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Quality Assurance Project Plan for the Preliminary Site Investigation for McMurdo Station, Ross Island, Antarctica

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Quality Assurance Project Plan for the Preliminary Site Investigation for McMurdo Station, Ross Island, Antarctica

by S.S. Prasad

Environmental Assessment and Information Sciences Division,
Argonne National Laboratory, 9700 South Cass Avenue, Argonne, Illinois 60439

May 1991

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Preface

The *Quality Assurance Project Plan for the Preliminary Site Investigation for McMurdo Station, Ross Island, Antarctica*, was prepared in December 1990 as a preliminary draft. The Argonne National Laboratory (ANL) preliminary site investigation team used the plan to guide field investigations implemented at McMurdo Station during the Antarctic austral summer in January 1991. This final draft of the plan reflects editorial changes made to the original preliminary draft document. The basic substance and informational content of the preliminary draft of the plan remain unchanged in this version.

This document was prepared by ANL under the direction of Douglas W. Canete, Environmental Assessment and Information Sciences Division (EID), ANL-Washington, D.C., who served as the program manager, and James E. Stefano, Environmental Research (ER) Division, ANL-Illinois, who acted as project manager for the preliminary site investigation. Surya S. Prasad, EID, ANL-Washington, D.C., was the principal author of the document. Peter Lindahl, Chemical Technology Division, ANL-Illinois, and Kurt Picel, ER, ANL-Illinois, provided review comments and input.

This document is designed to support research conducted on behalf of the U.S. National Science Foundation (NSF), Division of Polar Programs, Polar Operations Office, Safety, Environmental, and Health Implementation Team. Technical and management oversight of the ANL research program for NSF was provided by Thomas Forhan and I. Sam Higuchi.

Notation

The following is a list of the acronyms, abbreviations, and initialisms (including units of measure) used in this document. Acronyms used in tables only are defined in the respective tables.

Acronyms, Abbreviations, and Initialisms

ACL	Analytical Chemistry Laboratory
ANL	Argonne National Laboratory
ASTM	American Society for Testing and Materials
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
DOT	U.S. Department of Transportation
DSIR	Department of Scientific and Industrial Research (New Zealand)
EPA	U.S. Environmental Protection Agency
GC	gas chromatograph
GC/MS	gas chromatography/mass spectrometry
MAF	Ministry of Agriculture and Fisheries (New Zealand)
MDL	method detection limit
NSF	National Science Foundation
PCBs	polychlorinated biphenyls
PID	photoionization detector
PjRDL	project-required detection limit
PjRQL	project-required quantitation limit
QAPjP	quality assurance project plan
QA/QC	quality assurance/quality control
TCL	target compound list
TIC	tentatively identified compound

USDA	U.S. Department of Agriculture
VOCs	volatile organic compounds

Units of Measure

°C	degrees Celsius
d	day(s)
°F	degrees Fahrenheit
g	gram(s)
kg	kilogram(s)
L	liter(s)
µg	microgram(s)
mL	milliliter(s)
mo	month(s)
mrem	millirem(s)
ng	nanogram(s)
oz	ounce(s)
pCi	picocurie(s)
yr	year

Quality Assurance Project Plan for the Preliminary Site Investigation for McMurdo Station, Ross Island, Antarctica

by

S.S. Prasad

1 Purpose and Project Description

The quality assurance project plan (QAPjP) is designed to ensure that sampling and analysis activities are scoped and performed to obtain quality data during the preliminary site investigation for McMurdo Station, Ross Island, Antarctica. The QAPjP is prepared in accordance with the guidelines set forth and adopted by the U.S. Environmental Protection Agency (EPA) (1980a, 1986a, 1989a), Argonne National Laboratory (ANL) (1988), and Pentecost and Doctor (1990). This document presents the final QAPjP for the preliminary site investigation. A draft version of this report was presented to the National Science Foundation (NSF) in January 1991.

A description of the project and data quality objectives is provided in Section 3.1 of the work plan (Stefano et al. 1991). Specific health and safety precautions and procedures are presented in the health and safety plan (Wozny 1991).

2 Organization and Responsibilities

2.1 Organization

The ANL organizational structure for the preliminary site investigation for McMurdo Station is shown in Figure 2.1. As the principal consultant for NSF, ANL will implement the work plan and maintain overall control of the work plan in accordance with the conditions set forth by NSF. All agreements and modifications regarding the work plan will be made by and between NSF and ANL. In addition, ANL will perform all contract services required to implement the work plan, including field operations, laboratory testing, data management, and office analysis and reporting.

2.2 Responsibilities

2.2.1 Argonne National Laboratory

The program manager and project manager together will form the ANL program management team. Douglas Canete will be the ANL program manager. He will be authorized to commit ANL's resources to accomplish the project objectives and will represent ANL in all contractual matters with NSF. He ultimately will be responsible for all contractual matters and for ANL and subcontractor performance.

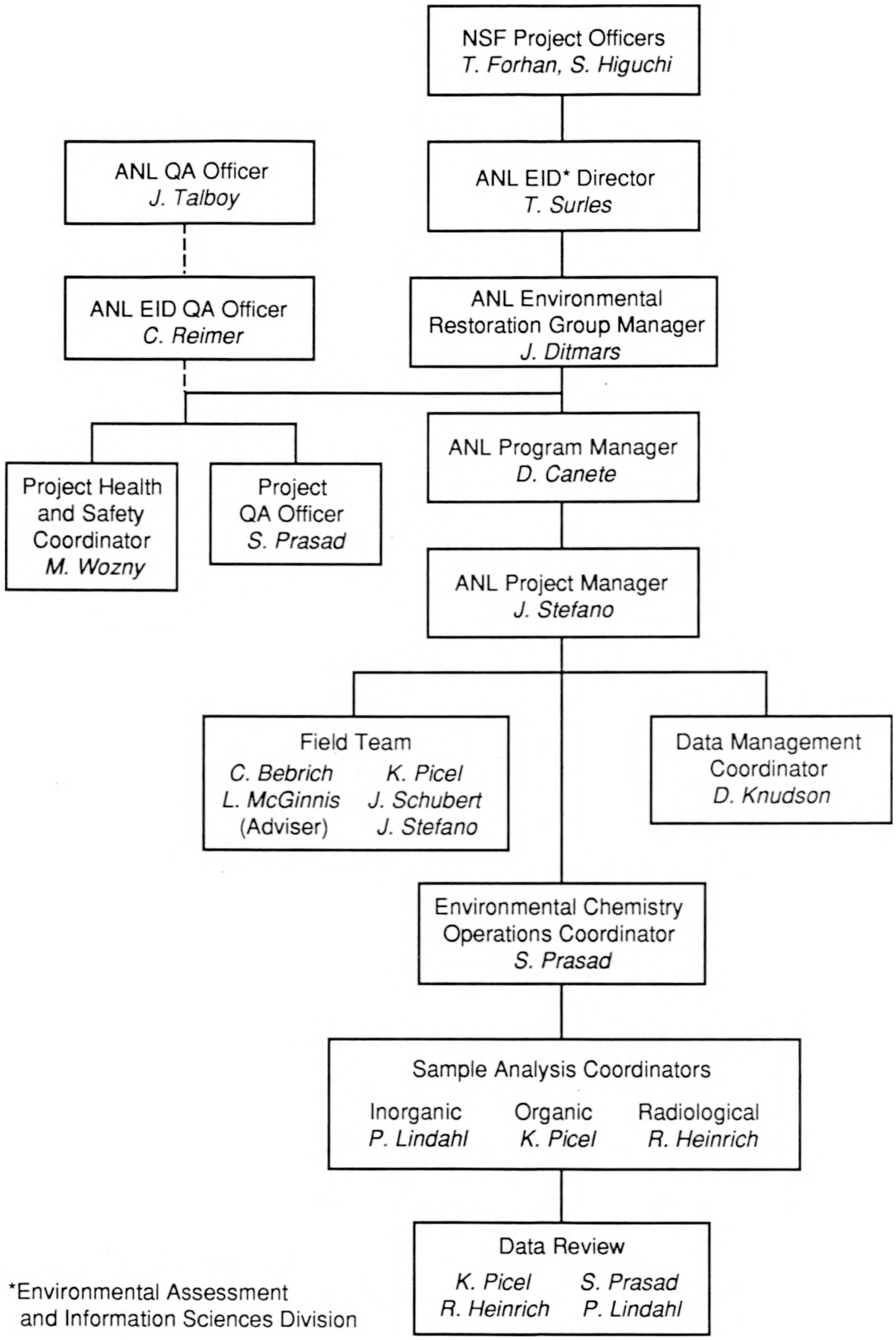
The ANL project manager, James Stefano, will report directly to Canete. He will be responsible for the day-to-day direction and management of ANL's field, laboratory, and office activities and will oversee the activities of ANL's subcontractors. Stefano will be authorized to procure necessary support services and equipment on behalf of NSF, subject to NSF approval, for implementing the work plan. He also will be responsible for staffing, scheduling, and reporting all ANL activities.

The ANL project QA officer, Surya Prasad, will be responsible for all aspects of quality assurance and quality control (QA/QC) related to the work plan. He will serve as liaison between the ANL project manager and the QA officers of all subcontractors and contract laboratories. He will report directly to the ANL program manager or NSF when corrective action is required as a result of system and performance audits.

Qualifications and experience profiles for the ANL project team are given in Appendix A.

2.2.2 Contract Laboratories

An off-site environmental laboratory, hereafter called the "contract laboratory," will provide analytical services for the project. Hazleton Laboratories, Madison, Wisconsin, will contract



*Environmental Assessment and Information Sciences Division

FIGURE 2.1 ANL Organizational Chart for the Preliminary Site Investigation for McMurdo Station, Ross Island, Antarctica directly with ANL to provide chemical testing services for environmental samples collected during field investigations. Hazleton Laboratories is expected to participate in the EPA Contract Laboratory Program (CLP) and is also expected to be permitted by the U.S. Department of Agriculture, Animal and Plant Health Inspection Service, to receive soil, sediment, and water samples from McMurdo Station.

