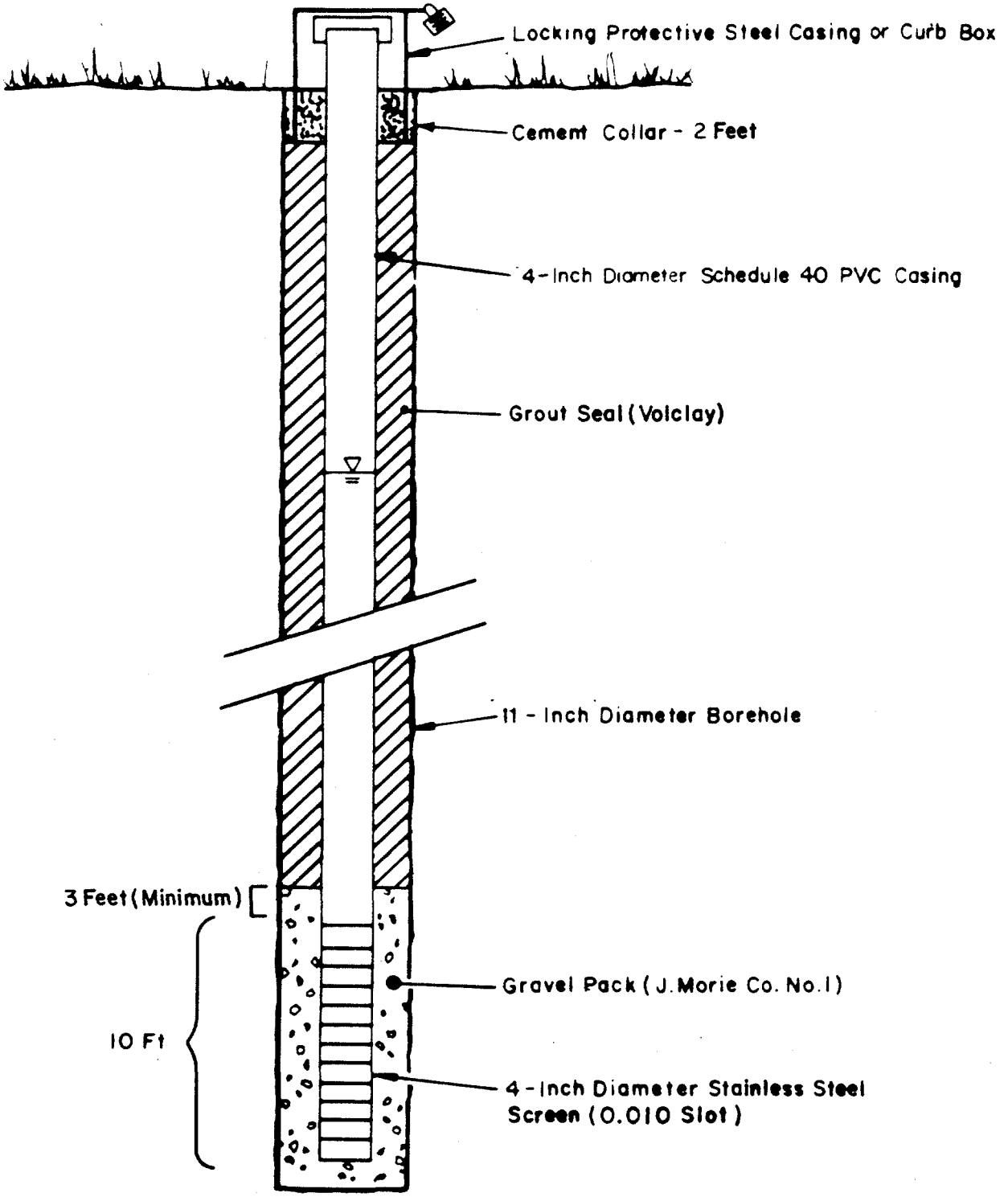
SUBJECT:

GENERALIZED CONSTRUCTION OF MONITORING WELL INSTALLED IN MUD ROTORY BORING, Syosset Landfill, Syosset, New York

FIGURE **4**

141991

GERAGHTY & MILLER, INC. Ground-Water Consultants	DRAWN BY V.J. GLASSER	DWG. NO. 8-91	SCALE NONE	PREPARED FOR LOCKWOOD, KESSLER & BARTLETT Syosset, New York
	CHECKED BY E. DeLUCA			
	APPROVED V.J. GLASSER	FILE NO. NY02908-1115		



