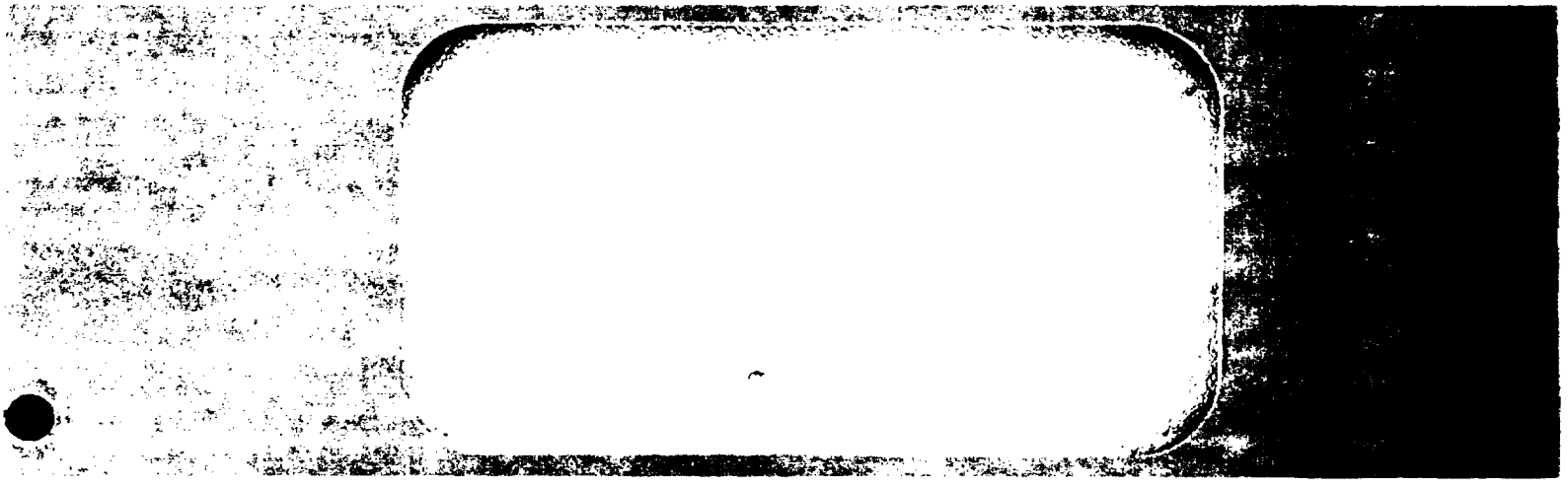
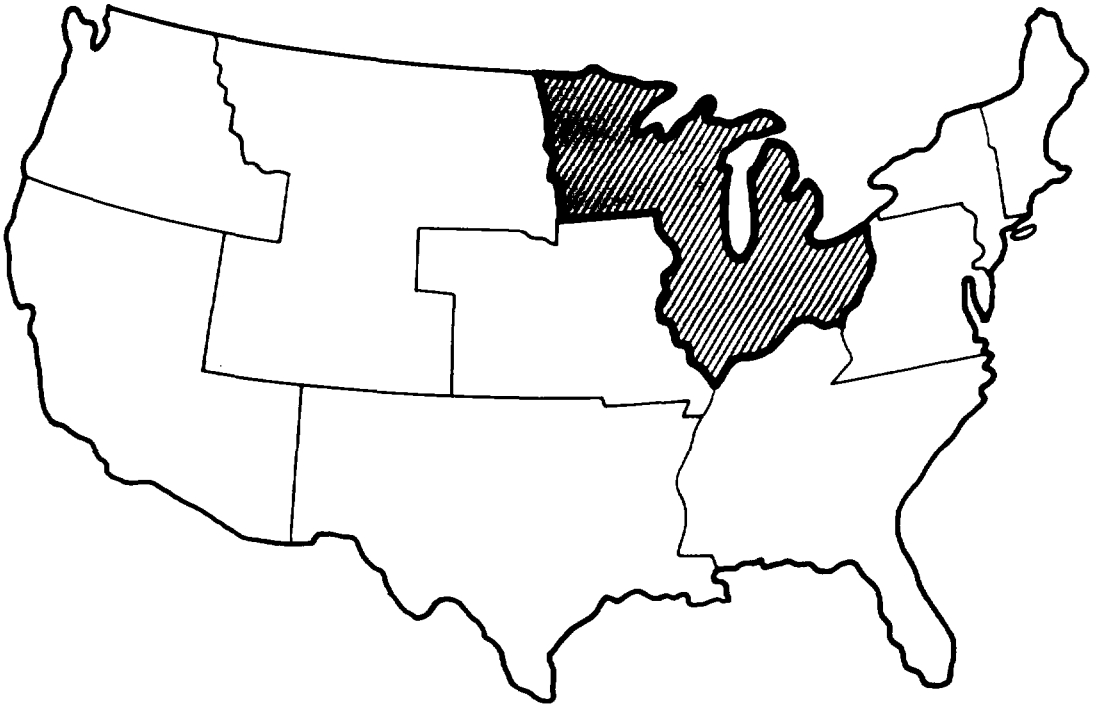
ARCS V

Remedial Activities at Uncontrolled Hazardous Waste Sites in Region V

EPA United States Environmental Protection Agency





CH2M HILL

Quality Assurance Project Plan

Kerr-McGee Kress Creek Site
West Chicago, Illinois

WA No. 62-5LQS
Contract No. 68-W8-0040

May 1993

Title Page
Quality Assurance Project Plan

Project Title: Kerr-McGee Kress Creek Site
West Chicago, Illinois

Work Assignment No.: 62-5LQS

EPA Contract No.: 68-W8-0040

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ACRONYMS AND INITIALISMS

AEA	Atomic Energy Act
AEC	Atomic Energy Commission
ARARs	applicable or relevant and appropriate requirements
ASL	Applied Science Laboratories
ASTM	American Society for Testing and Materials
CDO	Central District Office
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CLP	Contract Laboratory Program
CRL	Central Regional Laboratory
dbh	diameter, breadth, height
DI	deionized
DOE	Department of Energy
DOT	Department of Transportation
DQO	data quality objective
EDMS	Environmental Database Management System
EM	electromagnetic
EMSL	Environmental Monitoring Systems Laboratory
EP	Extraction Procedure
EPA	U. S. Environmental Protection Agency
FAA	furnace atomic adsorption
FCR	field change request
FS	Feasibility Study
FSP	Field Sampling Plan
GC/MS	gas chromatograph/mass spectrometer
HPLC	high-pressure liquid chromatography
ICP	inductively coupled plasma
I.D.	inside diameter
LSSS	Laboratory Scientific Support Section
MCL	maximum contaminant level
MS/MSD	matrix spike/matrix spike duplicate
NAREL	National Air and Radiation Environmental Laboratory
NEIC	National Enforcement Investigations Center
NGVD	National Geodetic Vertical Datum of 1929
NIST	National Institute of Standards and Technology
NPL	National Priorities List
NRC	Nuclear Regulatory Commission
OVA	organic vapor analyzer
PAH	polycyclic aromatic hydrocarbon
PARCC	precision, accuracy, representativeness, completeness, and comparability
PCB	polychlorinated biphenyl
PE	performance evaluation
PIC	pressurized ion chamber
%R	percent recovery
PRP	potentially responsible party
PVC	polyvinyl chloride
RPD	relative percent difference
QA	quality assurance
QAPjP	Quality Assurance Project Plan
QAS	Quality Assurance Section
QC	quality control
RAS	Routine Analytical Services

RI	Remedial Investigation
RI/FS	Remedial Investigation/Feasibility Study
RPD	relative percent difference
RSSI	Radiation Safety Services, Inc.
SAS	Special Analytical Services
SOP	Standard Operating Procedure
SOW	statement of work
SQL	standard query language
SVOC	semivolatile organic compound
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
TSP	trisodium phosphate
VOC	volatile organic compound

1.0 Introduction

The United States Environmental Protection Agency (EPA) requires that environmental monitoring and measurement efforts mandated or supported by EPA are controlled by a centrally managed quality assurance (QA) program.

Parties generating data under this program have to implement procedures so the precision, accuracy, representativeness, completeness, and comparability (PARCC) of its data are known and documented. To meet this objective, each party must prepare a written QA Project Plan (QAPjP) covering each project to be performed. All participants in the project, including subcontractors, will follow the procedures and protocols in this document.

This QAPjP presents the organization, objectives, functional activities, and specific QA and quality control (QC) activities associated with the Remedial Investigation/Feasibility Study (RI/FS) for the Kerr-McGee Kress Creek site. Activities at this site are under a fund-lead RI/FS. This QAPjP also describes the specific protocols that will be followed for sampling, sample handling and storage, chain of custody, and laboratory and field analysis.

QA/QC procedures will be in accordance with applicable technical standards, EPA requirements, government regulations and guidelines, and specific project goals and requirements. This QAPjP is prepared by CH2M HILL in accordance with EPA QAPjP guidance documents, in particular the Contract Laboratory Program (CLP) guidelines, Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans (QAMS-005/80), and the 1991 Region V Model QAPjP (EPA, 1991).

1.1 Project Description

Portions of the West Branch DuPage River and its tributary, Kress Creek, are located within the Kress Creek site (Figure 1-1). The Kress Creek site begins at the Kerr-McGee Rare Earths Facility, follows the storm sewer to the outfall, and extends down Kress Creek to its confluence with the West Branch DuPage River (approximately 1.4 miles). The site then extends down the West Branch DuPage River to Illinois Route 56 (approximately 2.6 miles). The site extends laterally to 400 ft on each side, incorporating the 100-year floodplain. These boundaries are preliminary definitions of the site. If contamination attributable to Kress Creek is found at the preliminary boundaries, the site boundaries will be expanded.

Kress Creek is located immediately south of the Kerr-McGee Rare Earths Facility and has received radiologically contaminated wastes via a storm sewer discharge from the Kerr-McGee Rare Earths Facility. The storm sewer carries surface drainage from the facility and surrounding areas and empties directly to the creek. It may have discharged process waste in the past.

The potential for offsite transport of materials from the Kerr-McGee Rare Earths Facility is largely a function of the geomorphological history of the Kress Creek drainage basin. The area is characterized by gently rolling topography common to northeastern Illinois. The soils are generally of glacial origin, ranging from tight clays to poorly graded sands, yet dominated by soils of low permeability. Urbanization has substantially and progressively changed the drainage pattern through increasing impervious coverings and the development of a grid of man-made channel storm sewers. This urbanization has resulted in an increase

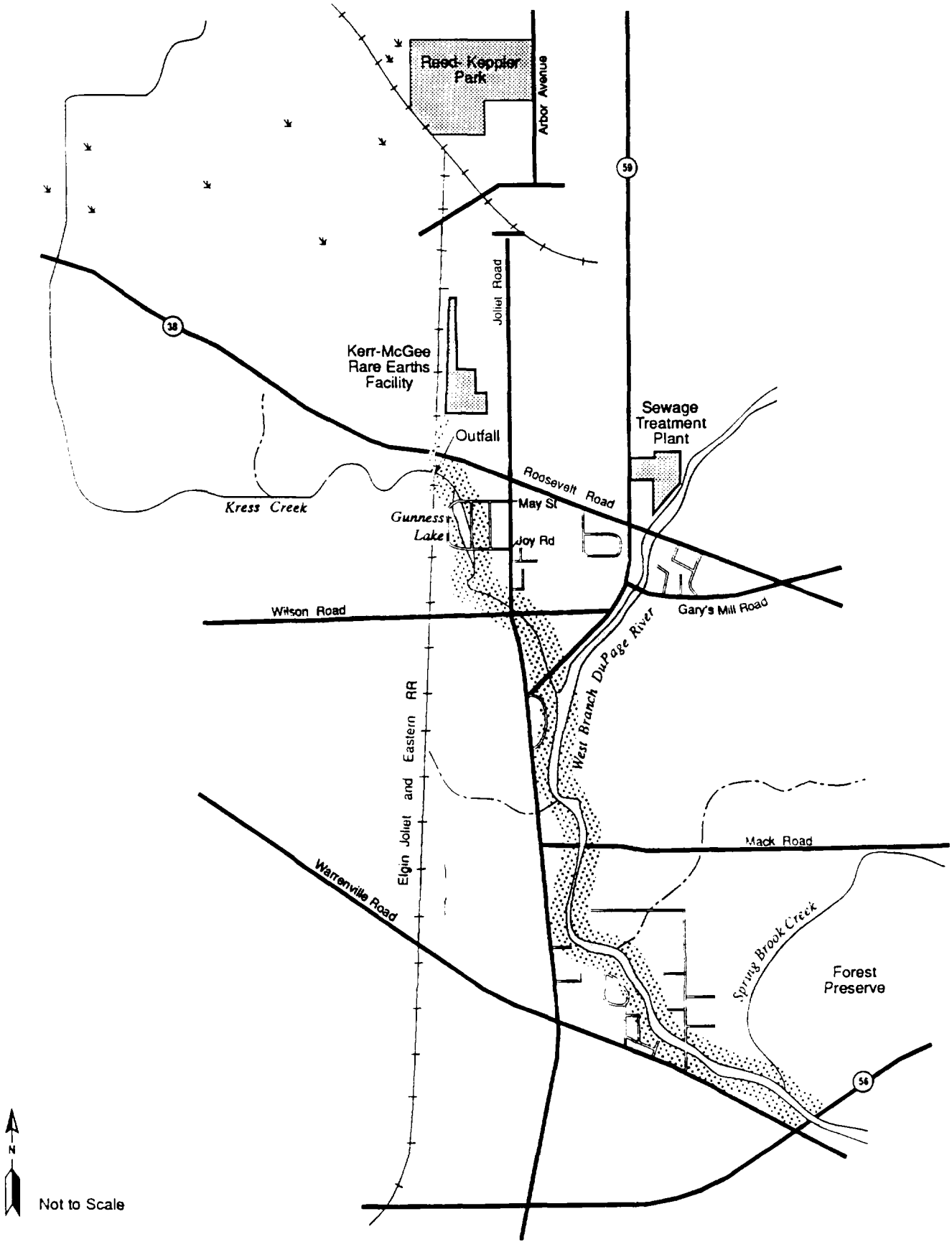


Figure 1-1
Site Location
Kress Creek RI/FS QAPjP

in the frequency of bank-full flood stages in Kress Creek with an attendant increase in the potential to transport pollutants. Urbanization is also characterized by periodic flushing and subsequent deposition of sediments from developing acreage. This recent and ongoing urbanization results in a stream, stream bed, and floodplain that is rapidly changing in a non-predictable fashion.