The laboratories of the Chemistry Division, Department of Scientific and Industrial Research (DSIR), New Zealand, will analyze a small number of split field samples for limited parameters. ANL will review data from DSIR to assess the laboratory's capabilities for future work and to examine and compare the effects of different sample holding times because of shipping requirements and logistics. The ANL Analytical Chemistry Laboratory (ACL) will provide radiological analysis for environmental samples collected and analytical support as needed for testing other chemical parameters.

Qualifications of and QA information for contract laboratories will be available in project files. Laboratory service representatives will coordinate sampling and chemical testing activities.

3 QA Objectives for Measurement Data

The findings of the preliminary site investigation will allow decisions to be made concerning the control and management of hazardous wastes and the need for remedial action at McMurdo Station. Management decisions will largely be based on the results of site-specific activities and quality analytical data generated in the contract laboratories. These data will be scientifically defensible according to required levels of precision and accuracy and will be aided by a comprehensive and well-documented QAPjP, as outlined in this section and supported by the work plan (Stefano et al. 1991).

The field team will use a three-level approach to characterize contamination found at the site. On level I, field data will be obtained through photoionization detector (PID) analysis. Field laboratory (level II) and contract laboratory (level III) analyses will provide additional information, including gas chromatograph (GC) analysis on limited field samples. The analytical levels are described in detail in the work plan (Stefano et al. 1991).

Quality assurance objectives for accuracy and precision, comparability, method detection limit (MDL), completeness, and representativeness have been established for the analytical parameters to be determined in the project (EPA 1981, 1989b). Key data quality indicators for laboratory measurements are defined below; specific qualitative and quantitative methods used to assess these indicators are described in Section 12.

3.1 Accuracy and Precision

Accuracy means the nearness of a result, or the mean of a set of results, to the true value. Accuracy is determined by checking the calibration of samples, matrix spike samples, and reference or laboratory control samples. The objective for accuracy is to equal or exceed the accuracy demonstrated during analysis of similar samples using the same methods. Accuracy must be within the established control limits for the methods used (EPA 1986b).

Precision is a measure of mutual agreement among individual measurements of the same sample, usually under similar prescribed conditions. Precision is assessed by analyzing duplicate samples in the laboratory. The objective for precision is to equal or exceed the precision demonstrated during analysis of similar samples. Precision must be within the established control limits for the methods used (EPA 1986b).

For organic constituents, accuracy and comparability of results will be based on EPA (1988a) method 624 for volatile organic compounds (VOCs), method 625 for semivolatiles, and either method 608 or EPA (1986b) method 8080 for polychlorinated biphenyls (PCBs). Samples will be analyzed within the times specified by the methods. The contract laboratories will follow standard operating procedures as long as they can demonstrate that their methods are equal to the EPA methods.

For VOCs and semivolatiles, method blanks, matrix spikes, and matrix spike duplicates (EPA 1988a) will be analyzed to ensure accuracy and precision. Method blanks will be prepared from reagent water spiked with a surrogate recovery standard and then analyzed according to established procedures. Method blanks will be run once for every set of samples from a given location, every 20 samples or every 72 hours, whichever is more frequent.

For samples that may contain VOCs, semivolatiles, or PCBs, a surrogate spiking solution specific to each fraction will be added before the sample is prepared for analysis. Surrogate compounds will be analyzed concurrently with sample analytes, and recoveries of surrogate compounds will be calculated for use in evaluating the effectiveness and integrity of sample extraction and preparation for each fraction analyzed. Comparing surrogate recovery values for similar sets of samples also measures the precision of the method.

To evaluate matrix effects on analyte recovery, periodic matrix spike samples will be analyzed for each organic fraction. Matrix spikes and matrix spike duplicates will be prepared from an aliquot or homogeneous portion, to the extent possible, of a corresponding sample. A spiking standard solution containing a specified subset of the target compound list (TCL) will be added to the sample prior to extraction. The procedures used to analyze the original set of samples will also be used to analyze the spiked samples. Matrix effects on analyte recovery will be evaluated by recovering the matrix spike compounds. The precision of the method will be further evaluated by comparing the matrix spike results with the duplicate matrix spike results.

3.2 Completeness

Completeness of data is a measure of the amount of valid data obtained from the laboratory versus the amount expected to be obtained for the number of samples collected. The objective for completeness of this project is 90%.

3.3 Method Detection Limit

The MDL is the minimum concentration of an analyte that can be measured and reported with 99% confidence. The MDLs will be determined by replicate measurements of samples containing the analytes listed in Tables 3.1 through 3.3 (EPA 1988a, 1989b). The MDL for this project must be less than or equal to the limits set forth in the tables. Project-required detection limits (PjRDLs) for inorganic target analytes are listed in Table 3.1. Project-required quantitation limits (PjRQLs) for organic target compounds are listed in Tables 3.2 and 3.3. Radiological parameters, including gross alpha, gross beta, and gamma activity, are presented in EPA (1976, 1979, 1980b) and in the *Federal Register* (1984).

Such techniques as flame atomic absorption, graphite atomic absorption, and cold vapor atomic absorption will be used to analyze inorganic samples for the presence of inorganic target analytes listed in Table 3.1. Analyses for the TCL organics listed in Tables 3.2 and 3.3 will

TABLE 3.1 Inorganic TAL for Laboratory Analysis

Analyte	PjRDL[a] (µg/L)	Analyte	PjRDL[a] (µg/L)
Aluminum	200	Nickel	40
Antimony	60	Potassium	5,000
Arsenic	10	Selenium	5
Barium	200	Silver	10
Beryllium	5	Sodium	5,000
Cadmium	5	Thallium	10
Calcium	5,000	Vanadium	50
Chromium	10	Zinc	20
Cobalt	50	Cyanide	10
Copper	25		
Iron	100		
Lead	3		
Magnesium	5,000		
Manganese	15		
Mercury	0.2		

[a] The project-required detection limits are the instrument detection limits obtained in pure water that must be met for the project. The detection limits for samples may be considerably higher, depending on the sample matrix.

Source: EPA 1989b.

be conducted by EPA methods 624, 625, and 608 (for PCBs only) for the required analytes and PjRQLs, which correspond to contract-required quantitation limits, respectively (EPA 1988a).

3.4 Comparability

Comparability is the degree to which one data set can be compared with another data set. For this project, data from samples analyzed for similar parameters by different laboratories will be compared. The use of standardized methods, traceable standards, and standardized reporting units and format will aid in making the comparisons.

3.5 Representativeness

Representativeness is a quality characteristic attributable to the type and number of samples taken. Samples must be representative of the sample population. Sampling strategy for field operations and screening methods used to determine possible sources of contamination are presented in Sections 3 and 3.6 of the work plan (Stefano et al. 1991), respectively. Sampling

TABLE 3.2 TCL for Volatile and Semivolatile Organic Compounds and Project-Required Quantitation Limits for Laboratory Analysis[a]

Organic Compound	Quantitation Limits[b] Water (µg/L)	Low Soil and Sediment[c] (µg/kg)
Volatiles		
Chloromethane	10	10
Bromomethane	10	10
Vinyl chloride	10	10
Chloroethane	10	10
Methylene chloride	5	5
Acetone	10	10
Carbon disulfide	5	5
1,1-Dichloroethene	5	5
1,1-Dichloroethane	5	5
1,2-Dichloroethene (total)	5	5
Chloroform	5	5
1,2-Dichloroethane	5	5
2-Butanone	10	10
1,1,1-Trichloroethane	5	5
Carbon tetrachloride	5	5
Vinyl acetate	10	10
Bromodichloromethane	5	5
1,2-Dichloropropane	5	5
cis-1,3-Dichloropropene	5	5
Trichloroethene	5	5
Dibromochloromethane	5	5
1,1,2-Trichloroethane	5	5
Benzene	5	5
trans-1,2-Dichloropropene	5	5
Bromoform	5	5
4-Methyl-2-pentanone	10	10
2-Hexanone	10	10
Tetrachloroethene	5	5
Toluene	5	5
1,1,2,2-Tetrachloroethane	5	5
Chlorobenzene	5	5
Ethyl benzene	5	5
Styrene	5	5
Xylenes (total)	5	5
Semivolatiles		
Phenol	10	330
bis(2-Chloroethyl) ether	10	330
2-Chlorophenol	10	330
1,3-Dichlorobenzene	10	330
1,4-Dichlorobenzene	10	330

TABLE 3.2 (Cont'd)

Organic Compound	Quantitation Limits[b] Water (µg/L)	Low Soil and Sediment[c] (µg/kg)
Benzyl alcohol	10	330
1,2-Dichlorobenzene	10	330
2-Methylphenol	10	330
bis(2-Chloroisopropyl) ether	10	330
4-Methylphenol	10	330
N-Nitroso-di-n-dipropylamine	10	330
Hexachloroethane	10	330
Nitrobenzene	10	330
Isophorone	10	330
2-Nitrophenol	10	330
2,4-Dimethylphenol	10	330
Benzoic acid	50	1,600
bis(2-Chloroethoxy) methane	10	330
2,4-Dichlorophenol	10	330
1,2,4-Trichlorobenzene	10	330
Naphthalene	10	330
4-Chloroaniline	10	330
Hexachlorobutadiene	10	330
4-Chloro-3-methylphenol (para-chloro-meta-cresol)	10	330
2-Methylnaphthalene	10	330
Hexachlorocyclopentadiene	10	330
2,4,6-Trichlorophenol	10	330
2,4,5-Trichlorophenol	50	1,600
2-Chloronaphthalene	10	330
2-Nitroaniline	50	1,600
Dimethylphthalate	10	330
Acenaphthalene	10	330
2,6-Dinotrotoluene	10	330
3-Nitroaniline	50	1,600
Acenaphthene	10	330
2,4-Dinitrophenol	50	1,600
4-Nitrophenol	50	1,600
Dibenzofuran	10	330
2,4-Dinotrotoluene	10	330
Diethylphthalate	10	330
4-Chlorophenyl-phenyl ether	10	330
Fluorene	10	330
4-Nitroaniline	50	1,600
4,6-Dinitro-2-methylphenol	50	1,600
N-nitrosodiphenylamine	10	330
4-Bromophenyl-phenylether	10	330
Hexachlorobenzene	10	330
Pentachlorophenol	50	1,600
Phenanthrene	10	330
Anthracene	10	330
Di-n-butylphthalate	10	330