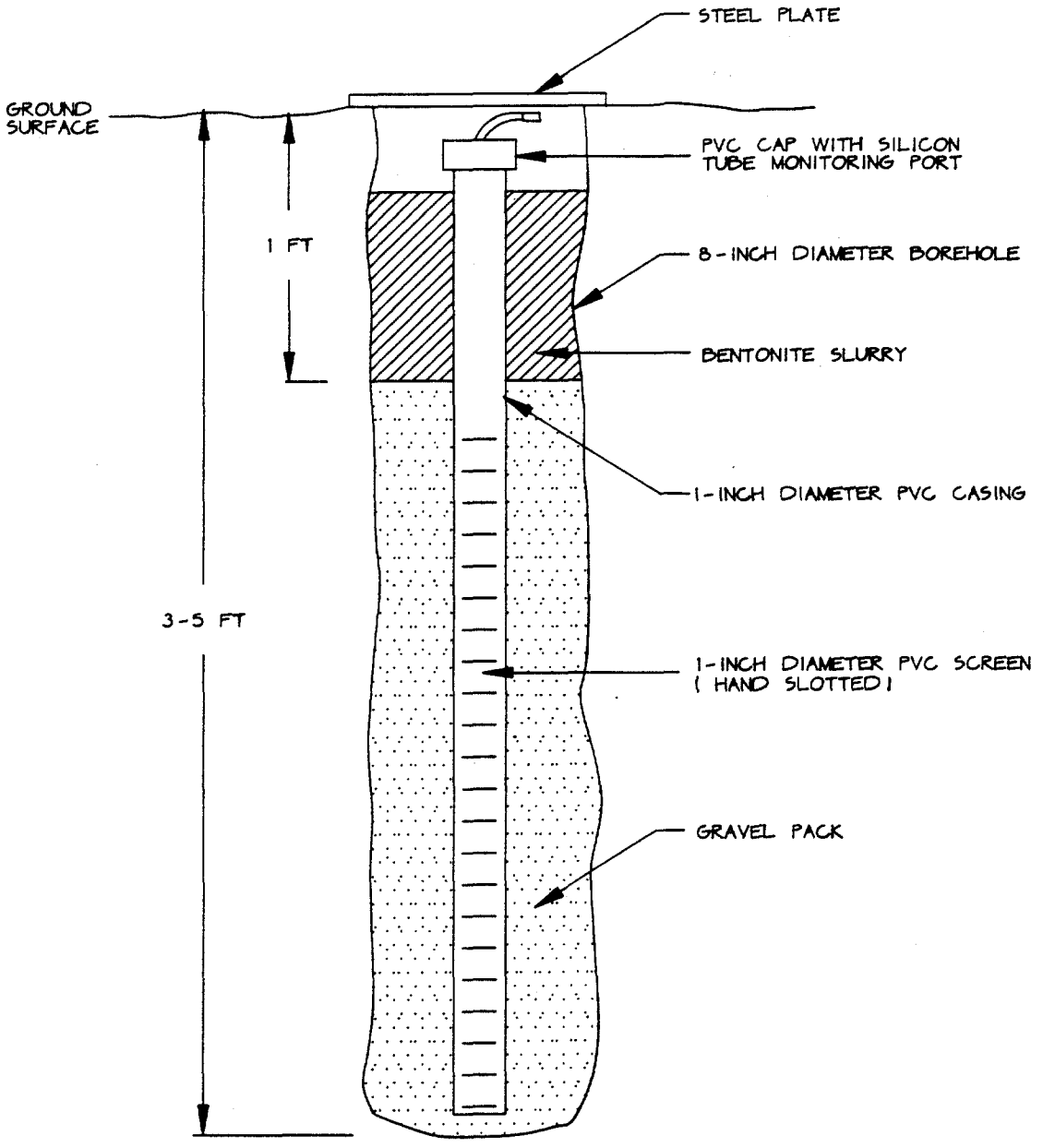
301473

SUBJECT: GENERALIZED CONSTRUCTION OF MONITORING WELL INSTALLED IN HOLLOW STEM AUGER BORING, Syosset Landfill, Syosset, New York

FIGURE 5

14188

DRAFTER: V. CARUNCHO | APPROVED: V. GLASSER | CHECKED: V. GLASSER | CAD FILE; GAS-WELL | FILE NO.: 1115 | PRJCT. NO.: NY02902 | DWG. DATE: 10-30-90



301474

SCALE
NOT TO SCALE

GERAGHTY & MILLER, INC.
Environmental Services

GENERALIZED CONSTRUCTION OF GAS MONITORING WELL

SYOSSET LANDFILL
SYOSSET, NEW YORK

FIGURE
6