The Kerr-McGee Rare Earths Facility was originally established in 1932 by the Lindsay Light and Chemical Corporation. The facility extracted thorium from monazite sands (phosphates of cerium and lanthanum that usually contain thorium and uranium) and rare earth materials from bastnaesite ore. The refined thorium was used for both the manufacture of gas lantern mantles and by government and private commercial industries for atomic (nuclear) research projects. The rare earth products were used for optical polishing compounds, catalysts, and other industrial applications [Nuclear Regulatory Commission (NRC), 1986]. Ownership of the processing facility changed through corporate mergers from Lindsay Light and Chemical Company to American Potash and Chemical in 1958, and then to Kerr-McGee Chemical Corporation in 1967. The facility was closed by Kerr-McGee in 1973.

During the operational period from 1932 to 1973, various chemical processes were used to produce the thorium and rare earths compounds. Wastes from these processes included sands, sludges, and sediments derived from the ores. These materials were primarily stored at the facility; however, some process wastes, tailings, and radioactive ore were disposed of in various areas within West Chicago. Tailings were often used by local property owners as fill material in the early days of plant operation.

Production of thorium became subject to federal regulation with the passage of the Atomic Energy Act (AEA) in 1954. Consequently, a license was granted to the Lindsay Light and Chemical Company in 1956 by the U.S. Atomic Energy Commission (AEC). The license was transferred in accord with facility ownership to Kerr-McGee in 1967. The Energy Reorganization Act of 1974 transferred the licensing and regulatory authority from the AEC to the NRC. The NRC's authority was transferred to the State of Illinois effective November 1, 1990.

The Department of Energy (DOE), the NRC, the EPA, the State of Illinois, the City of West Chicago, and Kerr-McGee have conducted numerous studies and investigations to identify the areas of contamination and risks associated with the contamination. In August 1990, the Kress Creek site was placed on the National Priorities List (NPL) under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA).

1.2 Project Data Quality Objectives

The objectives of data collection are:

- To collect soil and sediment information to support a baseline human health risk assessment to assess whether direct exposure, dermal contact, or ingestion are significant pathways of concern. The conceptual site model and the preliminary risk assessment identify these as probable exposure pathways.

- To collect biota and habitat information to identify potential receptors to contamination and to identify impacts on the ecosystem from potential remediation, thus supporting the FS.

- To collect fish contaminant data to assess if the risks due to fish ingestion by humans is a probable condition.
- To collect data relevant to the nature (particle size, contaminant) and extent (including transportability) of soil and sediment contamination so an evaluation of remedial action alternatives can be made.
- To collect contaminant leachability information to assess if leaching of contaminants to surface water or underlying soils and groundwater is a probable condition.
- It is not the objective of this project to conduct a quantitative ecological risk assessment and evaluate cause and effect relationships between contaminants and species found to present.

1.3 Sample Network Design and Rationale

The sample network design and rationale for sample locations are described in detail in Section 2 of the Field Sampling Plan (FSP) in Appendix A.

1.4 Parameters To Be Tested and Frequency

Contaminants of concern were defined in the work plan as those most likely to contribute to risk as a result of exposure. For the work plan, contaminants of concern are identified as contaminants disposed at the Kerr-McGee Rare Earths Facility (Th-232, U-238); contaminants resulting from the decay of the disposed contaminants (Th-228, Th-230, Ra-226, Ra-228, U-234, Rn-220, and Rn-222); and other contaminants detected at significant concentrations during earlier investigations (lead). U-235 and Th-227 are considered reasonable deviations to the contaminants of concern. While most of the contaminants of concern are a result of thorium waste disposal, lead has been included because it has been detected on the facility and at the other Kerr-McGee sites. Ionizing radiation has also been included because it can be measured, although it is not technically a contaminant. Although not considered as likely, constituents on the Target Compound List (TCL) and other metals on the Target Analyte List (TAL) are potentially present from a variety of sources other than the Kerr-McGee Rare Earths Facility. Because radionuclides will contribute most significantly to the risk assessment and can be directly attributed to facility activities, radionuclides will be used to determine the nature and extent of contamination for the FS. Lead is also directly attributable to Kerr-McGee; however, its presence could be from other sources. The analysis of TCL will be limited to support consideration of disposal options in the FS.

Sample matrices, analytical parameters, and frequencies of sample collection are located in Table 1-1, along with proposed field analyses for each media. The numbers in the table do not include QC samples. Additional discussion of the types of samples to be collected is located in the FSP. Section 3.2 of the QAPjP specifically identifies the contaminants belonging to each of the contaminant suites identified in Table 1-1.

Table 1-1
Summary of Sample Collection and Analyses

Media	Analytes				
	Th, U, Ra*	TAL Metals	Full TCL	Leachability for TAL Metals and Radionuclides	Field Analyses
Fish	18	18	-	-	HNu/OVA*, beta/gamma detector*
Soil (Floodplain)	50	50	-	-	Radiological walkover survey, HNu/OVA*, beta/gamma survey
Sediment	79	79	6	3	HNu/OVA*, beta/gamma detector, geomorphologic analysis, particle-size fractioning
Surface Water	30	30 (and TSS, TDS)	-	-	HNu/OVA*, beta/gamma detector*, pH, temperature, dissolved oxygen, conductivity, and hardness
Flora/Fauna	-	-	-	-	Characterization, habitat assessment

*For health and safety purposes only.
*Includes U-234, U-235, U-238, Ra-226, Ra-228, Th 227, Th-228, Th 230, Th-232.

1.5 Analytical Data Quality Objectives

Data quality objectives (DQOs) are qualitative and quantitative statements that specify the quality of the data required to support decisions made during RI/FS activities; DQOs are based on the end uses of the data to be collected. As such, different data uses are associated with different levels of data quality. Five analytical levels address various data uses and the QA/QC effort and methods required to achieve the desired level of quality. These levels are as follows:

- Screening (DQO Level 1). This level provides the lowest data quality but the most rapid results. It is often used for health and safety monitoring at the site, preliminary comparison to applicable or relevant and appropriate requirements (ARARs), initial site characterization to locate areas for subsequent and more accurate analyses, and engineering screening of alternatives (bench-scale tests). Types of data include those generated onsite through the use of pH, conductivity, temperature, and dissolved oxygen (for surface water). Portions of the radiological walkover data also will be Level 1.

- Field Analyses (DQO Level 2). This level provides rapid results and more QC checks than DQO Level 1. Depending on the level of QC exercised, this level may include mobile lab- generated data. Level 2 data are anticipated to be collected for hardness from field analysis by a Hach test and for particle size fractioning from wet sieving in the field.

- Engineering (DQO Level 3). This level provides an intermediate level of data quality and is used for site characterization. Engineering analyses may include mobile lab-generated data and some analytical lab methods (e.g., laboratory data with quick turnaround used for screening but without full QC documentation). Radiological exposure rate data will have Level 3 QC along with surface water total suspended solids (TSS) and total dissolved solids (TDS).

- Confirmational (DQO Level 4). This level provides the highest level of data quality and is used for risk assessment, evaluation of remedial alternatives, and determination of potentially responsible party (PRP). These analyses use full CLP analytical and data validation procedures in accordance with EPA recognized protocol. Data received from the laboratories for this project will have Level 4 QC data packages.

- Non-Standard (DQO Level 5): This level refers to analyses by non-standard protocols, for example, when lower detection limits or analysis of an unusual chemical compound is required. For this project, the level of QC will be similar to DQO Level 4 data; however, because of the potential radioactivity of the samples, analyses are considered non-standard.

Table 1-2 provides a summary of the type of data to be collected, the use of the data, and the associated analytical level.

Table 1-2
Data Uses and Associated EPA Analytical Levels

Media	Sampling Method and Analytes	Data Use	Analytical Level*
Surface Water	Direct measurements (pH, temperature, conductance, dissolved oxygen)	Determine surface water quality for ecological habitat assessment.	1
	Field analysis (hardness)	Determine surface water quality for ecological habitat assessment.	2
	Sample collection (metals, radionuclides)	Evaluate releases to surface water to support human health risk assessment and evaluation of potential impacts to environment. Determine naturally occurring conditions.	5
	Sample collection (TSS, TDS)	Determine surface water quality for ecological habitat assessment.	5
Sediment	Sample collection (metals, radionuclides)	Support human health risk assessment and evaluation of potential impacts to environment. Evaluate nature and extent of sediment contamination. Determine naturally occurring conditions.	5
	Sample collection (TCL organics)	Support evaluation of disposal options in FS.	5
	Field particle size fractioning	Support transportability and treatability evaluation of sediments.	2
	Sample collection (leachability)	Support transportability and treatability evaluation of sediments.	5
Soils	Sample collection (metals, radionuclides)	Support baseline human health. Evaluate nature and extent of contamination to support FS. Determine naturally occurring conditions	5
	Radiological walkover (gamma) survey	Areal extent of contamination to support human health risk assessment and FS. - gamma scan survey (FS) - gamma-ray exposure rate (risk assessment)	 1 3
Fish	Sample collection (metals, radionuclides)	Evaluate uptake by fish for human health risk assessment.	5

*For laboratory analyses Level 5, QA/QC follows Level 4 protocol, except for TSS, TDS on surface water and leachability (including hazardous constituent analysis) on sediments – follows Level 3 protocol.

1.6 Project Schedule

Samples are planned to be collected beginning in May 1993 and completing in July 1993. Figure 1-2 presents the proposed schedule.

The schedule is based on receiving approval of the QAPjP by April 19, 1993.

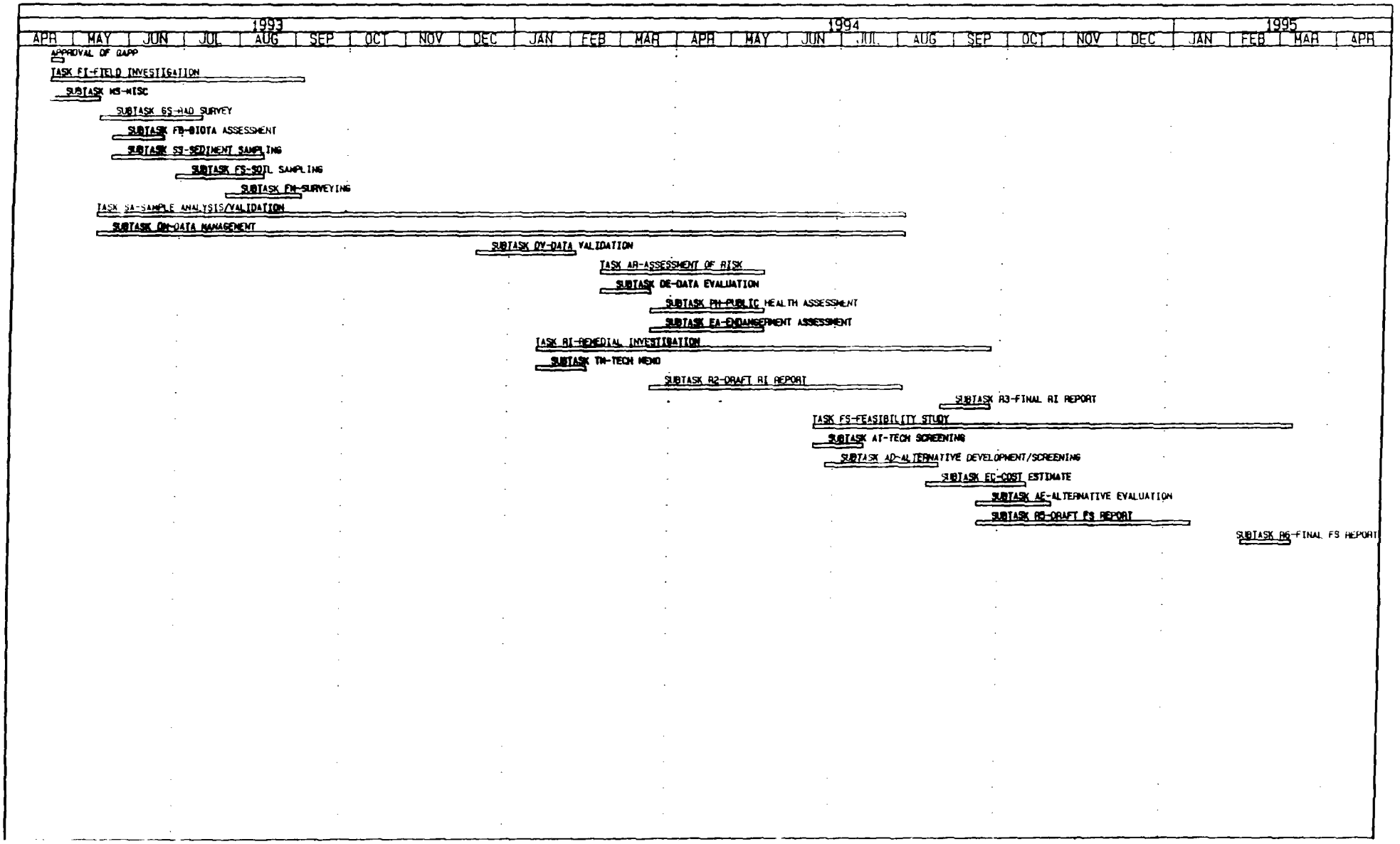


Figure 1-2
Schedule
Kress Creek RI/FS QAPjP

2.0 Project Organization and Responsibility

At the direction of the Remedial Project Manager, CH2M HILL has responsibility for conducting the RI/FS. CH2M HILL will perform the field investigation, prepare the RI report, and perform the subsequent FS. Project management will also be provided by CH2M HILL. The various QA and management responsibilities of key project personnel are defined below and shown in Figure 2-1.

EPA Region V Remedial Project Manager

The EPA Remedial Project Manager has the overall responsibility for each phase of the RI/FS.

CH2M HILL Program Manager

The Program Manager has overall responsibility for meeting EPA objectives and CH2M HILL quality standards. In addition, the program manager is responsible for technical quality control and project oversight.