TABLE 3.2 (Cont'd)

Organic Compound	Quantitation Limits[b] Water (µg/L)	Low Soil and Sediment[c] (µg/kg)
Fluoranthene	10	330
Pyrene	10	330
Butylbenzylphthalate	10	330
3,3'-Dichlorobenzidine	20	660
Benzo(a)anthracene	10	330
Chrysene	10	330
bis(2-Ethylhexyl)phthalate	10	330
Bi-n-octylphthalate	10	330
Benzo(b)fluoranthene	10	330
Benzo(k)fluoranthene	10	330
Benzo(a)pyrene	10	330
Indeno(1,2,3-dc)pyrene	10	330
Dibenz(a,h)anthracene	10	330
Benzo(g,h,i)perylene	10	330

[a] Specific quantitation limits are highly matrix dependent. These quantitation limits are provided for guidance and may not always be achievable.

[b] Quantitation limits listed for soil and sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil and sediment will be calculated on the basis of dry weight, as required by the project, and these limits will be higher.

[c] Medium soil and sediment project-required quantitation limits for volatile TCL compounds are 125 times the individual low soil and sediment PjRQLs.

Source: EPA 1988a.

devices will be cleaned between sampling points during analysis to ensure that the sample does not become contaminated. To determine whether the sampling equipment is completely free from contamination, a rinsate (equipment blank) of deionized, analyte-free water will be collected and composited. Each rinsate will be analyzed by EPA method 624, 625, or 608. These methods provide excellent indications of contamination resulting from improper equipment decontamination.

Trip blanks will also be shipped to the contract laboratory for VOC analysis. Trip blanks are vials filled with deionized water (American Society for Testing and Materials [ASTM] Type II organic-free water). These blanks will be shipped with VOC samples and will be used to screen for VOC contamination that might have occurred during transport. Trip blanks will be analyzed for VOCs only.

In addition, one field blank will be collected from every 20 blanks, that is, from 5% of the samples collected. Field blanks will consist of deionized water (ASTM Type II) transferred to I-Chem brand sample containers in the field; they will be handled in the same way as regular field samples. Field blanks will be used to screen for accidental contamination that might have occurred during field handling and transport of the samples.

Data QA objectives for inorganic parameters, organic parameters, and radiological parameters are summarized in Tables 3.4, 3.5, and 3.6, respectively.

TABLE 3.3 TCL for Polychlorinated Biphenyls and Project-Required Quantitation Limits for Laboratory Analysis[a]

	Quantitation Limits[b]	
PCBs	Water (μg/L)	Low Soil and Sediment[c] (μg/kg)
Aroclor-		
1016	0.5	80.0
1221	0.5	80.0
1232	0.5	80.0
1242	0.5	80.0
1248	0.5	80.0
1254	1.0	160.0
1260	1.0	160.0

[a] Specific quantitation limits are highly matrix dependent. These quantitation limits are provided for guidance and may not always be achievable.

[b] Quantitation limits listed for soil and sediment are based on wet weight. The quantitation limits calculated by the laboratory for soil and sediment will be calculated on the basis of dry weight, as required by the project, and these limits will be higher.

[c] Medium soil and sediment project-required quantitation limits for volatile TCL compounds are 125 times the individual low soil and sediment PjRQLs.

Source: EPA 1988a.

TABLE 3.4 QA Objectives for Measurement of Inorganic Parameters

Inorganic Parameter	Method[a]	Test Medium	Precision Standard Deviation (%)	Accuracy (%)	Completeness (%)
Metals	ICP	Soils, sediment, sludge	±20	±25	90
	AA	Water	±20	±25	90
	FAA	Water	±20	±25	90
Mercury	CVAA	Water	±20	±25	90
Cyanide	Spectro-photometry	Water	±20	±25	90

[a] Methods are defined in Tables 4.1 and 4.2.

Sources: EPA 1986b, 1989b.

TABLE 3.5 QA Objectives for Measurement of Organic Parameters

Organic Parameter	Testing Environment	Precision Standard Deviation (%)	Accuracy (%)	Completeness (%)
VOC	Laboratory	20	25	90
Semivolatiles	Laboratory	20	25	90
PCB	Laboratory	20	25	90
PID screen	Field laboratory	100	100	70
Field GC	Field laboratory	50	50	80

Sources: EPA 1988a for VOCs and semivolatiles; EPA 1986b and 1988a for PCBs; and EPA 1988b for PID screen (FM8) and field gas chromatography (FM18-22).

TABLE 3.6 QA Objectives for Measurement of Radiological Parameters

Radiological Parameter	Testing Environment	Precision Standard Deviation (%)	Accuracy (%)	Completeness (%)
Gross alpha	Laboratory	25-50	50-100	90
Gross beta	Laboratory	25-50	50-100	90
Gamma	Laboratory	10	20	90

Sources: EPA 1976, 1979, and 1980b.

4 Sampling and Analytical Procedures

4.1 Field Sampling Procedures

Field sampling procedures are described in Section 4 of the work plan (Stefano et al. 1991).

4.2 Analytical Procedures

4.2.1 Field Screening

A portable HNu meter with a PID will be used to screen areas suspected of being contaminated with VOCs or semivolatiles. To increase detectability and standardize measurements, samples will be placed in sealed jars and taken indoors. Once the sample reaches ambient room temperature, the PID will be used to measure the total ionizable concentration of organic vapors.

Quality assurance of PID measurements will be addressed by calibrating the instrument, standardizing the measurement procedure, and analyzing appropriate QA samples, including periodic blanks, duplicates, and spiked samples. Instruments will be calibrated frequently by using a certified calibrant gas, such as propane, to ensure accuracy and comparability. Precision will be evaluated by replicate analyses of a fixed percentage of samples. Standard data forms will be used to record data and ensure completeness.

4.2.2 Field Laboratory Analysis

A field laboratory will be set up in the laboratory facilities at McMurdo Station for the level II analysis to support site characterization. Field laboratory analyses will be performed with a GC equipped with both flame ionization and electron capture detectors. These instruments can be used to perform qualitative and quantitative analyses of organic mixtures. The extent of the analyses will be determined by the level of calibration possible as well as the degree of sample preparation performed.

The main objective of field laboratory analysis is to characterize contaminated materials identified in the PID screening (or by other means) and select samples for a detailed analysis at the contract laboratory. This characterization will also (1) determine the presence or absence of carbon-, oxygen-, and halogen-containing substances; (2) indicate the number and boiling point range of components in a complex mixture (total petroleum hydrocarbons); and (3) identify characteristic complex mixtures, such as jet and diesel fuels and PCBs. In addition, this system will be used to positively identify and quantify a limited number of targeted analytes, such as selected solvents.

The accuracy and comparability of GC measurements will be ensured primarily by analyzing calibration mixtures. For a limited number of selected analytes, including some PCB mixtures, a calibration standard containing these substances will be analyzed periodically for both qualitative and quantitative analyses. When possible, the identification of target compounds and mixtures will be confirmed by analysis on a second GC column of different polarity. Complex hydrocarbon mixtures, such as fuels, will be analyzed using acceptable standards in conjunction with a hydrocarbon index standard (Kovat's index), consisting of a mixture of alkanes.

Because of the uniform response of the PID to hydrocarbons by mass, it is possible to quantify most components of a fuel mixture by using a limited number of components in an index standard. Unknown hydrocarbons, as well as other substances, can be identified by their hydrocarbon index value if that value is available to the analyst. Prior to the investigation, a table of Kovat's index values for common and suspected contaminants on the GC columns used will be compiled to supplement calibration standards.

The accuracy and precision of sample analyses will be evaluated by analyzing a minimum of 5% of QA samples, including blanks, duplicates, and matrix spikes. Contamination control will be demonstrated by analyzing method blanks (either reagent water or simply reagents) that have run through the entire sample preparation and analysis procedure. Method blanks will be run once for every set of samples from a given location, every 20 samples or every 72 hours, whichever is more frequent.

Accuracy and precision will be further evaluated by analyzing duplicate matrix spikes. A known concentration of selected calibration compounds will be added to aliquots or homogenized portions of a corresponding sample before matrix spikes and matrix spike replicates are prepared and analyzed. The recovery and comparability of matrix spike compounds will be used to evaluate the accuracy and reproducibility, respectively, of the analytical methods.

Data completeness will be ensured by using bound, hard-cover laboratory notebooks with numbered pages. The notebooks will be used to record the sample number; standard runs; QC samples; analyses required; methods and dates of collection, extraction, and analysis; sample mass and concentration/dilution information; and analytical results. Appropriately labeled chromatograms will accompany the notebooks.

4.2.3 Contract Laboratory Analysis

4.2.3.1 Techniques

Detailed chemical analysis will be available only at the contract laboratory. The contract laboratory will use established methods of analysis under strict quality control measures, primarily gas chromatography/mass spectrometry (GC/MS). This method is the instrument of choice in environmental analysis because it positively identifies most organic environmental contaminants.