QA Manager

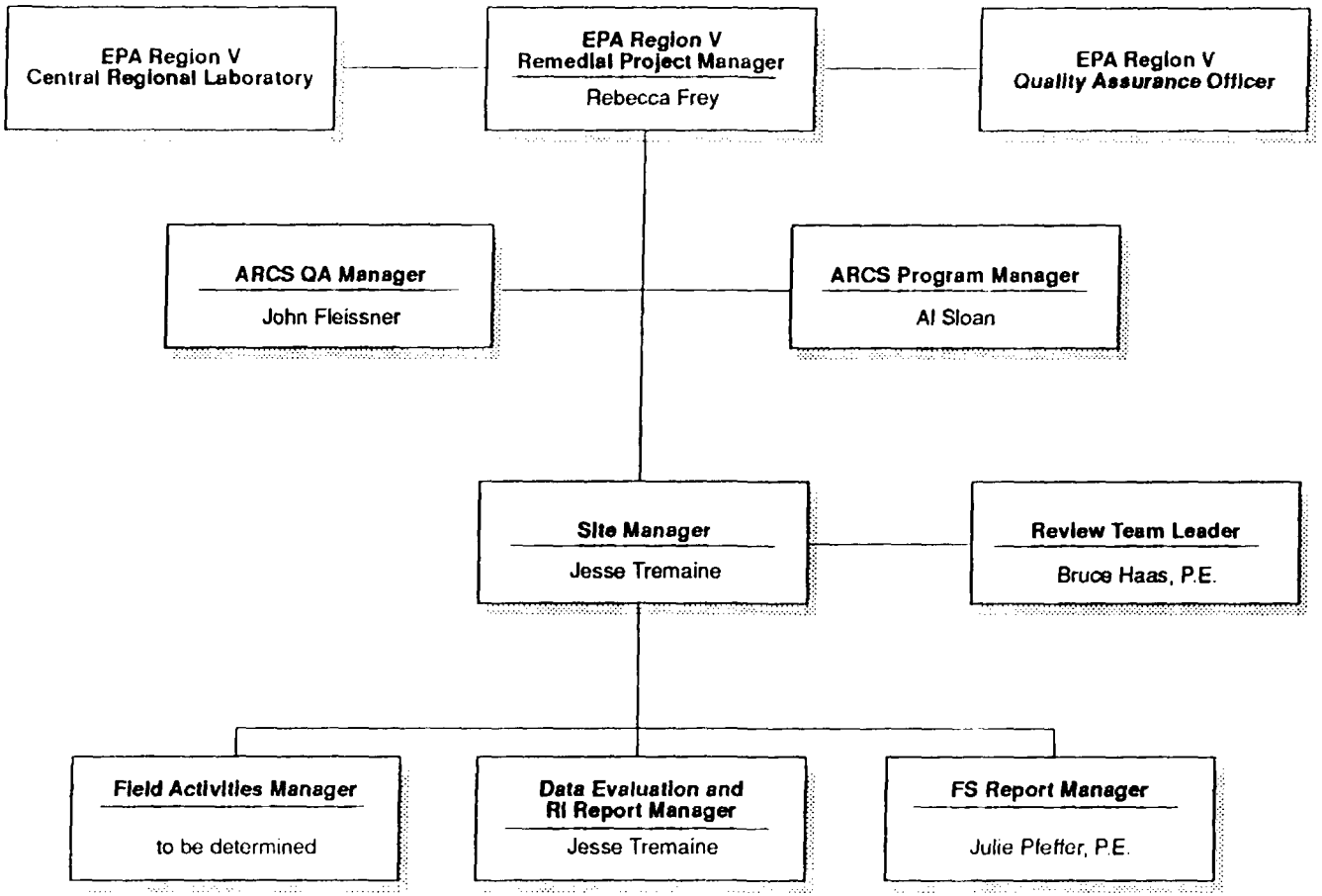
The QA Manager is John Fleissner. The QA Manager will remain independent of direct job involvement and day-to-day operations and has direct access to management staff to resolve QA disputes. Specific functions and duties include the following:

- Provide QA review of various phases of the project.
- Review QA plans and procedures.
- Provide QA technical assistance to project staff.
- Report the adequacy, status, and effectiveness of the QA program on a regular basis to the Program Manager and Site Manager.

CH2M HILL Site Manager

The Site Manager is responsible for implementing the project and is authorized to commit the resources to meet project objectives and requirements. The Site Manager's primary function is to achieve technical, financial, and scheduling objectives. The Site Manager will report directly to the EPA Region V Remedial Project Manager and will provide the major point of contact and control for matters concerning the project. More specifically, the Site Manager will:

- Define project objectives and develop a detailed work plan and schedule.
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task.



Technical Resources

George Stephens, C.H.P. — Health Physics
Nancy Schultz — Hydrologist
Steve Kucera — Risk Assessment
John Kubarewicz, P.E. — Chemical Engineer
Mike Mischuk — Aquatic Ecologist
Jack Dingledine — Terrestrial Ecologist

Figure 2-1
Project Organization
Kress Creek RI/FS QAPjP

- Acquire and apply technical and corporate resources to meet budget and schedule constraints.
- Orient field leaders and support staff with regard to the project's special considerations.
- Monitor and direct the field leaders.
- Develop and meet ongoing project or task staffing requirements, including mechanisms to review and evaluate each task product.
- Review the work performed on each task to provide quality, responsiveness, and timeliness.
- Review and analyze overall task performance with regard to planned schedule and budget.
- Review external reports (deliverables) before submission to EPA Region V.
- Accept responsibility for the preparation and quality of interim and final reports.
- Represent the project team at meetings and public hearings.

CH2M HILL Review Team Leader

The role of the Review Team Leader is to support the Site Manager in project management activities and to act as the coordinator of CH2M HILL internal reviews. The Review Team Leader will also be involved in the planning activities conducted at the beginning of and during the project.

CH2M HILL Field Activities Manager

The Site Manager will be supported by the Field Activities Manager. The Field Activities Manager is responsible for leading and coordinating day-to-day activities of the various field staff and will report directly to the Site Manager. Specific responsibilities of the Field Activities Manager include the following:

- Provide day-to-day coordination with the Site Manager.
- Implement field-related work plans.
- Coordinate and manage field staff, including sampling staff.
- Coordinate sample packaging and shipping.
- Implement QC for technical data provided by the field staff, including field measurement data.
- Adhere to work schedules provided by the Site Manager.

- Coordinate and provide oversight for technical efforts of subcontractors assisting the field team.
- Identify problems in the field and communicate between field staff and the Site Manager to resolve those problems.
- Prepare the field technical memorandums.
- Prepare QA audits on various phases of the field operations.

CH2M HILL Data Evaluation and RI Report Manager

The Data Evaluation and RI Report Manager will be responsible for tracking the data, overseeing the data evaluation, and writing the RI report. Specific responsibilities include the following:

- Oversee the tracking of samples and data from the time of field collection until results are placed in database storage.
- Coordinate activities with laboratories and data validators.
- Oversee production of data reports from the databases.
- Evaluate data usability.
- Coordinate the data interpretation.
- Oversee the risk assessment.
- Produce an RI report within the allotted time and budget constraints.

CH2M HILL FS Report Manager

The FS Report Manager is responsible for overseeing the development of remedial alternatives and the evaluation of the alternatives. Other duties include providing input to the data interpretation for analyses important to the FS.

CH2M HILL Technical Resources

The technical resources for this project will be drawn from CH2M HILL's corporate resources. The technical resources will be used to gather and analyze data and to prepare various task reports and support materials.

EPA Region V Quality Assurance Officer

The EPA Region V Quality Assurance Officer is responsible for review and approval of all QAPjPs.

Responsibilities for additional aspects of the project are as follows:

- **Scheduling of laboratory analyses**

  EPA Region V Central Regional Laboratory (CRL)
  EPA National Air and Radiation Environmental Laboratory (NAREL)

- **Data validation of chemical CLP Routine Analytical Services (RAS) and Special Analytical Services (SAS) results**

  EPA Region V CRL Laboratory Scientific Support Section (LSSS)

- **Data validation of radiological data**

  CH2M HILL

- **Preparation of SAS requests**

  CH2M HILL

- **Review and approval of CLP SAS**

  EPA Region V CRL LSSS
  EPA Region V QA Office

- **Performance and system audits of CLP laboratory**

  Analytical Operations Branch Office of Emergency and Remedial Response, EPA Headquarters
  EPA Region V CRL LSSS
  EPA Environmental Monitoring Systems Laboratory (EMSL) – Las Vegas
  EPA Sample Management Office

- **Review and approval of field and laboratory procedures**

  EPA Region V QA Section (QAS)
  EPA Remedial Project Manager

3.0 Quality Assurance Objectives for Measurement Data

The overall QA objective is to develop and implement procedures for field sampling, chain of custody, laboratory analysis, and reporting that will provide legally defensible results. Specific procedures for sampling, chain of custody, laboratory instruments calibration, laboratory analysis, data reporting, internal QC, audits, preventive maintenance for field equipment, and corrective action are described in other sections of this QAPjP. The purpose of this section is to address the specific objectives for PARCC. The methods for calculating the PARCC parameters are presented in Section 12.

3.1 Level of Quality Control Effort

Equipment rinseate blanks, duplicate, and matrix spike (MS) samples will be analyzed to assess the quality of the laboratory data of quality Level 4 or 5 resulting from the field sampling program. Duplicate measurements of DQO Level 3 field analysis will also be taken. Field QC measurements and samples will be taken or collected and prepared by the field team.

Equipment rinseate blanks are taken from water sampling equipment after decontamination to check for completeness of the decontamination process. The rinseate blank samples are taken every 10 or fewer investigative samples. Field duplicate samples are analyzed to check for sampling and analytical reproducibility. The field duplicates will be taken every 10 or fewer investigative samples. Duplicate exposure rate readings will also be taken every 10 or fewer investigative readings.

MSs provide information about the effect of the sample matrix on the digestion and measurement methodology. MSs are performed in duplicate and are hereinafter referred to as MS/matrix spike duplicate (MSD) samples. These samples are considered laboratory QC samples. Volume for one MS/MSD sample will be collected for every 20 or fewer investigative samples or every 14 days by the field team. MS/MSD samples are analyzed for the TCL and radionuclide analyses. Soil and sediment MS/MSD samples use no extra volume. However, radionuclides and TCL organics [except volatile organic compounds (VOCs)], MS/MSD samples must be collected at double the volume. MS/MSD samples for VOCs are collected at triple the volume.

The number of QC samples to be collected are listed in Table 3-1. Sampling procedures are specified in the FSP (Appendix A).

3.2 Analytical Sensitivity (Detection Limits)

The analytical procedures specified in Section 7.0 have been selected on the basis of their ability to meet identified detection limits. Suitable detection limits were identified from the use of the data. Risk-based levels and potential cleanup criteria have been identified for the contaminants of concern. These levels were compared with the detection limits provided by various analytical methods. The method that met the detection limit was selected. The various risk-based levels, cleanup levels, and selected detection levels and analytical methods for the contaminants of concern (radionuclides and lead) are presented in Table 3-2. The sensitivities (detection limits) for the organic and inorganic compounds, which also will be analyzed, are presented in Tables 3-3, 3-4, and 3-5.

Table 3-1
Summary Table of Sampling and Analysis Program

Sample Matrix	Field Parameter	Laboratory Parameter	Investigative Analyses	Field QC Samples			Matrix Total[b]
				Field Duplicate	Rinseate Blanks	MS/MSD[a]	
			No.	No.	No.	No.	
Soil (Floodplain)	HNu/OVA[c], beta/gamma, radiological walkover survey	TAL Metals	50	5	NA	NA	55
		Radionuclides	50	5	NA	3	58
Sediments	HNu/OVA[c], beta/gamma, particle size fractioning (24)	TAL Metals[d]	79	6	NA	NA	85
		Radionuclides[d]	79	6	NA	3	88
		TCL Organics	6	1	NA	1	8
Surface Water	pH, temperature, dissolved oxygen, conductance, hardness, HNu/OVA[c], beta/gamma[c]	Radionuclides	30	3	3	2	38
		TAL Metals	30	3	3	NA	36
		TSS/TDS	18	2	NA	NA	19
Fish Tissue	HNu/OVA[c], beta/gamma[c]	Radionuclides	18	NA	NA	1	19
		TAL Metals	18	NA	NA	NA	18
Investigation-Derived Wastewater		Radionuclides	2	NA	NA	NA	2
		TAL Metals	2	NA	NA	NA	2

[a]Matrix spike/matrix spike duplicate (MS/MSD) samples are required with the organic and radiological analyses. Samples designated for MS/MSD analyses will be collected at a frequency of one per group of 20 or fewer investigative samples per matrix. The following sample volumes will be required: double the volume for water radiological analyses and TCL, except VOCs; triple the volume for VOCs; and no additional volume for solid samples.

[b]The matrix total includes all investigative and field QC samples.

[c]For health and safety purposes only.

[d]Field QC not applied to 24 analyses associated with particle size fractioning.

NA = Not Applicable.

Table 3-2
Expected Analytical Detection Limits

Contaminant	Limiting Concentration for Residential Scenario[a]				Expected Background Concentration Ranges		Regulatory Criteria		Proposed Minimum Detection Levels	
	Soil/Sediment (pCi/g)		Water (pCi/L)							
	10^{-4}	10^{-6}	10^{-4}	10^{-6}	Soil/Sediment (pCi/g)	Water[b] (pCi/L)	Soil/Sediment (pCi/g)	Water (pCi/L)	Soil/Sediment/Fish[c] (pCi/g)	Water (pCi/L)
Ra-226	0.3	ND	40	ND	1.2[d]	0.4	5[e]	20[f]	0.5	1.0
Ra-228	1.4	ND	48	ND	2.3[d]	0.7	5[e]	20[f]	0.5	1.0
Th-227	26	ND	1,060	11	<1	<1	NA	400[f]	0.5	1.0
Th-228	0.7	ND	87	ND	2.3[d]	<1	NA	125[f]	0.5	1.0
Th-230	5,500	55	366	3.7	<1	<1	5[e]	79[f]	0.5	1.0
Th-232	6,200	62	400	4.0	2.3[d]	<1	5[e]	88[f]	0.5	1.0
U-234	4,600	46	300	3.0	1.3[h]	0.1-20	5[i]	j	0.5	1.0
U-235	17	ND	300	3.0	<1	<1	NA	j	0.5	1.0
U-238	110	1.1	170	1.7	1.3[g]	0.1-20	5[h]	j	0.5	1.0
Lead	NA	NA	NA	NA	NA	NA	NA	15[l] 338[k] µg/l	l	3 µg/l

[a]Concentrations of radionuclides corresponding to risk levels of 10^{-6} and 10^{-4} were determined using the residential scenario outlined in RAGs Part B. Risk factors were taken from the January 1992 HELP/AST tables.
[b]Concentrations shown for background ranges were taken from supporting documentation for FR 56 No. 138, 40 CFR Parts 141, 142, National Primary Drinking Water Regulations, Radionuclides Notice of Proposed Rulemaking, July 18, 1991.
[c]Soil and sediment concentration results expressed as pCi/g dry weight. Fish concentration results expressed as pCi/g wet (or fresh) weight.
[d]From NARL/UCR-0035, "Radiological Survey of the Reed Keppler Park Site, West Chicago, Illinois."
[e]Illinois Department of Nuclear Safety (IDNS) radiation protection regulations in Part 332, "Licensing Requirements for Source Material Milling Facilities." Specify concentration limits for total radium (above background) of 5 pCi/g of dry soil averaged over the first 15 cm below the surface, and 15 pCi/g of dry soil averaged over layers of 15-cm thickness thereafter. Activities are averaged over areas of 100 m^2.
[f]FR 56 No. 138 proposed revision to 40 CFR 141 and 142. Concentrations shown for thorium isotopes are based on a 10^{-4} lifetime (70-year) cancer incidence.
[g]From DOE/CH/8901, "A Manual for Implementing Residual Radioactive Material Guidelines," Appendix A: DOE Guidelines for Residual Radioactive Material (shown for reference).
[h]Eisenbud, Merril, Environmental Radioactivity from Natural Industrial and Military Sources, 1987. Average uranium concentration for igneous rock shown for reference.
[i]Federal Register 1981, Volume 46, No. 205, 10/23, 81, Nuclear Regulatory Commission, "Disposal or Onsite Storage of Uranium Wastes from Past Operations" (most restrictive limit is 10 pCi/g of U-234 plus U-128, 5 pCi/g each, assuming natural uranium).
[j]The proposed MCL for uranium is 20 µg/l (total). This is approximately 13 pCi/l for natural uranium (U-234 to U-238 activity ratio of 1.0). This MCL applies to the total activity of uranium.
[k]Safe Drinking Water Act, Trigger Level for Additional Monitoring, 56 FR 26460 (June 7, 1991).
[l]Ambient Water Quality Criteria for the Protection of Aquatic Organisms. Source: EPA, 1991. EPA Region IV Criteria Chart (October). Water hardness dependent (assumes 50 µg/l as $CaCO_3$).
[m]Matrix dependent.