The GC/MS technique can identify and quantify analytes from a TCL, even in complex environmental samples containing many compounds. It can also characterize unknown sample compounds, called tentatively identified compounds (TICs), by matching sample spectra to a spectral library, by interpreting sample spectra, or both. The combination of high-quality TCL analysis and the ability to characterize TICs yields a useful technique for environmental analysis.

To supplement GC/MS, the contract laboratory will also analyze PCBs by GC electron capture detection, a method that has a superior ability to detect these analytes. Both types of analysis will follow established EPA methods with the attendant QA/QC provisions.

4.2.3.2 Methods

Organics will be analyzed using EPA method 624 for VOCs, 625 for semivolatiles, and 608 for PCBs (EPA 1988a). The QA measures and required quantitation limits for the methods will apply. Likewise, inorganics will be analyzed by the appropriate EPA method (see Tables 4.1 and 4.2) with the attendant and required detection limits.

TABLE 4.1 EPA Methods Used in the Analysis of Metals[a]

	EPA Method	
Metal	Digestion	Analysis
Al	3005 3010 3050	6010
Al	3005 3010 3050	7020
Sb	3005 3040 3050	7041
Sb	3005 3010 3040 3050	6010
As	7060 7060 3050	7060
As	7061	7061
Ba	3005 3010 3050	6010
Be	3005 3010 3040 3050	6010
Ca	3005 3010 3050	6010
Ca	3005 3010 3050	7140
Cd	3005 3010 3040 3050	6010
Cd	3020 3040 3050	7131
Cr	3005 3010 3040 3050	6010
Co	3005 3010 3050	6010
Cu	3005 3010 3040 3050	6010
Fe	3005 3010 3040 3050	6010
Fe	3005 3010 3040 3050	7380
Pb	3020 3050	7421
Mg	3005 3010 3050	6010
Mg	3005 3010 3050	7450
Mn	3005 3010 3040 3050	6010

TABLE 4.1 (Cont'd)

	EPA Method	
Metal	Digestion	Analysis
Mn	3005 3010 3040 3050	7460
Hg	7470 7471	7470 7471
Ni	3005 3010 3040 3050	6010
K	3005 3010 3050	6010
K	3005 3010 3050	7610
Se	7740 3050	7740
Se	7741	7741
Ag	3005 3010 3050	6010
T1	3020 3050	7841
V	3005 3010 3040 3050	6010
Zn	3005 3010	6010

[a] Methods are described in Table 4.2.

Source: EPA 1986b.

TABLE 4.2 Description of EPA Methods for Metals Analysis by Method Number

EPA Method Number	Description of Analysis Method
3005	Acid digestion of waters for total recoverable or dissolved metals for analysis by flame atomic absorption spectroscopy or inductively coupled plasma spectroscopy
3010	Acid digestion of aqueous samples and extracts for total metals for analysis by flame atomic absorption spectroscopy or inductively coupled plasma spectroscopy
3020	Acid digestion of aqueous samples and extracts for total metals for analysis by furnace atomic absorption spectroscopy
3040	Dissolution procedure for oils, grease, or wax
3050	Acid digestion of sediments, sludges, and soils
6010	Inductively coupled plasma/atomic emission spectroscopy
7020	Aluminum (AA,[a] direct aspiration)
7041	Antimony (AA, furnace technique)
7060	Arsenic (AA, furnace technique)
7061	Arsenic (AA, gaseous hydride)
7131	Cadmium (AA, furnace technique)
7140	Calcium (AA, direct aspiration)
7380	Iron (AA, direct aspiration)
7421	Lead (AA, furnace technique)
7450	Magnesium (AA, direct aspiration)
7460	Manganese (AA, direct aspiration)
7470	Mercury in liquid waste (manual cold-vapor technique)
7471	Mercury in solid or semisolid waste (manual cold-vapor technique)
7610	Potassium (AA, direct aspiration)
7740	Selenium (AA, furnace technique)
7741	Selenium (gaseous hydride method)
7841	Thallium (AA, furnace technique)

[a] AA = atomic absorption.

Source: EPA 1989b.

5 Chain-of-Custody Procedures

The possession and holding of samples will be regulated and maintained through chain-of-custody procedures. Chain of custody is the means by which a sample and its container are traced from the time of collection by the field team to analysis at the contract laboratory. A person has custody of a sample if the sample is (1) actually in the person's possession; (2) within sight after being in the person's possession; and/or (3) in the person's possession at one time, but moved to a locked area to prevent tampering.

5.1 Sampling Containers

5.1.1 Preparation

Chain of custody begins when a sample is collected and sample containers are prepared. Sample containers will be purchased from I-Chem, a commercial supplier. Typical sample requirements, including containers, preservation, holding time, and sample volume requirements for all aqueous samples and for soils and sediments are presented in Tables 5.1 and 5.2, respectively.

5.1.2 Preservation

Steps to preserve environmental samples will begin immediately after the sample is collected. When an automated sampler is used (making it impossible to preserve every subsample), sample containers will be either maintained at 39°F (4°C) until compositing and sample splitting are completed or, for those samples being analyzed for metal constituents, acidified before attaching them to the automatic sampler. The pH and temperature of the final sample will be checked periodically and recorded before shipment to ensure adequate preservation. The field team will be equipped with field sample preservation kits, which may include nitric acid, sulfuric acid, hydrochloric acid, sodium hydroxide, and pH indicator paper.

Ice chests will be used to cool samples during field sampling, packaging, and shipment. A refrigerator or ice chest will be located in the site office for samples that require overnight refrigeration. The sampling team leader will monitor the refrigerator temperature and record the temperatures in the field logbook. Thermometers will be placed in ice chests used to transport samples from the field to the shipping area so that temperature can be checked periodically and recorded.

High-concentration, hazardous samples are samples with concentrations of from about 15% to 100% of any single contaminant. Concentrations are estimated on the basis of the source of the sample or results of field measurements. These samples generally are collected where little or no evidence of contaminant dilution exists, for example, surface impoundments, tanks, drums,

TABLE 5.1 Typical Sample Requirements for Aqueous Samples

	Container				
Analytical Parameter	Size (mL)	Type	Preservative	EPA-Recommended Holding Time[a]	Sample Volume
VOCs	40	Glass vial	4°C	10 d	120 mL/3- to 40-mL vials
Semivolatiles or PCBs	2,360	Amber glass jugs	4°C	Extract 5 d, analyze 40 d	2 L
Anions	125	HDPE[b]	4°C	48 h, NO_3 and PO_4; all others, 28 d	100 mL
ICP metals/ cations/Hg/Pb	500	HDPE	pH < 2 HNO_3	6 mo, except for 28 d for mercury	500 mL
Cyanide	1,000	HDPE	pH > 12[c]	14 d	1,000 mL
Gross alpha, beta screen	125	HDPE	pH < 2 HNO_3	Screen immediately	100 mL
Gamma analysis or screen	540	Plastic	pH < 2 HNO_3	1 yr	500 mL

[a] Holding times are from the date of collection.

[b] High-density polyethylene.

[c] *Federal Register* (1984).

Sources: EPA 1986b, except as noted.

spills, and direct discharges. Because of the high concentrations involved and the potential for preservatives to react violently with the constituents of the sample, no reagents or ice will be used with any of these samples.

5.2 Transport of Samples

5.2.1 Packaging and Handling

All samples will be packaged and transported in a manner that will protect the integrity of the sample and ensure against any detrimental effects from possible leakage. Packaging

TABLE 5.2 Typical Sample Requirements for Soils and Sediments

	Container				
Analytical Parameter	Size	Type	Preservative	EPA-Recommended Holding Time	Sample Volume
VOCs/ hydrocarbons	125 mL	Glass jar	4°C	10 d	50 g (minimum headspace)
Semivolatiles/ anions/PCBs	250 mL	Glass jar	4°C	Varied[a]	150 g
CLP metals/ICP metals/cations/ cyanide/metals/ Pb/Hg/Cr/Cr^{+b}/ As/Tl/Sn	250 mL	Glass jar	4°C	6 mo[b]	75 g
Gamma analysis/ gross alpha and beta analysis	60 oz	Plastic squat jar	None	1 yr	Fill to top

[a] Extract organics in 10 days; analyze within 40 days.

[b] Mercury within 28 days, cyanide within 14 days.

Sources: *Federal Register* 1984, EPA 1986a.

procedures will vary, depending on the suspected sample concentrations and the U.S. Department of Transportation (DOT) hazard class and international transportation regulations.

Custody seals will be placed on each shipping container, and clear, plastic tape will cover the seals to ensure they are not accidentally broken during shipment. The containers will be packaged in the following manner:

- Pack in a thermally insulated, environmentally controlled cooler.
- Pack sample container in a ziplock bag.
- Suspend bagged container in a sorbent material (vermiculite) and place in a large plastic (garbage) bag sealed with a twist tie.
- Place blue ice packs around the outer plastic (garbage) bag to maintain samples at about 39°F (4°C).

- Secure the package with three windings of reinforced strapping tape on each end.
- Incorporate a custody seal into each winding.
- Affix a packing list pouch listing the contents of the package.

The examining official executing the transfer of custody will sign the chain-of-custody record, and the record will remain with the cooler. An ANL staff member at the port of entry in New Zealand will take custody of the coolers and see that they are shipped or delivered for analyses at DSIR, Wellington, New Zealand, and Hazleton Laboratories, Madison, Wisconsin.

When a batch of coolers arrives at the laboratory, personnel will check the temperature. They will open one cooler per batch, place a thermometer inside and allow it to equilibriate, and record the temperature in a logbook. In addition, laboratory personnel will also check the temperature of a dedicated sample vial.

5.2.2 Holding Times

5.2.2.1 Regulations

The use of holding times for EPA programs originated with the National Pollutant Discharge Elimination System, authorized under the Clean Water Act, which was concerned with the loss of trace-level contaminants from wastewater samples collected and analyzed under this self-monitoring program. This practice continues under the CLP and is authorized under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) or Superfund, which implements essentially the same holding times for all water samples containing low levels of contaminants. The current recommended EPA holding times for water and for soils and sediments are shown in Tables 5.1 and 5.2, respectively.

All short-holding-time samples will be shipped "priority shipment" via air express courier in accordance with DOT regulations (48 CFR 171-178) and EPA sampling handling, packaging, and shipping methods (40 CFR 261.C.3).

5.2.2.2 Requirements of the Program

In accordance with the QA and analytical program requirements, Hazleton Laboratories and DSIR will process the samples as soon as possible after receipt. Sample holding times are critical and must not exceed the requirements given in Tables 5.1 and 5.2. Analytical protocols also dictate that, before laboratory processing begins, the sample containers within the cooler must neither be tampered with or opened at any time nor the package and its contents fumigated and/or sterilized during shipment. Only authorized laboratory representatives will handle samples.

If a sample is held too long, an artificially low analytical value for certain organic constituents may result because of chemical transformation or degradation due to microbiological influences. Some constituents are more sensitive than others to such degradations or transformations. As a result, samples must be extracted or analyzed within the specified holding time to ensure that results reflect the total values. Results of samples not analyzed within the specified holding times will be considered minimum values; that is, the actual concentration will be assumed for regulatory purposes to be equal to or greater than the concentration determined after the holding time has expired.

5.2.3 Shipping Procedures

5.2.3.1 Permits and Shipping Schedules

The shipment of field samples from Antarctica will be dictated by logistics. The following permits are needed: an import permit for samples retained for analysis within New Zealand, a permit for transshipment in New Zealand, and a U.S. Department of Agriculture (USDA) permit to ship samples to the laboratories within the United States from New Zealand. These mandated international stipulations and shipment procedures will influence the actual shipping time and receipt of samples by contract laboratories in New Zealand and the United States. Hazleton Laboratories should receive the sample shipment in about 7 days or longer after the shipment leaves New Zealand.

Applications were filed with the Ministry of Agriculture and Fisheries (MAF), Plant Protection Center, Auckland, New Zealand, on December 6, 1990, for an import permit and a transshipment permit for approximately 6 to 10 shipments from Antarctica between January 10 and February 28, 1991. Shipments to the DSIR laboratory are expected between January 10 and 30, 1991. Each shipment will consist of approximately two to five packages (coolers). Each cooler will contain multiple glass and/or plastic sample bottles containing environmental soil, rock, and liquid (water) samples. On December 13, 1990, the MAF granted permits for shipment. In early January 1991, the USDA granted permits to receive samples from New Zealand to ANL's Analytical Chemistry Laboratory and Hazleton Laboratories.

5.2.3.2 Operations

Shipping operations will involve the following steps:

- The ANL field team will ship the sample containers from Antarctica to Christchurch, New Zealand, by air.

- The project QA officer or designee in New Zealand will receive the shipment. This person must have an import permit to process shipments to the DSIR laboratory and also must have a transshipment permit to process shipments to the United States.

- The project QA officer or designee will oversee the shipment of containers within New Zealand or to the United States.

- A courier service representative in New Zealand will deliver the packages to the DSIR laboratory in Wellington, New Zealand.

- At the U.S. port of entry (Chicago), a custom broker of the international courier will ensure that the package proceeds through clearance.

- The custom-cleared shipment will be delivered to the U.S. contract laboratory via domestic courier.

Sample containers from McMurdo Station to New Zealand will be shipped via an air carrier whose flight schedule is generally limited to one or two flights per week. Operation of these flights is also governed by the prevailing weather. As a result, flights may be postponed for several days or cancelled because of inclement weather. These limitations impact sample receipt in New Zealand and ultimately by the contract laboratories, which influences the sample holding times. While the holding time requirement may not seriously affect the analytical data, it is important that all parties involved in this program are cognizant of the potential issues associated with the shipment and analysis of samples from Antarctica.

5.2.4 Radioactive Materials

5.2.4.1 Radiation Screening

Before a sample is prepared for shipment, qualified field personnel will screen the sample to determine if the sample will be shipped as a radioactive shipment, how it should be packaged, and to which laboratory it can be shipped for analysis. Field radiation screening will consist of the following steps:

- Soil will be surveyed in screening and homogenization pans for gamma radiation before the soil is placed in containers.

- Hand-held Geiger-Mueller instruments will be used to survey each sample. These instruments will be calibrated before use in the field. The survey will be conducted by qualified and trained personnel.

- A contact gamma survey will be performed on the outside of the sample container. In the case of a nonuniform sample, that is, soil or sludge samples, readings will be taken on all sides of the container. A contact reading will also be taken on the bottom of the sample bottle.

- All results will be recorded in a radiation screening logbook.

5.2.4.2 Packaging

For shipping purposes, a sample is considered radioactive if it contains a specific activity greater than 2×10^3 pCi/g (solids) or 2×10^3 pCi/L (liquids). Radioactive samples will be shielded to protect the health and safety of personnel and the public who will handle the sample shipments. Samples will be packaged in steel-belted coolers and checked by personnel on-site to ensure readings are less than 0.5 mrem/yr at contact.

5.3 Field Custody

5.3.1 Responsibilities

ANL field personnel are responsible for the custody of samples from the time samples are collected until transfer to the sample shipper or the laboratory. A minimum number of persons will handle the samples to reduce the number of transfers. Basic chain-of-custody procedures for the project are given below.

5.3.2 Procedures

Each sample collected will be identified with a unique ANL sample number by affixing a self-sticking, prenumbered label on the sample container. The sample number and all sampling information, including a list of preservatives that have been added, will be recorded in the field logbook. The sample will be placed on ice in a thermal chest. The chest will remain within sight of the sampler or will be locked in the field vehicle for temporary storage and transport to the sample staging area.

At the sample staging area, each sample container will be rinsed with organic-free water to remove any exterior debris. Information will be recorded on a chain-of-custody record (Figure 5.1) to account for each sample. ANL will send at least 5 to 10 blind replicate samples to Hazleton Laboratories. Blanks and other QA samples (see Section 5.4) also will be sent with each group of samples. To ensure the laboratory does not know the identity of these QA samples, information on the chain-of-custody records accompanying the samples will not describe the sample; however, all other information on the record will be included. The sample will be described only on the ANL copy of the chain-of-custody record. The ANL copy of the record will become a permanent record in the project files.

The sampler will sign and record the date and time in the appropriate block before relinquishing custody. The sample shipper will also sign and record the date and time in the appropriate block to accept custody. This procedure will be followed each time samples are transferred.

CHAIN-OF-CUSTODY RECORD

PROJ. NO.	PROJECT NAME
SAMPLER: (Signature)	

Sta. No.	Date	Time	Comp.	Grab	Station Location	No. of Con-tainers							REMARKS

Relinquished by:(Signature)	Date/Time	Received by:(Signature)	Relinquished by:(Signature)	Date/Time	Received by:(Signature)
Relinquished by:(Signature)	Date/Time	Received by:(Signature)	Relinquished by:(Signature)	Date/Time	Received by:(Signature)
Relinquished by:(Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks:	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

FIGURE 5.1 Chain-of-Custody Record

Sample shipment procedures are unique to the international location. The sample shipper will ensure samples are properly packaged in ice chests for dispatch to the appropriate laboratory. The sample shipper will sign and record the date and time in the appropriate block of the chain-of-custody record just before the cooler is closed. The laboratory copies of the custody records will be placed in plastic bags and taped to the inside cover of the appropriate cooler. Then the cooler will be sealed at each end with strapping tape. Samples will be shipped by available common carrier at the international location to the contract laboratory. The sample shipping receipts will be retained as part of the permanent chain-of-custody documentation.

When the contract laboratory receives the samples, the laboratory sample coordinator will sign the chain-of-custody forms and record the date and time. The sample coordinator will complete all the appropriate laboratory tracking sheets and logs and report any problems to the ANL sample shipper.

5.4 Laboratory Custody

The laboratory sample coordinator will be responsible for the custody of samples from the time the samples are received until they are discarded. The contract laboratory has established detailed standard operating procedures for sample handling, storage, and disbursement for analysis. These procedures include examples of the sample receiving and tracking information sheet, sample logging sheets, laboratory assignment sheet, and so on. The laboratory sample coordinator also will verify the preservation of samples when they are logged in at the time of arrival. The parameters (i.e., VOCs, semivolatiles, and PCBs) analyzed for this contract do not require chemical preservation, but they must be held at cool temperatures. Other nonroutine parameters, such as metals, do require chemical preservatives.

Holding conditions of the samples will be checked and recorded. Also, if the sample parameter requires chemical preservation for pH adjustment, a pH paper will be used to check the pH of the aliquot prior to sample preparation for analysis. Actual results will be recorded. This information will be given to the laboratory project manager.

6 Calibration Procedures and Frequency

Field measuring equipment will be calibrated according to the instrument manufacturer's specifications and/or as described in field testing methods (EPA 1987). Calibration procedures and frequency for the field PID and field laboratory GC measurements are described in Sections 4.2.1 and 4.2.2. For contract laboratory analysis of VOC and semivolatile TCL analytes by EPA methods 624 and 625 (EPA 1988a), an initial five-point calibration curve will be run. Thereafter, a daily standard at the 50-ng level will be run and verified against the initial curve before any samples are analyzed. A specified subset of the TCL will be used for verification. This subset is called the continuing calibration compounds, the relative response factors of which should be within 20% of their values in the initial curve. Before analyzing the daily standard, a system performance check solution containing specified performance-sensitive compounds will be analyzed and determined to meet minimum detectability requirements.

Tune compounds specified in methods 624 and 625 can be used to perform an initial and daily instrument tune before a GC/MS analysis. To ensure the integrity of sample spectra and their comparability to library spectra, the instrument will produce a spectrum on the tune compounds that meets the resolution and relative intensity requirements of the methods.