Notes
NA = Not Available
ND = Not Detectable

Table 3-3
Target Compound List and Contract Required Quantitation Limits

Organic Analytes	CAS Number	Quantitation Limits[a]			
		Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)	On Column (ng)
1. Chloromethane	74-87-3	10	10	1200	(50)
2. Bromomethane	74-83-9	10	10	1200	(50)
3. Vinyl Chloride	75-01-4	10	10	1200	(50)
4. Chloroethane	75-00-3	10	10	1200	(50)
5. Methylene Chloride	75-09-2	10	10	1200	(50)
6. Acetone	67-64-1	10	10	1200	(50)
7. Carbon Disulfide	75-15-0	10	10	1200	(50)
8. 1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9. 1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10. 1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11. Chloroform	67-66-3	10	10	1200	(50)
12. 1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13. 2-Butanone	78-93-3	10	10	1200	(50)
14. 1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15. Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16. Bromodichloromethane	75-27-4	10	10	1200	(50)
17. 1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18. cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19. Trichloroethene	79-01-6	10	10	1200	(50)
20. Dibromochloromethane	124-48-1	10	10	1200	(50)
21. 1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22. Benzene	71-43-2	10	10	1200	(50)
23. trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24. Bromoform	75-25-2	10	10	1200	(50)
25. 4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26. 2-Hexanone	591-78-6	10	10	1200	(50)
27. Tetrachloroethene	127-18-4	10	10	1200	(50)
28. Toluene	108-88-3	10	10	1200	(50)
29. 1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
30. Chlorobenzene	108-90-7	10	10	1200	(50)

Table 3-3
Target Compound List and Contract Required Quantitation Limits

	Organic Analytes	CAS Number	Quantitation Limits[a] Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)	On Column (ng)
31.	Ethyl benzene	100-41-4	10	10	1200	(50)
32.	Styrene	100-42-5	10	10	1200	(50)
33.	Xylenes (total)	1330-20-7	10	10	1200	(50)
34.	Phenol	108-95-2	10	330	10000	(20)
35.	bis(2-Chloroethyl) ether	111-44-4	10	330	10000	(20)
36.	2-Chlorophenol	95-57-8	10	330	10000	(20)
37.	1,3-Dichlorobenzene	541-73-1	10	330	10000	(20)
38.	1,4-Dichlorobenzene	106-46-7	10	330	10000	(20)
39.	1,2-Dichlorobenzene	95-50-1	10	330	10000	(20)
40.	2-Methylphenol	95-48-7	10	330	10000	(20)
41.	2,2'-oxybis-(1-Chloropropane)[b]	108-60-1	10	330	10000	(20)
42.	4-Methylphenol	106-44-5	10	330	10000	(20)
43.	N-Nitroso-di-n-dipropylamine	621-64-7	10	330	10000	(20)
44.	Hexachloroethane	67-72-1	10	330	10000	(20)
45.	Nitrobenzene	98-95-3	10	330	10000	(20)
46.	Isophorone	78-59-1	10	330	10000	(20)
47.	2-Nitrophenol	88-75-5	10	330	10000	(20)
48.	2,4-Dimethylphenol	105-67-9	10	330	10000	(20)
49.	bis(2-Chloroethoxy)methane	111-91-1	10	330	10000	(20)
50.	2,4-Dichlorophenol	120-83-2	10	330	10000	(20)
51.	1,2,4-Trichlorobenzene	120-82-1	10	330	10000	(20)
52.	Naphthalene	91-20-3	10	330	10000	(20)
53.	4-Chloroaniline	106-47-8	10	330	10000	(20)
54.	Hexachlorobutadiene	87-68-3	10	330	10000	(20)
55.	4-Chloro-3-methylphenol	59-50-7	10	330	10000	(20)
56.	2-Methylnaphthalene	91-57-6	10	330	10000	(20)
57.	Hexachlorocyclopentadiene	77-47-4	10	330	10000	(20)
58.	2,4,6-Trichlorophenol	88-06-2	10	330	10000	(20)
59.	2,4,5-Trichlorophenol	95-95-4	25	800	50000	(100)
60.	2-Chloronapthalene	91-58-7	10	330	10000	(20)

Table 3-3
Target Compound List and Contract Required Quantitation Limits

Organic Analytes		CAS Number	Quantitation Limits[a]			
			Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)	On Column (ng)
61.	2-Nitroaniline	88-74-4	25	800	50000	(100)
62.	Dimethylphthalate	131-11-3	10	330	10000	(20)
63.	Acenaphthylene	208-96-8	10	330	10000	(20)
64.	2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
65.	3-Nitroaniline	99-09-2	25	800	50000	(100)
66.	Acenaphthene	83-32-9	10	330	10000	(20)
67.	2,4-Dinitrophenol	51-28-5	25	800	50000	(100)
68.	4-Nitrophenol	100-02-7	25	800	50000	(100)
69.	Dibenzofuran	132-64-9	10	330	10000	(20)
70.	2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71.	Diethylphthalate	84-66-2	10	330	10000	(20)
72.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73.	Fluorene	86-73-7	10	330	10000	(20)
74.	4-Nitroaniline	100-01-6	25	800	50000	(50)
75.	4,6-Dinitro-2-methylphenol	534-52-1	25	800	50000	(50)
76.	N-Nitrosodiphenylamine	86-30-6	10	330	10000	(20)
77.	4-Bromophenyl-phenyl ether	101-55-3	10	330	10000	(20)
78.	Hexachlorobenzene	118-74-1	10	330	10000	(20)
79.	Pentachlorophenol	87-86-5	25	800	50000	(50)
80.	Phenanthrene	85-01-8	10	330	10000	(20)
81.	Anthracene	120-12-7	10	330	10000	(20)
82.	Carbazole	86-74-8	10	330	10000	(20)
83.	Di-n-butylphthalate	86-74-2	10	330	10000	(20)
84.	Fluoranthene	206-44-0	10	330	10000	(20)
85.	Pyrene	129-00-0	10	330	10000	(20)
86.	Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87.	3,3-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88.	Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89.	Chrysene	210-81-9	10	330	10000	(20)
90.	bis(2-Ethylhexyl)phthalate	117-81-7	10	330	10000	(20)

Table 3-3
Target Compound List and Contract Required Quantitation Limits

	Organic Analytes	CAS Number	Quantitation Limits[a]			
			Water (µg/L)	Low Soil (µg/kg)	Med. Soil (µg/kg)	On Column (ng)
91.	Di-n-Octylphthalate	117-84-0	10	330	10000	(20)
92.	Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93.	Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)
94.	Benzo(a)pyrene	50-32-8	10	330	10000	(20)
95.	Indeno(1,2,3-cd)pyrene	193-39-5	10	330	10000	(20)
96.	Dibenzo(a,h)anthracene	53-70-3	10	330	10000	(20)
97.	Benzo(g,h,i)perylene	191-24-2	10	330	10000	(20)

[a]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.
[b]Previously known by the name of bis (2-chloroisopropyl) ether.

Table 3-4
PCBs and Pesticide Contract Required Quantitation Limits

	Pesticides/Aroclors	CAS Number	Quantitation Limits		
			Water (µg/L)	Soil (µg/kg)	On Column (ng)
1.	alpha-BHC	319-84-6	0.05	1.7	5
2.	beta-BHC	319-85-7	0.05	1.7	5
3.	delta-BHC	319-86-8	0.05	1.7	5
4.	gamma-BHC (Lindane)	58-89-9	0.05	1.7	5
5.	Heptachlor	76-44-8	0.05	1.7	5
6.	Aldrin	309-00-2	0.05	1.7	5
7.	Heptachlor epoxide	1024-57-3	0.05	1.7	5
8.	Endosulfan I	959-98-8	0.05	1.7	5
9.	Dieldrin	60-57-1	0.10	3.3	10
10.	4,4´-DDE	72-55-9	0.10	3.3	10
11.	Endrin	72-20-8	0.10	3.3	10
12.	Endosulfan II	33213-65-9	0.10	3.3	10
13.	4,4´-DDD	72-54-8	0.10	3.3	10
14.	Endosulfan sulfate	1031-07-8	0.10	3.3	10
15.	4,4´-DDT	50-29-3	0.10	3.3	10
16.	Methoxychlor	72-43-5	0.50	17.0	50
17.	Endrin ketone	53494-70-5	0.10	3.3	10
18.	Endrin aldehyde	7421-36-3	0.10	3.3	10
19.	alpha-chlordane	5103-71-9	0.05	1.7	5
20.	gamma-chlordane	5103-74-2	0.05	1.7	5
21.	Toxaphene	8001-35-2	5.0	170.0	500
22.	Aroclor-1016	12674-11-2	1.0	33.0	100
23.	Aroclor-1221	11104-28-2	1.0	67.0	200
24.	Aroclor-1232	11141-16-5	2.0	33.0	100
25.	Aroclor-1242	53469-21-9	1.0	33.0	100
26.	Aroclor-1248	12672-29-6	1.0	33.0	100
27.	Aroclor-1254	11097-69-1	1.0	33.0	100
28.	Aroclor-1260	11096-82-5	1.0	33.0	100

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.

There is no differentiation between the preparation of low and medium samples in this method for the analysis of pesticides/aroclors.

Table 3-5
Inorganic Target Analyte List and Contract Required Detection Limit

Analyte	Contract Required Detection Limit[a,b] (µg/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20

[a]Subject to the restrictions specified in the first page of Part G, Section IV of Exhibit D (Alternate Methods–Catastrophic Failure) any analytical method specified in SOW Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit requirements. Higher detection limits may only be used in the following circumstances:

If the sample concentration exceeds five times the detection limit of the instrument or method in use, the values may be reported even though the instrument or method detection limit may not equal the Contract Required Detection Limit. This is illustrated in the example below:

For lead:
Method in use = ICP
Instrument Detection Limit = 40
Sample concentration = 200
Contract Required Detection Limit = 3
The value of 200 may be reported even though instrument detection limit is greater than the Contract Required Detection Limit. The instrument or method detection limit must be documented.

[b]The Contract Required Detection Limits are the instrument detection limits obtained in pure water that must be met using the procedure in Exhibit E of the SOW. The detection limits for samples may be considerably higher depending on the sample matrix.

3.3 Accuracy and Precision of Analysis

The QA objective for accuracy and precision of laboratory analytical data is to achieve the QC acceptance criteria of the analytical protocols. The procedure for determining accuracy and precision is provided in Section 12, while the requirements for handling data outside the acceptable limits are provided in Section 9.4.

The accuracy and precision requirements for RAS from the CLP are specified in the current Statement of Work (SOW)/OLM01.8 for organics and SOW/ILM02.1 for inorganics. The accuracy and precision requirements for SAS for the CLP are specified in each individual SAS request contained in Appendix C. Table 3-6 shows the projected accuracy, precision, and completeness for radionuclide analyses. The Standard Operating Procedures (SOPs) for the field equipment to measure pH, conductivity, gamma and alpha emissions, dissolved oxygen, and temperature are outlined in Appendix B. Also included is the procedure for field analysis of hardness. Accuracy and precision requirements, as appropriate, for field analyses are included in the SOPs.

3.4 Completeness, Representativeness, and Comparability

Completeness is a measure of the amount of valid data (not rejected by validation) obtained from a measurement system compared to the amount that was expected to be obtained under normal conditions. It is expected that the CLP laboratory will provide data that meet QC acceptance criteria of 95 percent completeness or more for samples tested using the RAS methods. It is also expected that NAREL will provide data that meet QC acceptance criteria of 90 percent or more for radiological data. All valid data will be used to meet project objectives. If the completeness goals are not met, the need for additional sampling will be determined in discussions with EPA.

Representativeness expresses the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Representativeness is a qualitative parameter that is dependent on the design of the sampling program and the laboratory protocol. The sampling network was designed to provide data that are representative of site conditions. During development of this network, consideration was given to past waste disposal practices, existing analytical data, physical setting, and constraints inherent to the Superfund program. The rationale of the sampling network is discussed in detail in the FSP. Representativeness will be satisfied by following the FSP, using specified sampling techniques, following specified analytical procedures, and staying within holding times of the samples.

Comparability expresses the confidence with which one data set can be compared to another. The extent to which existing and planned analytical data will be comparable depends on the similarity of sampling and analytical methods. The procedures used to obtain the planned analytical data, as documented in the QAPjP, are expected to provide comparable data. These new analytical data, however, may not be directly comparable to existing data because of difference in procedures and QA objectives.

Table 3-6
Radionuclide QA Objectives

Parameter	Method	Accuracy	Recovery	Precision (Relative Percent Difference)	Completeness (Percent)
Uranium Isotopes	OO-07 Water OO-05, OO-06 Solids	30 to 105%	3σ	20% Water 35% Solid	90
Thorium Isotopes	OO-07 Water OO-05, OO-06 Solids	20 to 105%	3σ	20% Water 35% Solid	90
Ra-226	Ra-03, Ra-04 Water Ra-01, Ra-04 Solids	NA	3σ	20% Water 35% Solid	90
Ra-228	Ra-03, Ra-04 Water Ra-01, Ra-04 Solids	20 to 105%	3σ	20% Water 35% Solid	90

Note: σ is the standard deviation of the sample count rate (counting error) as supplied by the laboratory.

4.0 Sampling Procedures

Sampling procedures are described in the FSP, Appendix A.

5.0 Sampling Custody

It is EPA Region V's policy to follow the sample custody or chain-of-custody protocols described in *NEIC Policies and Procedures*, EPA-330/9-78DDI-R, Revised June 1985. Custody occurs during sample collection, laboratory analysis, and final evidence filing.