For PCBs, an initial five-point calibration curve (EPA method 8080 [1986b]) will be run on at least one of the seven analyzed commercial PCB mixtures known as Aroclors. A high-chlorination mixture, such as Aroclor-1254, is preferred to establish the detection limits and linearity range of the analytical system. Thereafter, a daily standard of each Aroclor at an intermediate concentration will be run before any samples are run. If a different Aroclor is detected in any sample run, a five-point calibration of that Aroclor will be run to establish linearity. A sample analysis that is not in the linear range will be diluted accordingly and rerun. To check system performance, after every fifth sample a standard of the PCB surrogate recovery compound will be run.

For inorganic analysis, instrument calibration and frequency will follow procedures outlined in EPA (1986b, 1989b) and described in the contract laboratory's standard operating procedures. Inorganic calibration standards will be purchased from chemical suppliers who can certify purity and concentration. The laboratory will check calibration standards routinely against EPA or National Institute of Standards and Technology traceable standards (EPA 1986b, 1986b). Calibration protocol requires an initial blank and calibration verification and continuing blank and calibration verifications at a frequency of 10%. Inorganic analysis instruments will be calibrated daily before they can be used in the analysis of samples for this project.

7 Analytical Procedures

7.1 Field Measurements

Procedures for field measurements during sampling are discussed in Section 4 of the work plan (Stefano et al. 1991).

7.2 Laboratory Analytical Procedures

Analytical procedures are discussed in Section 4.2 of this plan.

8 Data Analysis and Reduction, Validation, and Reporting

8.1 Analysis and Reduction

Data analysis and reduction for field PID and field laboratory GC methods are described in Sections 3.1, 3.2, 4.2.1, and 4.2.2. For contract laboratory analysis of VOCs, semivolatiles, and PCBs, data analysis and reduction are described in EPA methods 624, 625, and 608, respectively (EPA 1986b, 1988a). For VOCs and semivolatiles analyzed by GC/MS, data reduction includes identification of TCL compounds by relative retention time and by comparison of sample and standard spectra. Concentrations of TCL compounds are calculated by measuring areas of extracted ion profiles of specified ions for both analyte and internal standard, and by applying a relative response factor determined in the daily calibration standard run. This value is translated to sample concentration by applying the required sample size and dilution/concentration factors. Retention time of selected reference peaks and general appearance of the chromatogram are used to identify PCBs. The areas of the reference peaks in the external standard method are used to determine concentrations.

Form I is used to report PCB and TCL results, as provided in EPA (1988a) for the respective methods. Other data are reported on forms as follows:

- Form II, surrogate recovery results;
- Form III, matrix spike and matrix spike duplicate recovery results;
- Form IV, blank summaries;
- Form V, GC/MS tune results;
- Form VI, five-point calibrations;
- Form VII, continuing calibration results;
- Form VIII, internal standards summaries;
- Form IX, pesticide and PCB standards summary (PCBs only); and
- Form X, pesticide and PCB identification reports.

In addition to the reporting forms, the contract laboratory will provide hard copies of total ion chromatograms for all samples and standards for VOCs and semivolatiles. Labeled hard copies of sample and standard spectra for any TCL compounds identified also will be required. Likewise, for TICs reported, hard copies of the computer-generated library matches showing sample and library spectra will be required.

Inorganic analyses data will be reported on the forms used in EPA (1986b) or (1989b).

8.2 Validation

ANL will review data reported by the contract laboratory and check the data to determine if they meet performance requirements and guidelines provided in the methods. The validity and usefulness of data outside requirements and guidelines will be determined on the basis of the professional experience of team members with these types of data. When specific questions arise related to the data quality, the contract laboratory will be consulted. The validity of the data in question will be judged on the basis of the information provided.

8.3 Reporting

The contract laboratories will provide tables that summarize quality control information and quality control data elements. These analytical data tables will include sample number, sample matrix description, parameters tested and compounds detected, and their detection limits.

No formal report of analytical data resulting from field screening of samples is planned. The sampling and analysis program for field screening will be strictly adhered to for satisfactory confirmation for laboratory analysis for required chemical parameters.

Results will be incorporated in the sampling and analysis plan for the preliminary site investigation report as data tables, maps showing sampling locations and possibly locations showing high values, and supporting text. No formal interim reports are planned; however, results will be discussed with NSF technical staff by telephone and will be reported in memoranda if requested.

9 Quality Control Checks and Frequency

Quality control checks for soil, sediment, surface water, and groundwater sampling and analyses are summarized in Table 9.1.

9.1 Blank Samples

Data quality requirements mandate that a percentage of samples shipped to the laboratory must be composed of samples known to contain no measurable concentrations of the chemicals for which the sample is being analyzed. These samples will be used for quality control checks of both the container preparation procedures and the laboratory methodology. The containers for soil, sediment, surface water, and groundwater samples will be filled with organic-free water for organic water analysis and distilled water for inorganic water analysis. The samples will be labeled, documented, and handled in the same manner as other similar (field) samples. A field blank will be sent to the laboratory for each batch of shipping containers, and a trip blank will be sent to the laboratory for each batch of shipping containers for volatile organic analysis.

9.2 Field Blanks

At least 5% of the samples collected for each sample matrix will be field blanks. These samples will be used as a quality control check of decontamination procedures for sampling devices used during soil, sediment, surface water, and groundwater sampling.

9.3 Laboratory Quality Control Checks

Laboratory quality control checks will include internal quality control methods, such as analysis of spike samples, split samples, internal standards, zero and span gases, quality control samples, calibration standards, calibration devices, and reagent checks. Quality control checks for the contract laboratory are largely implicit in the methods used, such as demonstration of

Table 9.1 Quality Control Checks for Soil, Sediment, Surface Water, and Groundwater Sampling and Analysis

Quality Control Checks	Minimum	Maximum
Field replicates (%)	5	20
Field blanks (%)	5	20
Trip blank (per shipment)	1	1

GC/MS tune, calibration curves and daily standards, system performance checks, multiple internal standards for sample analysis, surrogate recovery standards in each sample, method blanks for control of system contamination, and analysis of matrix spikes and matrix spike duplicates to evaluate matrix effects and reproducibility. In addition to these requirements, an on-site assessment of laboratories of DSIR, New Zealand, will be performed. Furthermore, the U.S. contract laboratory will submit results of their most recent EPA-sponsored performance evaluation samples.

10 Performance and System Audits

The ANL project QA officer or designee will plan at least one performance and system audit if feasible during field sampling. If not feasible, the project QA officer will monitor activities via teleconferences with the project field team.. This audit will ensure that the procedures used to conduct field operations and field laboratory analyses and to assess data precision, accuracy, and completeness have been properly followed. On the basis of the QA audit, the QA officer will issue a nonconformance notice, formulate and recommend a corrective action acceptance report, prepare an audit report, and coordinate with contract laboratory QA officers. Audit protocol will be based on the quality control procedures outlined in standard operating procedures for each of the field operations or laboratory procedures.

10.1 Audit Responsibilities

The ANL project QA officer will appoint independent auditors for each specific set of activities. Auditors will be thoroughly familiar with the QA/QC requirements of the work plan and also will be technically proficient in areas relevant to their assigned audit activities. Each auditor will outline specific tasks in conjunction with the project QA officer and meet with appropriate key personnel to become familiar with QA/QC plans, the scope of the audit, the auditing plan and schedule, and the target date for completion of the audit. Auditors will use a checklist and will document and report all audit results to the project QA officer. If a nonconformance item is identified, the auditor will file a nonconformance report in addition to the audit report.

10.2 Field Audits

The ANL project QA officer or designee will conduct at least one field audit during field sampling, if feasible. This systems and performance audit will monitor field measurements during sampling. The system audit will focus on the appropriateness of personnel assignments and expertise, the availability of field equipment, adherence to standard operating procedures for sample collection and identification, sample handling and transport, use of QA/QC samples, chain-of-custody procedures, equipment decontamination, and documentation with respect to all QA/QC requirements. A system audit checklist for field operations is shown in Appendix B. The auditor will use only appropriate items on the checklist during the audit. Performance audits will focus on methods of field measurements, including water levels, chemical parameters, and instrument calibration. All nonconformance items will be documented and addressed as described in Section 13. The project QA officer will retain a written report for each audit. To ensure the quality of data, replicate samples will be sent to Hazleton Laboratories for analyses to determine the accuracy, precision, and comparability of data generated by laboratories of the DSIR, New Zealand.

10.3 Laboratory Audits

The ANL project QA officer or designee will make at least a quality control check either before or during sample processing for on-site laboratory assessment of DSIR, New Zealand. Routine internal performance and systems audits at the contract laboratory will monitor all functions to which a sample is exposed, including laboratory chain of custody, internal sample tracking, analytical data documentation, instrument calibration and QA/QC protocols, and data reporting to ANL. These procedures will be inspected for adherence to internally established standard operating procedures by the laboratories. The laboratory will send relevant audit report materials to the ANL project QA officer. The ANL project QA officer will retain a copy of the report. All nonconformance items will be documented and addressed as described in Section 13.

In addition, the contract laboratories participate in several external performance and system audits sponsored by government and independent organizations. The QA officer will supply reports of such audits.

Any modifications in field or laboratory procedures that result from systems and performance audits must be approved by the project manager and project QA officer and documented in writing. Copies of all systems and performance audit reports will be maintained in the project files.

11 Preventive Maintenance

Preventive maintenance has three objectives: ensure accuracy of measurement systems, minimize downtime, and collect critical spare parts and backup systems and equipment. Field and laboratory operational personnel will perform routine maintenance and keep tools and spare parts available for this purpose. Maintenance that cannot be performed by equipment maintenance personnel will be performed by a person certified or trained to repair the instrument.