A sample is under custody if it is in the sampler's or chemist's possession; in the sampler's or chemist's view, after being in the sampler's or chemist's possession; in the sampler's or chemist's possession and then placed in a designated location; or in a designated storage area.

Final evidence files, including originals of laboratory reports and purge files, are maintained under document control in a specified area.

5.1 Field Chain-of-Custody Procedures

The sample packaging and shipment procedures summarized below will allow the samples to arrive at the laboratory with the chain of custody intact and then allows the sample to be shipped with proper custody procedures from the laboratories to the specified storage facility described in Appendix E. The protocols for specific sample numbering using case numbers and traffic report numbers (if applicable) and other sample designations are included in Section 3.0 of the FSP, Appendix A of the QAPjP.

5.1.1 Field Procedures

The field sampler is personally responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible should handle the samples.

Bottles will be tagged with sample numbers and locations. The Sample Management Office number and stickers will be affixed. Unless inhibited by weather conditions, waterproof ink should be used to complete the sample tags for each sample. For example, a logbook notation would explain that a pencil was used to complete the sample tag because the ballpoint pen would not function in freezing weather.

The Remedial Project Manager will review field activities to evaluate whether custody procedures were followed during the fieldwork and decide whether additional samples are to be taken.

5.1.2 Field Logbooks and Documentation

Field logbooks will provide the means for recording data collection activities. As such, entries will be described in as much detail as possible so that persons going to the site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound, field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a document control center when not in use. Each logbook will be identified by the project-specific document number.

The title page of each logbook will contain the following information:

- Project name and EPA Work Assignment Number
- Project number
- Site Manager's name
- Sequential logbook number
- Project start date
- End date

Entries into the logbook will contain a variety of information. The date, start time, weather conditions, names of sampling team members present, level of personal protection being used, and the signature of the person making the entry will be logged at the beginning of each entry. The names of visitors to the site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected, or a measurement is made, a description of the location of the station, which includes compass and distance measurements, will be recorded. Photographs that are taken of the station will also be noted. Equipment used to make field measurements and the date of calibration will be identified.

Samples will be collected following the sampling procedures documented in the FSPs. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, and volume and number of containers. Sample identification numbers will be assigned during sample collection. Field duplicate samples, which will receive a separate sample identification number, will be noted under sample description.

5.1.3 Transfer of Custody and Shipment Procedures

(a) Samples are accompanied by a completed chain-of-custody form. The sample numbers and locations will be listed on the chain-of-custody form. A new custody record will be used to track the sample residuals to the interim storage area. The original custody record will accompany the data results after analysis. The individuals who relinquish and receive the samples during transfer will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the permanent laboratory, or to and from a storage area.

(b) Samples will be packaged for shipment and dispatched to the appropriate laboratory for analysis, with a separate signed custody record enclosed in each sample box or cooler. A new custody record will be used to track the sample residuals to the interim storage area. The original custody record will accompany the data results after analysis. Shipping containers will be secured with strapping tape and EPA custody seals for shipment to the laboratory. The preferred procedure includes use of a custody seal attached to the front right and back left of the cooler. The custody seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

(c) When samples are co-located with a source or government agency, a separate Sample Receipt is prepared for those samples and marked to indicate the source or government agency where the samples are being co-located. The person relinquishing the samples to the facility or agency will request a representative's signature to acknowledge sample receipt. If the representative is unavailable or refuses to sign, notation is made in the "Received By" space.

(d) Shipments will be accompanied by the Chain-of-Custody Record that identifies the contents. The original record will accompany the shipment, and the pink and yellow copies will be retained by the sampler for return to the sampling office.

(e) If the samples are sent by commercial carrier, a bill of lading will be used. Receipts for bills of lading will be retained as part of the permanent documentation. If sent by mail, the package will be registered with return receipt requested. Commercial carriers are not required to sign off on the custody form as long as the custody forms are sealed inside the sample cooler and the custody seals remain intact.

(f) Samples will be sent to the laboratories for analysis the same day they are collected.

5.2 Laboratory Chain-of-Custody Procedures

The chain-of-custody procedures for the CLP laboratory are described in the SOWs for RASs. The same custody procedure applies to SASs. These custody procedures are described in the appropriate SOW (OLM01.8 for organics and ILM02.1 for inorganics).

5.3 Final Evidence Files Custody Procedures

CH2M HILL is the custodian of the evidence file and maintains the contents of evidence files for the RI, including relevant records, reports, logs, field logbooks, photographs, subcontractor reports, correspondence, and chain-of-custody forms under custody of the Site Manager.

6.0 Calibration Procedures and Frequency

This section describes procedures for maintaining the accuracy of instruments and measuring equipment that are used for conducting field tests and laboratory analyses. These instruments and equipment will be calibrated before each use or on a scheduled, periodic basis.

6.1 Field Instruments and Equipment

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated with sufficient frequency and in such a manner that accuracy and reproducibility of results are consistent with the manufacturer's specifications.

Equipment to be used for field sampling will be examined to evaluate its operating condition. This includes checking the manufacturer's operating manual and the instructions for each instrument to identify maintenance requirements. Field notes from previous sampling trips will be reviewed for indication that prior equipment problems have not been overlooked and that necessary repairs to equipment have been carried out. A spare electrode will be sent with each pH/temperature meter to be used for field measurements. Two thermometers will be sent to sampling locations where measurement of temperature is to occur, including those locations where a specific conductance probe thermometer is to be used.

Calibration of field instruments will be performed at the intervals specified by the manufacturer or more frequently as conditions dictate. Field instruments will include a pH/temperature meter, dissolved oxygen meter, specific conductivity meter, Organic Vapor Analyzer (OVA), HNu, gamma scintillation detectors, pressurized ion chamber, Geiger-Mueller detectors, and alpha scintillation detectors. In the event that an internally calibrated field instrument fails to meet calibration or checkout procedures, it will be returned to the manufacturer for service.

Calibration of field instruments is governed by the specific SOP for the applicable field analysis method. Such procedures take precedence over the following general discussion.

6.1.1 pH/Temperature Meter Calibration

The pH/temperature meter will be calibrated with standard buffer solutions prior to the field trip. In the field, the meter will be calibrated daily with two buffers before use. Calibration procedures and frequency will be recorded in a field logbook along with the lot numbers of the buffer. The pH meter must be recalibrated every time it is turned off and turned back on, or if it gives erratic results. The calibrations performed, standard used, and sample pH values recorded are to be entered in the field logbook. Appropriate new batteries will be purchased and kept with the meters to facilitate replacement in the field. Details of the calibration are provided in the field procedures.

6.1.2 Dissolved Oxygen Meter Calibration

The dissolved oxygen meters are calibrated at the equipment pool center shortly before use in the field. The Winkler filtration method, as specified in *Standard Method for the Examination of Water and Wastewater*, 14th ed. (1985), is used. The meters should not be shut off during the monitoring event.

6.1.3 Conductivity Meter Calibration

The conductivity cells of the specific conductivity meter will be cleaned and checked against known conductivity standards before each field trip. In the field, the instrument will be calibrated daily with standard calibration solution and a temperature vs conductivity calibration chart, as described in the field procedures.

The calibrations performed, standard used, and sample conductivity values recorded are to be entered in the field logbook. If appropriate, new batteries will be purchased and kept with the meters to facilitate replacement in the field.

6.1.4 Organic Vapor Analyzer (OVA) Calibration

By analyzing a gas of known concentration, the OVA is calibrated. Methane in air at a concentration of 100 ppm is typically used as the calibration mixture, although the OVA can be calibrated to other compounds. Primary calibration of an OVA is performed at the factory. The OVA is field calibrated daily.

6.1.5 HNu Calibration

By analyzing a gas of known concentration, the HNu is calibrated. Isobutylene is typically used as the calibration gas with the instrument calibrated to benzene equivalents. The procedure in the SOP is used to calibrate the HNu daily or after maintenance.

6.1.6 Gamma Scintillation Detectors (NaI Detectors) Calibration

The field (source check) and manufacturer calibration procedures for NaI detectors for exposure rate measurements are provided in Appendix B, Field Procedures, Procedure RP-26.

6.1.7 Pressurized Ion Chamber (PIC) Calibration

Calibration of the PIC to a primary standard traceable to NIST is performed by the manufacturer. The PIC is recalibrated twice a year by the manufacturer. For field operability (source) checks, the following steps are performed:

1. Assemble the PIC and turn on the instrument. Check each of the batteries and allow the instrument to stabilize for 5 min. Allow the instrument to stabilize to the temperature of the environment it is to be used in.

2. Determine the background exposure rate and record the results.

3. Determine the instrument response to a 1 μCi Cs-137 source (in contact with the ionization chamber wall) and record the results.

6.1.8 Geiger-Mueller Detectors Calibration

Geiger-Mueller detectors are calibrated twice a year by the manufacturer. The HP-210AL and HP-260 are calibrated to Sr-90 and Tc-99 standards.

6.1.9 Alpha Scintillation Detectors Calibration

Alpha scintillation detectors (AC-3) are calibrated by the manufacturer twice a year.

6.2 Laboratory Instruments

6.2.1 Calibration Procedures and Frequency for RAS Analyses

The CLP calibration procedure and frequencies are specified in the CLP organic and inorganic SOWs.

6.2.2 SAS Calibration Procedure and Frequency

The SAS calibration procedures and frequencies are specified in the SAS requests. The SAS requests are located in Appendix C of the QAPjP.

6.2.3 Radionuclide Analysis Calibration and Frequency

The referenced or attached NAREL analytical procedures discuss the instrument calibration procedure and frequency.

7.0 Analytical Procedures

Sediment, surface water, biota, and soil samples collected during field sampling activities for the Kress Creek RI will be analyzed by the CLP for chemical analysis and by NAREL for radionuclides, as specified in the FSP. Both CLP and NAREL will also perform leachability tests.

7.1 CLP Laboratory Analysis

The analytical procedures to be used for performing the SAS analyses are described in the SAS requests in Appendix C of this QAPjP. SAS analysis will be performed by CLP. Also specified in the SAS requests are calibration procedures, frequency of calibration, and the internal QC checks used for each analysis.

Samples for VOC analysis will be screened as recommended in VOC CLP RAS SOW/OLM01.8. Samples, that as a result of the screening would normally be quantified using the VOC CLP "Low" method, will be analyzed by the SOP for Volatiles with the Low Detection Limit method. If screening results indicate that the VOC CLP "Medium" method should be used, the sample will be quantified according to the CLP SOW for Organic analysis, Multimedia, High Concentration, dated September 1988, revision April 1989. This screening and multimethod approach is necessitated by the low detection limits required by the state and EPA while accommodating the potential for samples with high concentrations of VOCs. It is expected that VOC concentrations in media from the Kress Creek site can be analyzed by the Low Detection Limit method.

7.2 Non-CLP Laboratory Analysis (NAREL)

Radioisotopes Th-227, Th-228, Th-230, Th-232, Ra-226, Ra-228, U-234, U-235, and U-238 will be analyzed by NAREL. Table 7.1 summarizes the EPA method to be used for each radioisotope. The methods for Ra-226 and Ra-228 are currently being revised. The draft modifications are included in Appendix F. Methods for leachability testing by NAREL are also included in Appendix F.

7.3 Field Analytical Procedures

The procedures for field measurement of pH, temperature, specific conductivity, dissolved oxygen, hardness, particle size fractioning, surface gamma radiation, and gamma-ray exposure rate are described in Appendix B, Field Procedures.

Table 7-1
Non-Contract Laboratory Program Analytical Methods

Radionuclide	Solids[a]	Water[a]
Ra-226	Ra-01, Ra-04	Ra-03, Ra-04
Ra-228	Ra-01, Ra-04	Ra-03, Ra-04
Th-227	OO-05, OO-06	OO-07
Th-228	OO-05, OO-06	OO-07
Th-230	OO-05, OO-06	OO-07
Th-232	OO-05, OO-06	OO-07
U-234	OO-05, OO-06	OO-07
U-235	OO-05, OO-06	OO-07
U-238	OO-05, OO-06	OO-07

Note: Some samples will be prepared for analysis with leachability (SOP: SCA-301).

[a]From "Eastern Environmental Radiation Facility, Radiochemistry Procedures Manual," EPA, Office of Radiation Programs, EPA 520/5-84-006, 1987.

8.0 Internal Quality Control Checks

8.1 Field Sample Collection

The FSP includes discussion on QC samples that will be collected in the field. The assessment of field sampling precision and accuracy will be made through collection of field duplicates and MS/MSDs. These samples are collected in accordance with the applicable procedures described in the FSP at the frequency indicated in Section 3.0 of the QAPjP.

8.2 Field Measurement

QC procedures for pH, conductivity, temperature, dissolved oxygen, and hardness measurements are limited to checking the reproducibility of the measurement by obtaining multiple readings on a single sample or standard and by calibrating the instruments. Appendix B includes radiological survey QC procedures.

8.3 Laboratory Analysis

QC checks for SAS analyses are identified in the QC requirements Section of the SAS requests. QC checks for radiological analyses are included in the standard EPA procedures from NAREL. These specifications include the types of QC checks required (method blanks, calibration standards, internal standards, surrogate spike standards, laboratory duplicate analysis), compounds and concentrations to be used, the frequency of audits, and the QC acceptance criteria for these audits. Method blanks monitor potential contamination introduced during the analysis. Calibration standards are used to calibrate the instrumentation, thereby controlling the accuracy of the results. Once an instrument has been calibrated, periodically it is confirmed that the system remains in calibration by monitoring the instrument response to internal standards.

For some analysis, such as those performed by gas chromatograph/mass spectrometer (GC/MS), a sample and a blank are spiked with a surrogate compound chosen for properties similar to the analyte of interest. The surrogate spikes evaluate the efficiency of the analytical procedure in recovering the true amount of a known compound. Laboratory duplicates are performed in certain instances (especially for inorganic analysis) to evaluate the reproducibility of the method.

9.0 Data Reduction, Validation, and Reporting

9.1 Sample Collection and Management

RI activities will generate data, including sampling locations, measurements of field parameters, and the results of laboratory analyses. Reports regarding the collection and analysis of sample data will also be generated. The RI process entails the flow of data collected in the field and generated by the analytical laboratory work to those involved in project evaluation and decisionmaking. Figure 9-1 illustrates the project information flow. Management of data collected during RI activities will provide accessibility of data to support environmental data analyses, risk assessments, and the evaluation of remedial action alternatives.

Data collected from the field will be recorded in field logbooks. As samples are collected in the field, the logbooks will be maintained by field team personnel with sample collection, field measurements, and field activity data as discussed in Section 3.3 of the FSP. The field logbooks will include field recordings, calibration information, instrument output reading calculations, deviations from the SAP, and other applicable information.