Maintenance activities will be performed in accordance with the manufacturer's specifications and standard operating procedures. Instruments will be calibrated to proper specifications following maintenance to ensure proper completion of the maintenance procedure. The date of maintenance will be recorded in the master calibration and maintenance logbooks for the associated instrumentation. Adequate spare parts will be available to ensure that appropriate quality control is maintained; these parts and supplies are in addition to those normally required.

11.1 Field Equipment

Minimal maintenance is required for field testing equipment. Spare parts will be kept on-hand. Battery checks for all instruments will be made before sampling begins and periodically through the day. The sampling crew will keep extra batteries to use for replacement, as needed. Damaged instruments will be replaced in a sufficient turnaround time for the field sampling and measurements to continue.

11.2 Laboratory Equipment

Proper maintenance of laboratory equipment is essential to the success of the investigation, particularly because of the remote location of the site and tight time constraints involved with the field laboratory. Likewise, good maintenance practices at the off-site contract laboratory are critical to meet the performance and time requirements of the methods. In chromatographic analyses, good maintenance practices will include periodically running column performance check solutions, trimming capillary column ends, properly adjusting gas flows, cleaning injector liners, cleaning detectors, changing injector septa, and cleaning syringes.

In addition to these maintenance measures, the GC/MS system will require periodic source cleaning, filament replacement, system breakouts, leak testing, and vacuum and cooling system maintenance. A regular tune on a perfluorinated calibration compound is standard practice in the operation of GC/MS systems. This tune is in addition to the tune requirements of the EPA methods, which use different tune compounds.

12 Procedures Used to Assess Measurement Data

The QA objective for measurement data is to ensure that characterization data are of known and acceptable quality. Accuracy, precision, and completeness are essential measures for assessing the quality of the analysis data and for applying the data appropriated during the decision-making process.

The QA objectives for analytical data from the samples collected include descriptions of accuracy, precision, representativeness, comparability, and completeness, which are discussed in Section 3. The specific equations to assess accuracy, precision, completeness, comparability, MDL, and limit of quantitation (LOQ) for the program are given in the following sections.

12.1 Accuracy

Accuracy is expressed as percent recovery (%R). For situations where a standard reference material is used, %R is calculated as follows:

$$\%R = \frac{C_m}{C_{srm}} \times 100$$

where:

C_m = measured concentration value obtained by analyzing the sample, and

C_{srm} = "true" or certified concentration of the analyte in the sample.

For measurements where matrix spikes are used, %R is calculated as follows:

$$\%R = \frac{S - U}{C_{sa}} \times 100$$

where:

S = measured concentration in the spike aliquot,

U = measured concentration in the unspiked aliquot, and

C_{sa} = actual concentration of the spike added.

12.2 Precision

Precision is expressed either as the relative percent difference (RPD) for duplicate measurements or the relative standard deviation (RSD) for three or more replicate measurements. For duplicate measurements, RPD is calculated as follows:

$$\mathrm{RPD} = \frac{C_1 - C_2}{(C_1 + C_2)/2} \times 100$$

where C_1 and C_2 are the two values obtained by analyzing the duplicate samples (C_1 is the larger of the two observed values).

For three or more replicate measurements, RSD is calculated as follows:

$$\mathrm{RSD} = s/\bar{y} \times 100$$

where:

s = standard deviation, and

$\bar{y}$ = mean replicate sample analyses.

The standard deviation is defined as follows:

$$s = \sqrt{\sum_{i=1}^{n} \frac{(y_i - \bar{y})^2}{n - 1}}$$

where:

y_i = measured value of the i of the replicate sample analysis measurement, and

n = number of replicate analyses.

12.3 Completeness

Laboratory completeness, expressed as the percent complete (%C), is calculated as follows:

$$\%C = \frac{V}{n} \times 100$$

where:

V = number of valid analytical results obtained, and

n = number of determinations required for the actual number of samples collected.

12.4 Method Detection Limit

The MDL for all measurements is defined as follows:

$$\text{MDL} = t_{(n-1,\ 1-\alpha = 0.99)} \times s$$

where:

$t_{(n-1,\ 1-\alpha = 0.99)}$ = t distribution value appropriate to a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom, and

s = standard deviation of replicate measurements.

12.5 Limit of Quantitation

The limit of quantitation (LOQ) for all measurements is defined as follows:

$$\text{LOQ} = 3.3 \times \text{MDL}$$

where the MDL is the method detection limit.

13 Corrective Action

If problems develop in the contract laboratory, the ANL project QA officer will be notified. The QA officer will determine the necessary corrective action on the basis of technical judgment and knowledge of established procedures or on predetermined limits under the work plan (Stefano et al. 1991). Data may be rejected as a result of either data validation procedures or quality control checks. If so, the sample matrix will be resampled and reanalyzed, if possible.

Project tasks or items that do not conform to the QA/QC requirements, based on field standard operating procedures, will be detected by system and performance audits or identified by project members who know or suspect an activity is not being performed in accordance with those requirements. The ANL project QA officer will be informed of all such defects and will act in a timely manner to verify if corrective action is necessary. If corrective action is necessary, the QA officer will use the following protocol to ensure that the nonconforming activity meets specified QA/QC requirements:

- Nonconformance reports will be filed for all activities not performed in accordance with the established requirements.

- The project QA officer, in conjunction with the project manager, will review the activity to identify the source of the problem and develop a plan to correct the nonconforming items. Corrective actions for field sampling and testing problems will be developed with assistance from the field team; corrective actions for laboratory problems will be developed with assistance from the laboratory manager.

- Any work dependent on the nonconforming activity will be halted until the problem is corrected. The project manager will have the ultimate responsibility for ensuring that corrective actions are fully implemented.

- The project QA officer will be notified when corrective actions are completed. At that time, a follow-up audit will be conducted, and a written report of corrective action acceptance will be filed with the program manager.

14 Reports to Management

The QA officer will prepare periodic QA reports at the end of an activity for distribution to the program manager and the project manager. At the end of the preliminary site investigation, ANL will submit a QA report to NSF. The report will give significant QA problems encountered, if any, and proposed corrective actions and changes in the work plan.

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Appendix A:

Qualifications and Experience Profiles of the ANL Project Team

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Qualifications and Experience Profiles of the ANL Project Team

Argonne National Laboratory (ANL) is committing a core team of qualified professionals to implement the work plan for the preliminary site investigation for McMurdo Station, Ross Island, Antarctica. The project team will be supported by staff members of ANL's Illinois, Washington, D.C., and other offices as needed. The organization of the ANL team is shown in Figure 2.1. Members of the project team are experienced in conducting hazardous waste management studies. Relevant experience and qualifications of key team members are summarized below.

Douglas Canete, who will be the program manager for the project, has served in that capacity for many other projects involving a range of services, including records searches, initial site evaluations, contamination assessments, and remedial action planning and design. Canete has extensive experience in the environmental assessment of facilities for long-term storage of wastes. He currently serves as program manager for the remedial investigation and feasibility study (RI/FS) of a steel mill site in Chicago, Illinois, for the U.S. Department of Commerce (DOC). Studies at this site involve the evaluation and selection of remedial action alternatives for several contaminated areas.

Canete has been program manager for projects involving diverse environmental concerns, including environmental audits for industrial and commercial properties, projects involving a compliance program with a wide environmental compliance data base, and hazardous waste regulations and programmatic initiatives for such federal agencies as the DOC, National Science Foundation (NSF), U.S. Department of Energy (DOE), and the Department of the Army. Canete also has served as a private consultant for numerous Fortune 500 firms, such as AT&T Consumer Products and Honeywell Information Systems. He is well versed in many areas of environmental and energy-related policy and planning, policy evaluation, and environmental impacts analysis. Canete received a B.S. in entomology from the University of California, Berkeley, in 1974.

James Stefano, who will be the project manager and geotechnical investigations coordinator, joined ANL in early 1990. Formerly, Stefano was employed as an engineering geologist by the U.S. Corps of Engineers, Baltimore District. During his four years with the Corps, Stefano developed, implemented, and supervised drilling and sampling programs for geotechnical and environmental investigations in a variety of geologic and geographic settings. Prior to his employment with the Corps, Stefano worked for the Illinois Department of Natural Resources as a research assistant studying the proposed Super Collider site at Fermi National Laboratory. His expertise includes environmental and geotechnical drilling, analysis of geologic structures, groundwater monitoring, and borehole geophysics. Stefano received a B.S. in geology from Illinois State University in 1982 and an M.S. in geology from Fort Hays State University in 1985.

Surya Prasad, who will be the project quality assurance (QA) officer, has extensive professional experience in environmental assessments and hazardous waste site investigations. He joined ANL in mid-1990 and has been involved in environmental assessments and hazardous waste management studies based on current environmental laws, regulations, and guidelines. Most of his experience before joining ANL was in the environmental consulting industry.

Prior to joining industry, Prasad was involved in research and teaching in academia. He is a certified professional soil scientist and has completed EPA-approved QA/QC and health and safety training courses for hazardous waste management studies and site investigations. He has prepared QA/QC plans as input on hazardous waste management studies and also has served as the QA officer and site manager on RI/FSs at hazardous waste sites under the Comprehensive Environmental, Response, Compensation, and Liability Act (CERCLA) and Resource Conservation and Recovery Act for private industries, potentially responsible parties, and federal agencies. In that capacity, he prepared QA plans for deep soil and bedrock borings, well installation, and aquifer testing; surface water, sediment, soil, and waste sampling; and groundwater investigations. He has conducted systems, performance, and method-specific audits during field and laboratory investigations and is familiar with U.S. Environmental Protection Agency (EPA) QA guidelines used in hazardous waste site investigations. As a principal investigator, he has conducted environmental impact statements, risk and endangerment characterizations, contamination fate and transport assessments, and toxicological assessment studies of potentially hazardous chemicals at waste sites for the EPA, U.S. Department of the Interior/Bureau of Land Management, Department of the Army, U.S. Army Toxic and Hazardous Materials Agency (USATHAMA), and other federal agencies and private chemical industries within the United States. Prasad received a B.S. in agriculture from the A.P. Agricultural University in India in 1969, an M.A. in agriculture from Sam Houston State University in 1971, and a Ph.D. in soil chemistry from Rutgers University in 1977.

Mary Wozny, who will be the health and safety officer for the project, has been on the ANL staff since June 1990. Wozny also served as an intern at ANL's Washington, D.C., office as a legislative tracker and analyst. Recently, she was appointed the health and safety officer for the Wisconsin steel mill site investigation. Her educational background includes the fundamentals of industrial hygiene, risk assessment, air and water monitoring, hazardous waste site assessment, community health, and epidemiology. Wozny received a B.S. in health arts from the College of St. Francis in 1986 and an M.S. in public health in environmental and occupational health from the University of Illinois at Chicago in 1990.

Peter Lindahl, who will be the sample analysis coordinator for inorganic parameters and will review analytical data, joined the Analytical Chemistry Laboratory (ACL) at ANL in 1984. From August 1986 to September 1989, he served as ACL project manager for the DOE Environmental Survey Program. In December 1987, he was appointed leader of the Environmental Analysis Group. Lindahl has held staff positions at Exxon Production Research Company, Perkin-Elmer Corp., and the Illinois State Geological Survey. He has experience in the area of atomic spectroscopic applications to geological and environmental analyses. His primary research interests are in QA/QC and data quality assessment for environmental analyses, and in the development of analytical methods for determining inorganic elements in environmental samples and coal. Lindahl received a B.A. in chemistry from Lake Forest College in 1964, an M.A. in inorganic chemistry from Southern Illinois University in 1967, and a Ph.D. in analytical chemistry from Southern Illinois University in 1972.

Carl Bebrich, who will serve as a member of the field team, has directed many large-scale focused site investigations for federal sponsors, including DOE, Western Area Power Administration (WAPA), Department of the Navy, USATHAMA, and DOC. These investigations have included sites with wide-ranging contamination (organic, inorganic, and radionuclide wastes) and sites with single analytes. Bebrich has extensive experience in field activity management, logistics planning, and sampling design. He served as a sampling and analysis liaison to the DOE Environmental Survey Program, a large-scale study of DOE facilities, including nuclear weapons manufacturing and non-defense-related research and development facilities. In this role, he developed detailed guidelines concerning sampling protocol, sampling design, sample and document control, health and safety, and QA. More recently, Bebrich has been involved in the development of an interagency agreement to undertake an RI/FS and cleanup of a large steel mill with extensive organic and inorganic contamination (DOC); an analysis of the Navy's environmental compliance organization; and site inspections for a variety of single and multiple contaminant releases at numerous facilities (WAPA, Navy, and USATHAMA).

Bebrich is currently ANL liaison to DOE's Environmental Restoration Group. He has been with ANL for six years and has developed and managed applied research programs involving hazardous waste sites throughout the continental United States and Hawaii. Bebrich received a B.A. in anthropology (with a minor in chemistry) from the University of California at Los Angeles in 1966 and an M.A. in anthropology (with a minor in statistics) from Pennsylvania State University in 1971.

Kurt Picel, who will be the sample coordinator for organic analysis and will review analytical data, is experienced in environmental sampling and analysis. He has specialized in organic contaminant analysis and worked as a chemist and group leader in an environmental testing laboratory from 1979 to 1983. At ANL, Picel has conducted research on environmental fate processes for coal-derived liquid fuels and by-products while setting up and operating a full-capability organic analysis laboratory in support of his work and for regulatory-driven environmental analysis.

Picel is currently involved in a remedial investigation of a former steel manufacturing and processing site, overseeing sampling and regulatory functions for site demolition, and conducting background investigations for preparing remedial action plans. Picel has more than 11 years of gas chromatography (GC) and GC/MS experience. He joined ANL as a doctoral candidate in 1983, continued as a postdoctoral fellow in 1985, and joined the scientific staff in 1986. He received a B.S. in chemistry from Western Michigan University in 1976, an M.S. in environmental health sciences from the University of Michigan in 1979, and a Ph.D. in environmental chemistry from the University of Michigan in 1985.

Duane Knudson, who will be the data management coordinator for the project, has extensive experience in data reduction and translation. He is currently manager of the Environmental Management Systems Group, which is developing environmental information management systems, models, decision support tools, and expert systems for environmental applications. Knudson's experience includes management of data reduction, data quality review, and report preparation for several sampling and analysis projects and development of configuration control procedures for the ANL computer systems development program for the Joint Chiefs of Staff.

Jeffery Schubert, who will be the principal investigator in hydrogeology, has been with ANL for 15 years. While on the staff at ANL, he has conducted basic and applied research at mining and waste disposal sites throughout the United States in a variety of hydrogeologic settings. He is currently investigating geochemistry and subsurface transport mechanisms of radionuclides and metals in basalts and tuffs at a uranium mine in Oregon. He also is completing a project in Hawaii that involves the migration potential of polychlorinated biphenyls in basalts and basalt residual soils. Schubert's expertise includes sampling and analytical design, well installation, pumping tests, groundwater modeling, geochemical analyses, and surveying. He received a B.S. in geology from the University of Illinois in 1973 and an M.S. in hydrogeology from Pennsylvania State University in 1975.

Lyle McGinnis, who will be the project geophysicist and will advise the field team during subsurface investigation and field data interpretation, has 35 years of experience in environmental geology and geophysics. Since joining ANL in 1985, he has been involved in environmental geology and geophysical studies throughout the United States. Most of his previous 30 years of experience were in academia, where he served as chair and faculty member in the geology departments at Northern Illinois University and Louisiana State University. McGinnis presently holds an adjunct professor appointment at Northern Illinois University.

McGinnis has held staff or collaborative appointments with state geological surveys in Illinois, Minnesota, and Louisiana, and with the U.S. Geological Survey in Woods Hole, Massachusetts. He has conducted research with the Desert Research Institute in Nevada; the United Nations Development Program in Kabul, Afghanistan; and NSF's Division of Polar Programs in East and West Antarctica, beginning during the International Geophysical Year in 1957 and continuing until 1985. In addition, he has been employed by Carter Oil Company, a subsidiary of Standard Oil New Jersey (now Exxon), where he worked on seismic crews in the Rocky Mountain region as an interpreter. McGinnis received a B.S. in physics from St. Norbert College in 1959, an M.S. in geophysics from St. Louis University in 1960, and a Ph.D. in geology from the University of Illinois in 1965.

Terri Patton, who is a geologist with the Environmental Restoration Group at ANL, is responsible for geological and hydrological characterizations, groundwater monitoring program evaluation, sampling plan development, field sampling, and data analysis for environmental site assessments related to the CERCLA program. Since joining ANL in 1986, she has researched methods for extracting tin isotopes from sediments and has studied the behavior of nuclides in surface waters. Before joining the staff at ANL, Patton was employed as a radiochemist by Teledyne Isotopes Midwest Laboratories. During three years with Teledyne, she performed and supervised chemical analyses for radionuclides in various environmental media. Patton received a B.S. in geology from Southern Illinois University in 1982 and an M.S. in geology from Northeastern Illinois University in 1989.

Robin Limberger, who will assist in compiling laboratory chemical data, has served as a technical assistant on several environmental projects, including projects relating to agricultural environmental issues. She also assisted with research in areas ranging from water and soil pollution, multiple crop production, and integrated cropping systems. Limberger received a B.S. in agronomy from the University of Maryland in 1989.

Appendix B:

System Audit Checklist for Field Operations

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System Audit Checklist for Field Operations

Project Name ____________________

Project Number ____________________ Date ____________________

Location ____________________

Team Members ____________________ Field Team Leader ____________________

Name of Auditor ____________________ Signature of Auditor ____________________

Yes__________ No__________ 1) Is a set of accountable field documents checked out to the site manager?
Comments: ____________________

Yes__________ No__________ 2) Is the transfer of field operations from the site manager to field participants documented in a logbook?
Comments: ____________________

Yes__________ No__________ 3) Is there a written list of sampling locations and descriptions?
Comments: ____________________

Yes__________ No__________ 4) Are samples collected as stated in the work plan or as directed by the site manager?
Comments: ____________________

Yes__________ No__________ 5) Are samples collected in the type of containers specified in the project plan or as directed by the site manager?
Comments: ____________________

Yes________ No________ 6) Are samples preserved as specified in the project plan or as directed by the site manager?
Comments: ________________________

Yes________ No________ 7) Are the number, frequency, and type of samples collected as specified in the project plan or as directed by the site manager?
Comments: ________________________

Yes________ No________ 8) Are the number, frequency, and type of measurements taken as specified in the project plan or as directed by the site manager?
Comments: ________________________

Yes________ No________ 9) Are samples identified with sample labels?
Comments: ________________________

Yes________ No________ 10) Are blank and duplicate samples properly identified?
Comments: ________________________

Yes________ No________ 11) Are samples and serial numbers for samples split with other organizations recorded in a logbook or on a chain-of-custody record?
Comments: ________________________

Yes________ No________ 12) Are samples listed on a chain-of-custody record?
Comments: ________________________

Yes________ No________ 13) Is chain of custody documented and maintained?
Comments: ________________________

Yes________ No________ 14) Are quality assurance checks performed as directed?
Comments: ________________________

Yes__________ No__________ 15) Are photographs documented in logbooks as required?
Comments: ______________________________

Yes__________ No__________ 16) Are all documents accounted for?
Comments: ______________________________

Yes__________ No__________ 17) Have any documents been voided?
Comments: ______________________________

Yes__________ No__________ 18) Have any documents been destroyed?
Comments: ______________________________