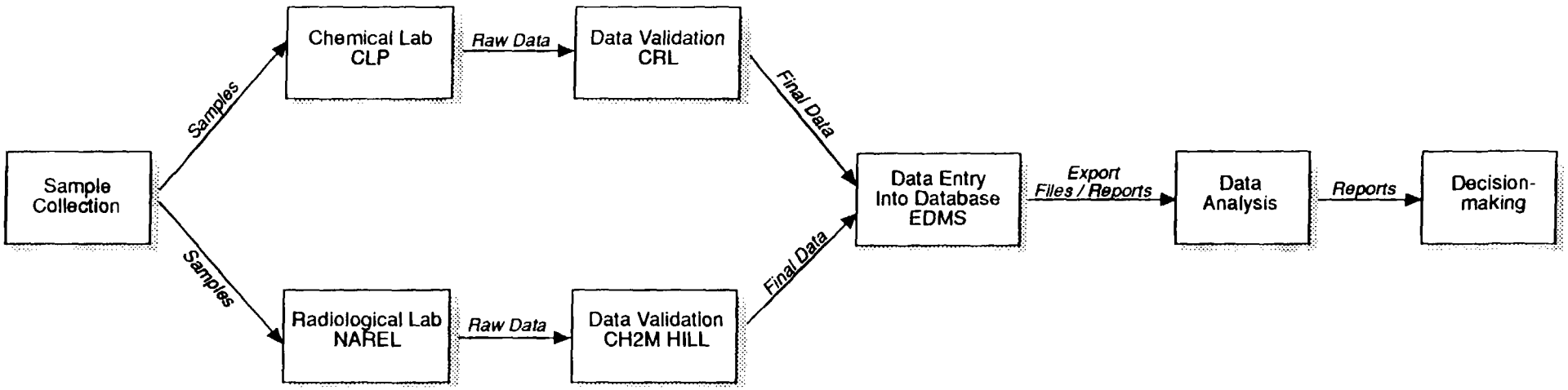
Samples will be tracked from the field collection activities to the analytical laboratories through return of sample residuals from the laboratories following chain-of-custody protocols described in Section 3.0 in the FSP and Section 5.0 in the QAPjP. Sample information recorded on the chain of custody will be input to a sample tracking database (developed by CH2M HILL) to track the status of samples from the time of collection through analysis and validation. Information entered into the sample tracking database includes sample and subsample identification, sample location, sample matrix, sample type (e.g., duplicate), date and time collected, sampler description and comments, and field screening results. The sample tracking database will produce reports to inform the project team of potential delays or problems related to sample analysis and validation.

9.2 Field Data Reduction and Reporting

Direct measurement field procedures include HNu, OVA, pH, temperature, dissolved oxygen, and conductivity. Results from these field procedures and field analysis of hardness and particle size fractioning will be entered into the field logbook. Information from the ecological surveys will be entered in a separate field logbook. Information to be included is described in Section 7.0 of the FSP.

Measurements from the surface gamma scan will be directly recorded on the form associated with the procedure; no further reduction of data is anticipated to occur (other than plotting this data on maps).

RP-28 is the data reduction procedure for the exposure rate measurements. These measurements will be documented on the form associated with the field procedure. Since the data from this procedure will be used in the risk assessment, they will be appropriately validated. This will occur by checking calculations, evaluating duplicate results (should be within 20 percent of each other), and reviewing instrument calibrations and source checks.



NAREL — National Air and Radiation Environmental Laboratory

Figure 9-1
Data Information Flow
Kress Creek RI/FS QAPjP

9.3 Laboratory Reduction and Reporting

Samples collected will be sent to the CLP or NAREL for analysis. Data reduction and reporting for samples analyzed by the CLP will be performed according to specifications outlined in the CLP RAS SOW (OLM01.8) and SOW (ILM02.1). Chemical results will then be sent to the EPA, Region V, LSSS, CRL for data validation. Radiological results will be sent to CH2M HILL for data validation.

The CLP analytical laboratories will prepare and submit full analytical and QC reports to EPA Region V in compliance with requirements of the CLP to include the following (as applicable):

1. Narrative including statement of samples received, description of deviation from RAS or SAS standard procedures, explanation of qualifications regarding data quality, and other significant problems encountered during analysis

2. Up to 20 extractable organic compounds not included in the RAS analytes, tentatively identified and quantified against the nearest internal standard

3. An organic QA/QC report including Forms I to X, surrogate spike results for each sample, MS/MSD results, method blank results, and initial and continuing calibration checks

4. An inorganic QA/QC report including Forms I to XIII, spike and duplicate results, method blank results, and initial and continuing calibration checks

5. Field and laboratory chain-of-custody documentation pertaining to each sample delivery group analyzed

The radiological data packages will be sent by NAREL directly to CH2M HILL for data validation. The package from the NAREL laboratory will include the following:

1. Transmittal Memorandum and General Case Narrative, which includes discussion of method used along with difficulties in adapting the method to the sample. QC frequency, overall performance, and exceptions to QAPjP criteria are included along with unresolved issues on sample collection, preservation, shipping, receipt, or identification.

2. Data on each sample are reported on a data form. Sample identifiers (both field and laboratory) are given along with collection and laboratory receipt dates. Sample results, 2 sigma counting errors, and sample-specific minimum detectable activity (when available) are given.

3. The results of QC samples (duplicates, MS/MSD, and method blanks) are reported.

4. The following data are supplied for each sample:

   - Counter/detector used
   - Total sample count or count rate

- Counting time and date
- Background count or count rate
- Background counting time and date
- Efficiency
- Geometry
- Self absorption factor (alpha/beta)
- Radiochemical yield
- Sample size analyzed
- Spectra printout (alpha and gamma)
- Gamma analysis programs summary sheets

5. The following data are submitted for each data package:

- Counter/detector efficiencies
- Calibration dates
- Calibration standards listing
- Standards certificates
- Daily check source counts for the period covered by the data submitted
- Background counts by detector for the period covered by the data submitted

9.4 Data Validation

Validation will be accomplished by comparing the contents of the data packages and QA/QC results to the requirements contained in RAS methods, SAS methods, and radiological analysis SOPs. Raw data such as GC/MS, Total Ion Current chromatograms, GC chromatograms and mass spectra, inductively coupled plasma (ICP) and furnace atomic adsorption (FAA) data reports, and data station printouts will be examined to evaluate the accuracy of reported results. One hundred percent of the analytical data will be validated to the extent that DQO Level IV data packages allow. The chemical data will be validated by EPA Region V LSSS. The protocol for RAS analyte data validation is presented in the following:

- *Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses.* U.S. EPA, February 1988.

- *Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses.* U.S. EPA, July 1988.

Radiological data will be validated by CH2M HILL. Procedures for validating the radiological data are presented in Appendix D. The procedures are consistent in approach to the functional guidelines for validating inorganic and organic data.

The only field data that will be validated are the exposure rate measurements. The validation will consist of reviewing field and calibration procedures used and checking the reproducibility of the measurements by evaluating duplicate measurements as presented in Section 12.1. CH2M HILL will conduct this validation.

Data that do not meet the required limits may be deemed of insufficient quality for the intended use. However, the data may be of use for other aspects of the project. For instance, data of insufficient quality

for the risk assessment may still be useful in determining the probable site conditions for the FS. Larger numbers of data of lower quality will result in an increase in uncertainty. At the point when the uncertainty can no longer be managed as a reasonable deviation, additional data collection may be warranted.

9.5 Data Storage and Retrieval

Analytical results, applicable QA/QC data, validation flags, and chain-of-custody information will be entered into an environmental database management system (EDMS) for storage and query during data evaluation and RI report development. Data will be manually entered into the database from validated summary forms received from EPA for organic and inorganic compounds and from CH2M HILL for radiological analytes. The data entry will be checked by printing out data reports and manually comparing them with the validated summary analytical forms received from the validators.

The environmental database, EDMS, is a data management system that is based on the INFORMIX database management system. INFORMIX uses Standard Query Language (SQL) combined with a macro programming language and software tools for building menus, online forms, and stored report formats to provide a database management software application system.

Upon completion of validated data entry into EDMS, data evaluation will be conducted to include a review of duplicate samples, a comparison of sample results to blank contamination, an evaluation of detection limits, a comparison to site-background sample results, and an evaluation of contaminants of concern. The database will have the capacity to generate a variety of data reports to aid in data interpretation through database query. Tabular presentations will be used for organizing and evaluating the RI data. The database will be structured to calculate arithmetic mean, standard deviation, and frequency of detection, minimum, and maximum, to assist in the data evaluation.

The data collected during the radiological walk-over survey will be summarized and stored in the form of a technical memorandum. The technical memorandum will include tabular listings for eventual data evaluation.

10.0 Performance and System Audits

Performance and system audits of field and laboratory activities will be conducted to verify that sampling and analysis are performed in accordance with the procedures established in the FSP and QAPjP. The audits of field and laboratory activities include two separate, independent parts: internal and external audits.

10.1 Field Audits

Internal audits of field activities (sampling and measurements) will be conducted by the CH2M HILL Field Activities Manager. The audits will include examination of field sampling records, field instrument operating records, sample collection, handling and packaging in compliance with the established procedures, maintenance of QA procedures, and chain of custody. These audits will occur during the project to demonstrate that established procedures are followed.

An external audit may be conducted by EPA Region V CRL or by the Central District Office (CDO), at their discretion.

10.2 Laboratory Audits

The CLP RAS and SAS laboratories are audited on a regular basis by the EPA. EPA EMSL—Las Vegas conducts the system audits of the CLP laboratories on an annual basis and conducts performance audits on a quarterly basis.

The system audits will include examination of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation and analysis, and instrument operating records. The performance audits will consist of sending performance evaluation (PE) samples to CLP laboratories for ongoing assessment of laboratory precision and accuracy. The analytical results of PE samples are evaluated by EPA to assess the performance of the laboratory.

Additional audits of the SAS laboratories may be conducted by the EPA Region V CRL.

11.0 Preventive Maintenance Procedures

11.1 Field Equipment Instructions

The field equipment for this project includes pH/temperature meter, conductivity meter, HNu, OVA, portable rate meters, gamma and alpha scintillation detectors, Geiger-Mueller detectors, pressurized ion chamber, and dissolved oxygen meters. Specific preventive maintenance procedures to be followed for field equipment are those recommended by the manufacturer and are included in the field procedures in Appendix B.

Field instruments will be checked and calibrated in the warehouse before they are delivered to the field. These instruments will be checked and calibrated daily before use. Calibration checks will be documented in the field logbook.

Essential spare parts such as tape, paper, pH probes, electrodes, and batteries will be kept onsite to minimize instrument downtime. Backup instruments and equipment will be available onsite (as appropriate) to avoid delays in the field schedule.

11.2 Laboratory Instruments

Laboratories participating in the CLP are required, under the CLP RAS SOW (OLM01.8) for organic compounds and SOW (ILM02.1) for inorganic analytes, to have SOPs for preventive maintenance for each measurement system and support activity. Maintenance activities are required to be documented in logbooks to provide a history of maintenance records. NAREL also has preventive maintenance procedures for radiological analytical instruments.

12.0 Specific Routine Procedures to Assess Data Precision, Accuracy, Sensitivity, and Completeness

12.1 Field Measurements

Radiological exposure rate measurement quality will be assessed by reviewing the field results for compliance with the established QC criteria that are specified in the QAPjP and FSP. Accuracy and sensitivity of the field measurements will be assessed using daily instrument calibration and calibration checks. Precision will be assessed on the basis of reproducibility by multiple readings of a single sample. Data completeness will be calculated using the following equation.

$$Completeness\ (\%) = \frac{No.\ of\ Valid\ Data\ Obtained}{No.\ of\ Total\ Data\ Planned} \times 100$$

Valid field data are those data taken in the field under controlled conditions where all test equipment is calibrated as described in Section 6.

12.2 Laboratory Data

Laboratory results will be assessed for compliance with required precision, accuracy, completeness, and sensitivity criteria. The acceptance criteria for these parameters are presented in Section 3.0.

12.2.1 Precision

Precision of laboratory analysis will be assessed by comparing the analytical results between MS and MSD analysis. The relative percent difference (RPD) will be calculated for each pair of duplicate analyses using the following equation.

$$RPD = \frac{|S - D|}{(S + D)/2} \times 100$$

where:

S = first sample value (original or MS value)
D = second sample value (duplicate or MSD value)

12.2.2 Accuracy

Accuracy of laboratory results will be assessed for compliance with the established QC criteria that are described in Section 3.0 of this QAPjP, using analytical results of spiked samples. The percent recovery (%R) of spiked samples will be calculated as below.

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = spiked sample result
SR = sample result (no spike added)
SA = spike added

All units must be the same.

12.2.3 Completeness

The data completeness of laboratory analyses results will be assessed by comparing the amount of valid data to the amount of data required for decisionmaking. Completeness is calculated for laboratory analysis the same way as for field measurements (Section 12.1). For laboratory completeness, the valid data are data that have been conformed to QC protocol and that have not been rejected.

12.2.4 Sensitivity

The achievement of method detection limits depends on instrument sensitivity and matrix effects. Therefore, it is important to monitor the instrument sensitivity to provide data quality through constant instrument performance. The instrument sensitivity will be monitored through the analysis of method blanks, calibration check sample, laboratory control samples, etc.

13.0 Corrective Actions

Corrective actions may be used for two classes of problems: (1) analytical and equipment problems and (2) noncompliance problems. Analytical and equipment problems may occur during sampling and sample handling, sample preparation, laboratory instrument analysis, and data review.

For noncompliance problems, a formal corrective action program will be defined and implemented at the time the problem is identified. The person who identifies the problem is responsible for notifying the Site Manager. If the problem is analytical, information will be promptly communicated to the EPA. Implementation of corrective action will be confirmed in writing through the same channels.

Nonconformance with the established QC procedures in the QAPjP or FSP will be identified and corrected in accordance with the QAPjP. The Site Manager or designee will issue a Nonconformance Report for each nonconformance condition.

Corrective actions will be implemented and documented in the field logbook. No staff member will initiate corrective action without prior communication of findings with the Site Manager. If corrective actions are deemed insufficient by the Remedial Project Manager, work may be halted by a stop-work order.

13.1 Sample Collection and Field Measurements

Technical staff and project personnel will be responsible for reporting suspected technical or QA nonconformances or suspected deficiencies of any activity or issued document by reporting the situation to the Site Manager or designee. The Site Manager will be responsible for assessing the suspected problems and will make a decision based on the potential for the situation to affect data quality. If the situation warrants a reportable nonconformance that requires corrective action, a nonconformance report will be initiated by the Site Manager.

The Site Manager will be responsible for initiating corrective action for nonconformances by the following methods:

- evaluating reported nonconformances
- controlling additional work on nonconforming items
- evaluating disposition or action to be taken
- maintaining a record of nonconformances
- reviewing nonconformance reports and corrective actions taken
- including, in project files, nonconformance reports in the final site documentation

If appropriate, the Site Manager will control additional work that is dependent on the nonconforming activity. In most instances this will involve nonperformance of work until the corrective actions are completed.

Corrective actions for field measurements may include the following:

- Repeat the measurement to check the error.
- Check for adjustments for ambient conditions such as temperature.

- Check the batteries.
- Recalibrate.
- Check the calibration.
- Replace the instrument or measurement devices.
- Stop work.

The Field Activities Manager or designee is responsible for all site activities. In this role, the Field Activities Manager at times may adjust the site programs to accommodate site-specific conditions. When program modification is necessary, the responsible person notifies the Site Manager of the anticipated change and implements those changes after obtaining approval of the Site Manager and, if appropriate, the Remedial Project Manager. The change in the program will be documented on the field change request (FCR) that will be signed by the initiators and the Field Activities Manager. The FCRs will be numbered serially. The FCR will be attached to the file copy of the QAPjP. The Site Manager must approve the change in writing or verbally prior to field implementation, if feasible. If unacceptable, the action taken during the period of deviation will be evaluated to assess the significance of any departure from established program practices and other action will be taken.

The Field Activities Manager is responsible for controlling, tracking, and implementing the identified changes. Reports on changes will be distributed to affected parties, which includes the EPA Remedial Project Manager. The Remedial Project Manager will be notified whenever program changes are made in the field.

13.2 Laboratory Analyses

Laboratory Corrective Actions – CLP RAS Plus SAS

For the CLP RAS plus SAS, corrective actions are implemented at several different levels. The laboratories participating in the CLP are required to have a written SOP specifying corrective action to be taken when an analytical error is discovered or when the analytical system is determined to be out of control. The SOP requires documentation of the corrective action and notification by the analyst in regard to the errors and corrective procedures.

EPA may request corrective action for contractual nonconformance identified by audits or data validation. The CRL may request, through the Sample Management Office, corrective action by the laboratories for nonconformances identified in the data validation process or, for minor problems, the laboratory may be contacted directly. Corrective action may include the following:

- Reanalyzing the samples, if holding time criteria permits
- Resampling and analyzing
- Evaluating and amending sampling procedures
- Evaluating and amending analytical procedures
- Accepting data and acknowledging the level of uncertainty

If resampling is to be performed as a result of laboratory problems, the Remedial Project Manager must identify the approach, including cost recovery from the CLP, for the additional sampling effort.

Laboratory Corrective Actions – SASs

For SAS requests, corrective actions per specification will be implemented as required by the SAS request and according to the procedure described in Section 13.2.

14.0 Quality Assurance Reports to Management

In addition to the audit reports submitted to the Site Manager in accordance with QAPjP Section 10.0, a monthly progress report that addresses QA issues will be submitted to the EPA Remedial Project Manager and to CH2M HILL program management. The final RI report will contain a Data Evaluation section that summarizes data quality information collected during the project.

The monthly progress reports will contain the following information:

- Progress made during the reporting period and activities planned for the next period; audits conducted
- Problems (including QA/QC issues), recommended solutions, and corrective actions implemented
- Deliverables submitted
- Personnel changes
- Subcontractor services used, travel taken, and laboratory services used
- Discussion on the performance of the project along with current schedule and budget information; results of audits

The RI report will contain discussions of the following data QA/QC issues.

- Data quality assessment in terms of PARCC and method detection limits
- The degree to which DQOs were met
- Limitations of the measurement data; usability of the data
- Applicability of the data to site conditions

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Appendix A

Field Sampling Plan
Kress Creek/West Branch DuPage River

PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET

DATE: _____
FIELD CREW: _____
PROJECT #: _____
PROJECT NAME: _____
STATION NAME/NUMBER: _____

PHYSICAL CHARACTERIZATION

RIPARIAN ZONE/WATER

Predominant Surrounding Land Use:

Forest Field/Pasture Agriculture Residential Commercial Industrial Other

High Water Mark_____ (m) Velocity _____ Dam Present: Yes___ No___ Channelized: Yes___ No___

Sample Area : Ave. Depth _____ Width _____ Length _____

Canopy Cover: Open Partly Open Partly Shaded Shaded

SEDIMENT/SUBSTRATE:

Sediment Odors: Normal None Sewage Petroleum Chemical Anaerobic Other_____

Sediment Oils: Absent Slight Moderate Profuse

Sediment Deposits: Sludge Sawdust Paper Fiber Sand Relict Shells Leaf Litter/Woody Debris Other____

Are the undersides of stones which are not deeply embedded black? Yes___ No___

Inorganic Substrate Components			Organic Substrate Components		
Substrate Type	Diameter	Percent Composition In sampling area	Substrate Type	Characteristic	Percent Composition In Sampling Area
Bedrock			Detritus	Sticks, Wood, Coarse Plant Material (CPOM)	
Boulder	>256mm (10 in.)				
Cobble	64-256mm (2.5-10 in.)		Muck-Mud	Black, Very Fine Organic (FPOM)	
Gravel	2-64mm (0.1-2.5 in.)				
Sand	0.06-2.00mm (gritty)				
Silt	0.004-.06mm		Marl	Grey, Shell Fragments	
Clay	<0.004mm (slick)				

Figure 7-6
Physical Characterization / Water Quality Field Data Sheet
Kress Creek RI/FS FSP

PHYSICAL CHARACTERIZATION/WATER QUALITY FIELD DATA SHEET

DATE: _____
PROJECT #: _____
STATION NAME/NUMBER: _____

WATER QUALITY

Stream Type:	Coldwater		Warmwater			
Water Odors:	Normal	Sewage	Petroleum	Chemical	None	Other _____
Water Surface Oils:	Slick	Sheen	Globs	Flecks	None	
Turbidity:	Clear	Slightly Turbid		Turbid	Opaque	Water Color _____

WATER CHEMISTRY

Time _____

Dissolved Oxygen _____ MG/L

D.O. Temperature _____ (C)

Conductivity _____ µmhos/cm

Cond. Temperature _____ (C)

pH _____

pH Temperature _____ (C)

Hardness _____ ml as $CaCO_3$

Hardness Temperature _____ (C)

_____

_____

NOTES:

Figure 7-6
Physical Characterization / Water Quality Field Data Sheet (continued)

- Estimated Creek Depth—Measure the vertical distance from water surface to stream bottom at a representative depth of each habitat type (riffle, run, pool) that exists within each segment.

- High Water Mark—Estimate the vertical distance from the creek bank to the peak overflow level.

- Velocity—Estimate stream velocity along three transects within each segment with the use of a March-McBirney flow meter. For shallow depths (<2 ft), the velocity reading at each point will be taken within a foot of the surface. For deeper depths (>2 ft), readings will be taken at 1-ft intervals to within 1 ft of the bottom.

- Dam Presence—Observe any upstream obstruction to flow.

- Channelization—Observe whether or not the segment has been altered due to channelization.

- Canopy Cover—Estimate the proportion of open areas to shaded areas within each segment.

- Sediment Odors—Note any unusual odors associated with the sediment of each segment.

- Sediment Oils—Note any sediment oils and relative amounts observed within each segment.

- Sediment Deposits—Note any unusual deposits (fiber, sludge, ferric hydroxide deposits, etc.) that may be in each segment. Also indicate whether the underside of rocks that are not deeply embedded are black (may indicate low dissolved oxygen or anaerobic conditions).

- Inorganic Substrate Components—Estimate relative proportions of bedrock, boulder, cobble, gravel, sand, silt, and clay.

- Organic Substrate Components—Estimate relative abundance of detritus, organic mud/muck, and clay.

- Stream Type—Note if the creek is a cold- or warm-water stream.

- Water Odors—Note any unusual odors associated with the water in each segment.

- Water Surface Oils—Note the presence and amount of any water surface oils in each segment.

- Turbidity—Record visual observations of the amount of suspended material in the water column (clear, slight turbid, turbid, opaque, water color).

7.7.3 Habitat Assessment

The habitat assessment is intended to support the biological survey by providing additional information about the parameters that would support a diverse assemblage of aquatic organisms. The following habitat parameters will be assessed and entered on the appropriate form (Figure 7-7):

DATE/TIME: _____ PROJECT NAME: _____

FIELD CREW: _____ STATION NAME/NUMBER _____

PROJECT #: _____

Habitat Parameter				
1. Bottom substrate/ available cover (a)	Greater than 50% rubble, gravel, submerged logs, undercut banks, or other stable habitat. 16 - 20	30-50% rubble, gravel or other stable habitat. Adequate habitat. 11 -15	10 30% rubble, gravel or other stable habitat. Habitat availability less than desirable. 6 -10	Less than 10% rubble, gravel or other stable habitat. Lack of habitat is obvious. 0 - 5
2. Embeddedness (b)	Gravel, cobble, and boulder particles are between 0 and 25% surrounded by fine sediment. 16 - 20	Gravel, cobble, and boulder particles are between 25 and 50% surrounded by fine sediment. 11 - 15	Gravel, cobble, and boulder particles are between 50 and 75% surrounded by fine sediment. 6 - 10	Gravel, cobble, and boulder particles are over 75% surrounded by fine sediment. 0 - 5
3. Flow at rep. low flow (a)	Cold > 2 cfs Warm > 5 cfs 10 -20	1 - 2 cfs 2 - 5 cfs 11 - 15	0.5 - 1 cfs 1 - 2 cfs 6 - 10	< 0.5 cfs < 1 cfs 0 - 5
4. Channel alteration (a)	Little or no enlarge - ment of islands or point bars, and/or no channelization. 12 - 15	Some new increase in bar formation, mostly from coarse gravel; and/or sone channel - ization present. 8 - 11	Moderate deposition of new gravel, coarse sand on old and new bars; pools partially filled w/silt; and/or embank - ments on both banks 4 - 7	Heavy deposits of fine material, increased bar development; most pools filled w/silt; and/or extensive channelization 0 - 3
5. Bottom scouring and deposition (a)	Less than 5% of the bottom affected by	5 - 30% affected . Scour at constrictions and	30 - 50% affected. Deposits and scour at	More than 50% of the bottom changing

Figure 7-7
Habitat Assessment Field Data Sheet
Kres k RI/FS FSP

	scouring and deposition 12 - 15	where grades steepen. Some deposition in pools 8 - 11	obstructions. con - strictions and bends. Some filling of pools. 4 - 7	nearly year long. Pools almost absent due to deposition. Only large rocks in riffle exposed. 0-3
6. Pool/riffle, run/bend ratio (a) (distance between riffles divided by stream width)	5 - 7. Variety of habitat. Deep riffles and pools. 12 - 15	7 - 15. Adequate depth in pools and riffles. Bends provide habitat. 8 - 11	15 - 25. Occassional riffle or bend. Bottom contours provide some habitat. 4 - 7	> 25. Essentially a straight stream. Generally all flat water or shallow riffle. Poor Habitat. 0 - 3
7. Bank stability (a)	Stable. No evidence of erosion or bank failure Side slopes generally < 30%. Little potential for future problem. 9 - 10	Moderately stable. Infrequent, small areas of erosion mostly healed over. Side slopes up to 40 % on one bank. Slight potential in extreme floods. 6 - 8	Moderately unstable. Moderate frequency and size of erosional areas. Side slopes up to 60 % on some banks. High erosion potential during extreme high flow. 3 - 5	Unstable. Many eroded areas. Side slopes > 60 % common. "Raw" areas frequent along straight sections and bends. 0 - 2
8. Bank vegetation stability (b)	Over 80% of the streambank surfaces covered by vegetation of boulders and cobble. 9 - 10	50 - 79% of the streambank surface covered by vegetation, gravel, or larger material. 6 - 8	25 - 49% of the stream-bank surfaces covered by vegetation, gravel, or larger material. 3 - 5	Less than 25% of the streambank surfaces covered by vegetation, gravel, or larger material. 0 - 2
9. Streamside cover (b)	Dominant vegetation is shrub.	Dominant vegetation is of tree form.	Dominant vegetation is grass or forbes.	Over 50% of the streambank has no vegetation and

Figure 7-7
Habitat Assessment Field Data Sheet (con't.)
Kress Creek RI/FS FSP

				dominant material is soil, rock, bridge material, culverts, or mine tailings.
	9 - 10	6 - 8	3 - 5	0 - 2
	_____	_____	_____	_____
Column Totals	_____	_____	_____	_____

Score _____

(a) From Ball 1982
(b) from Platts et al. 1983

COMMENTS:

Figure 7-7
Habitat Assessment Field Data Sheet (con't.)

- Bottom Substrate—Availability of habitat for support of aquatic organisms. Observe the variety of substrate materials and habitat types (rock/cobble, stumps/submerged logs, etc.) within each segment.

- Embeddedness—Degree to which rock, rubble, and gravel are surrounded by fine sediment. Indicates suitability of the stream substrate as habitat for benthic invertebrates and for fish spawning and egg incubation.

- Stream Flow and/or Stream Velocity—Stream flow relates to the ability of a stream to provide and maintain a stable aquatic environment, directly influencing the structure of both benthic macroinvertebrates and fish community.

- Channel Alternation—Observe and note any deposition of sediment materials, siltation of pool and riffle areas, loss of stream sinuosity, and potential for scouring.

- Bottom Scouring and Deposition—Relates to the destruction of in-stream habitat resulting from channelization. Deposition and scouring is rated as a percentage of the valuated reach that is scoured or silted.

- Pool/Riffle or Run/Bend Ratio—Riffles and bends provide more diverse habitat than a straight (run) or uniform depth stream. A ratio is calculated by dividing the average stream distance between riffles or bends by the average stream width. Comparisons are made between stream reaches and a reference location or a preferred/ideal condition.

- Bank Stability—Observe existing or potential detachment of soil from upper and lower stream bank and its potential movement into the stream.

- Bank Vegetation Stability—An estimate of the density of bank vegetation (or rock, cobble, gravel material) covering the bank and providing bank stability.

- Streamside Cover—A rating obtained by visually determining the dominant vegetation type covering the exposed stream bottom, bank, and top of bank.

8. Field Measurements and Screening

Standard Operating Procedures (SOPs) (HNu, OVA, pH/temperature, conductivity, dissolved oxygen, and various radioactivity measurements) for the field measurement and screening techniques are provided in Appendix B.

9. Preventive Maintenance Procedure and Schedule

Field team members will refer to the field procedure SOP and/or the manufacturers' instrument manuals for the appropriate preventive maintenance procedures for the field equipment used at the site.

10. Sample Disposal

Investigation-derived waste generated during the field activities will be managed in accordance with the Waste Management Plan presented in Appendix E.

Attachment I

Standard Abbreviation for Birds

STANDARD ABBREVIATIONS FOR COMMON NAMES OF BIRDS

LOONS

1	COLO	'Common Loon'	(Gavia immer)
2	YBLO	'Yellow-billed Loon'	(Gavia adamsii)
3	ARLO	'Arctic Loon'	(Gavia arctica)
4	RTLO	'Red-throated Loon'	(Gavia stellata)

GREBES

5	RNGR	'Red-necked Grebe'	(Podiceps grisegena)
6	HOGR	'Horned Grebe'	(Podiceps auritus)
7	EAGR	'Eared Grebe'	(Podiceps nigricollis)
8	LEGR	'Least Grebe'	(Tachybaptus dominicus)
9	WEGR	'Western Grebe'	(Aechmorphorus occidentalis)
10	PBGR	'Pied-billed Grebe'	(Podilymbus podiceps)
	UNGR	'Unidentified Grebe'	

ALBATROSSES

11	BFAL	'Black-footed Albatross'	(Diomedea nigripes)
12	LAAL	'Laysan Albatross'	(Diomedea immutabilis)

SHEARWATERS

13	NOFU	'Northern Fulmar'	(Fulmarus glacialis)
14	GRSH	'Greater Shearwater'	(Puffinus gravis)
15	SOSH	'Sooty Shearwater'	(Puffinus griseus)
16	MASH	'Manx Shearwater'	(Puffinus puffinus)
17	COSH	'Cory's Shearwater'	(Calonectris diomedea)
18	PFSH	'Pink-footed Shearwater'	(Puffinus creatopus)
19	STSH	'Short-tailed Shearwater'	(Puffinus tenuirostris)
20	BLSH	'Buller's Shearwater'	(Puffinus bulleri)
21	AUSH	'Audubon's Shearwater'	(Puffinus lherminieri)
22	BEPE	'Bermuda Petrel'	(Pterodroma cahow)
23	BOPE	'Bonin Petrel'	(Pterodroma hypoleuca)

STORM-PETRELS

24	FTSP	'Fork-tailed Storm-Petrel'	(Oceanodroma furcata)
25	LSPE	'Leach's Storm-Petrel'	(Oceanodroma leucorhoa)
26	ASPE	'Ashy Storm-Petrel'	(Oceanodroma homochroa)
27	BSPE	'Black Storm-Petrel'	(Oceanodroma melania)
28	LTPE	'Least Storm-Petrel'	(Oceanodroma microsoma)
29	WSPE	'Wilson's Storm-Petrel'	(Oceanites oceanicus)

TROPICBIRDS

30	RBTR	'Red-billed Tropicbird'	(Phaethon aethereus)
31	WTTR	'White-tailed Tropicbird'	(Phaethon lepturus)
32	RTTR	'Red-tailed Tropicbird'	(Phaethon rubricauda)

PELICANS

33 AWPE	'American White Pelican'	(Pelecanus erythrorhynchos)
34 BRPE	'Brown Pelican'	(Pelecanus occidentalis)

BOOBIES, GANNETS

35 MABO	'Masked Booby'	(Sula dactylatra)
36 BLBO	'Blue-footed Booby'	(Sula nebouxii)
37 BRBO	'Brown Booby'	(Sula leucogaster)
38 RFBO	'Red-footed Booby'	(Sula sula)
39 NOGA	'Northern Gannet'	(Sula bassanus)

CORMORANTS

40 GRCO	'Great Cormorant'	(Phalacrocorax carbo)
41 DCCO	'Double-crested Cormorant'	(Phalacrocorax auritus)
42 OLCO	'Olivaceous Cormorant'	(Phalacrocorax olivaceus)
43 BNCO	'Brandt's Cormorant'	(Phalacrocorax penicillatus)
44 PECO	'Pelagic Cormorant'	(Phalacrocorax pelagicus)
45 RFCO	'Red-faced Cormorant'	(Phalacrocorax urile)

ANHINGAS

46 ANHI	'Anhinga'	(Anhinga anhinga)

FRIGATEBIRDS

47 MAFR	'Magnificent Frigatebird'	(Fregata magnificens)
48 GRFR	'Great Frigatebird'	(Fregata minor)
49 LEFR	'Lesser Frigatebird'	(Fregata ariel)

HERONS

50 GBHE	'Great Blue Heron'	(Ardea herodias)
51 GRHE	'Green-backed Heron'	(Butorides striatus)
52 LBHE	'Little Blue Heron'	(Egretta caerulea)
53 CAEG	'Cattle Egret'	(Bubulcus ibis)
54 REEG	'Reddish Egret'	(Egretta rufescens)
55 GREG	'Great Egret'	(Casmerodius albus)
56 SNEG	'Snowy Egret'	(Egretta thula)
57 TRHE	'Tricolored Heron'	(Egretta tricolor)
58 BCNH	'Black-Crowned Night Heron'	(Nycticorax nycticorax)
59 YCNH	'Yellow-Crowned Night Heron'	(Nycticorax violaceus)
60 LEBI	'Least Bittern'	(Ixobrychus exilis)
61 AMBI	'American Bittern'	(Botaurus lentiginosus)
UNHE	'Unidentified Heron/Egret'	

STORKS

62 WOST	'Wood Stork'	(Mycteria americana)

IBISES

63 GLIB	'Glossy Ibis'	(Plegadis falcinellus)
64 WFIB	'White-faced Ibis'	(Plegadis chihi)
65 WHIB	'White Ibis'	(Eudocimus albus)
66 SCIB	'Scarlet Ibis'	(Eudocimus ruber)
67 ROSP	'Roseate Spoonbill'	(Ajaia ajaja)

FLAMINGOS

68 GAFL	'Greater American Flamingo'	(Phoenicopterus ruber)

WATERFOWL

69 MUSW	'Mute Swan'	(Cygnus olor)
70 WPSW	'Whooper Swan'	(Cygnus cygnus)
71 BESW	'Bewick's Swan'	(Cygnus columbianus bewickii)
72 TUSW	'Tundra Swan'	(Cygnus columbianus)
73 TPSW	'Trumpeter Swan'	(Cygnus buccinator)
74 CAGO	'Canada Goose'	(Branta canadensis)
75 BRAN	'Brant'	(Branta bernicla)
76 BAGO	'Barnacle Goose'	(Branta leucopsis)
77 EMGO	'Emperor Goose'	(Chen canagica)
78 GWFG	'Greater White-fronted Goose'	(Anser albifrons)
79 SNGO	'Snow Goose'	(Chen caerulescens)
80 ROGO	'Ross' Goose'	(Chen rossii)
81 BBWD	'Black-bellied Whistling-Duck'	(Dendrocygna autumnalis)
82 FWDU	'Fulvous Whistling-Duck'	(Dendrocygna bicolor)
83 MALL	'Mallard'	(Anas platyrhynchos)
84 ABDU	'American Black Duck'	(Anas rubripes)
85 MODU	'Mottled Duck'	(Anas fulvigula)
86 GADW	'Gadwall'	(Anas strepera)
87 NOPI	'Northern Pintail'	(Anas acuta)
88 GWTE	'Green-winged Teal'	(Anas crecca)
89 BWTE	'Blue-winged Teal'	(Anas discors)
90 CITE	'Cinnamon Teal'	(Anas cyanoptera)
91 GATE	'Garganey Teal'	(Anas querquedula)
92 UNTE	'Unidentified Teal'	
93 EUWI	'Eurasian Widgeon'	(Anas penelope)
94 AMWI	'American Widgeon'	(Anas americana)
95 NOSH	'Northern Shoveler'	(Anas clypeata)
96 WODU	'Wood Duck'	(Aix sponsa)
97 REDH	'Redhead'	(Aythya americana)
98 RNDU	'Ring-necked Duck'	(Aythya collaris)
99 CANV	'Canvasback'	(Aythya valisineria)
100 GRSC	'Greater Scaup'	(Aythya marila)
101 LESC	'Lesser Scaup'	(Aythya affinis)
102 COGO	'Common Goldeneye'	(Bucephala clangula)
103 BWGO	'Barrow's Goldeneye'	(Bucephala islandica)
104 BUFF	'Bufflehead'	(Bucephala albeola)
105 OLDS	'Oldsquaw'	(Clangula hyemalis)

108	COEI	'Common Eider'	(Somateria mollissima)
109	KIEI	'King Eider'	(Somateria spectabilis)
110	SPEI	'Spectacled Eider'	(Somateria fischeri)
111	WWSC	'White-winged Scoter'	(Melanitta fusca)
112	SUSC	'Surf Scoter'	(Melanitta perspicillata)
113	BLSC	'Black Scoter'	(Melanitta nigra)
114	RUDU	'Ruddy Duck'	(Oxyura jamaicensis)
115	MADU	'Masked Duck'	(Oxyura dominica)
116	HOME	'Hooded Merganser'	(Lophodytes cucullatus)
117	COME	'Common Merganser'	(Mergus merganser)
118	RBME	'Red-breasted Merganser'	(Mergus serrator)
	UNDU	'Unidentified Duck'	

VULTURES

119	TUVU	'Turkey Vulture'	(Cathartes aura)
120	BLVU	'Black Vulture'	(Coragyps atratus)
121	CACO	'California Condor'	(Gymnogyps californianus)

KITES, HAWKS

122	WTKI	'White-tailed Kite'	(Elanus caeruleus)
123	ASTK	'American Swallow-tailed Kite'	(Elanoides forficatus)
124	MIKI	'Mississippi Kite'	(Ictinia mississippiensis)
125	SNKI	'Snail Kite'	(Rostrhamus sociabilis)
126	NOGO	'Northern Goshawk'	(Accipiter gentilis)
127	SSHA	'Sharp-shinned Hawk'	(Accipiter striatus)
128	COHA	'Cooper's Hawk'	(Accipiter cooperii)
129	RTHA	'Red-tailed Hawk'	(Buteo jamaicensis)
130	RSHA	'Red-shouldered Hawk'	(Buteo lineatus)
131	BWHA	'Broad-winged Hawk'	(Buteo platypterus)
132	SWHA	'Swainson's Hawk'	(Buteo swainsoni)
133	ZTHA	'Zone-tailed Hawk'	(Buteo albonotatus)
134	WTHA	'White-tailed Hawk'	(Buteo albicaudatus)
135	STHA	'Short-tailed Hawk'	(Buteo brachyurus)
136	RLHA	'Rough-legged Hawk'	(Buteo lagopus)
137	FEHA	'Ferruginous Hawk'	(Buteo regalis)
138	GRHA	'Gray Hawk'	(Buteo nitidus)
139	HAHA	'Harris' Hawk'	(Parabuteo unicinctus)
140	CBHA	'Common Black Hawk'	(Buteogallus anthracinus)
141	GOEA	'Golden Eagle'	(Aquila chrysaetos)
142	BAEA	'Bald Eagle'	(Haliaeetus leucocephalus)
143	NOHA	'Northern Harrier'	(Circus cyaneus)
	UNHA	'Unidentified Hawk'	

OSPREY

144	OSPR	'Osprey'	(Pandion haliaetus)

FALCONS

145	CRCA	'Crested Caracara'	(Polyborus plancus)
146	GYRF	'Gyrfalcon'	(Falco rusticolus)
147	PRFA	'Prairie Falcon'	(Falco mexicanus)
148	PEFA	'Peregrine Falcon'	(Falco peregrinus)
149	APFA	'Aplomado Falcon'	(Falco femoralis)
150	MERL	'Merlin'	(Falco columbarius)
151	AMKE	'American Kestrel'	(Falco sparverius)
	UNFA	'Unidentified Falcon'	

CHACHALACA

152	PLCH	'Plain Chachalaca'	(Ortalis vetula)

GROUSE, PTARMIGAN

153	BUGR	'Blue Grouse'	(Dendragapus obscurus)
154	SPGR	'Spruce Grouse'	(Dendragapus canadensis)
155	RUGR	'Ruffed Grouse'	(Bonasa umbellus)
156	WIPT	'Willow Ptarmigan'	(Lagopus lagopus)
157	ROPT	'Rock Ptarmigan	(Lagopus mutus)
158	WTPT	'White-tailed Ptarmigan'	(Lagopus leucurus)
159	GPCH	'Greater Prairie-Chicken'	(Tympanuchuscupido)
160	LPCH	'Lesser Prairie-Chicken'	(Tympanuchus pallidicinctus)
161	STGR	'Sharp-tailed Grouse'	(Tympanuchus phasianellus)
162	SAGR	'Sage Grouse'	(Centrocercus urophasianus)

QUAIL, PHEASANT

163	NOBO	'Northern Bobwhite'	(Colinus virginianus)
164	MABW	'Masked Bobwhite'	(Colinus virginianus ridgwayi)
165	SCQU	'Scaled Quail'	(Callipepla squamata)
166	CAQU	'California Quail'	(Callipepla californica)
167	GAQU	'Gambel's Quail'	(Callipepla gambelii)
168	MOQU	'Mountain Quail'	(Oreortyx pictus)
169	MZQU	'Montezuma Quail'	(Cyrtonyx montezumae)
170	RNPH	'Ring-necked Pheasant'	(Phasianus colchicus)
171	CHUK	'Chukar'	(Alectoris chukar)
172	GRPA	'Gray Partridge'	(Perdix perdix)
173	BLFR	'Black Francolin'	(Francolinus francolinus)

TURKEY

174	WITU	'Wild Turkey'	(Meleagris gallopavo)

CRANES

175	WHCR	'Whooping Crane'	(Grus americana)
176	GSCR	'Greater Sandhill Crane'	(Grus canadensis tabida)
177	SACR	'Sandhill Crane'	(Grus canadensis)

LIMPKIN

178 LIMP	'Limpkin'	(Aramus guarauna)

RAILS, GALLINULES, COOTS

179 KIRA	'King Rail'	(Rallus elegans)
180 CLRA	'Clapper Rail'	(Rallus longirostris)
181 VIRA	'Virginia Rail'	(Rallus limicola)
182 SORA	'Sora'	(Porzana carolina)
183 YERA	'Yellow Rail'	(Coturnicops noveboracensis)
184 BLRA	'Black Rail'	(Laterallus jamaicensis)
185 PUGA	'Purple Gallinule'	(Porphyrula martinica)
186 COMO	'Common Moorhen'	(Gallinula chloropus)
187 AMCO	'American Coot'	(Fulica americana)

JACANA

188 NOJA	'Northern Jacana'	(Jacana spinosa)

OYSTERCATCHERS

189 AMOY	'American Oystercatcher'	(Haematopus palliatus)
190 ABOY	'American Black Oystercatcher'	(Haematopus bachmani)

PLOVERS

191 NOLA	'Northern Lapwing'	(Vanellus vanellus)
192 CRPL	'Common Ringed Plover'	(Charadrius hiaticula)
193 SEPL	'Semipalmated Plover'	(Charadrius semipalmatus)
194 PIPL	'Piping Plover'	(Charadrius melodus)
195 SNPL	'Snowy Plover'	(Charadrius alexandrinus)
196 WIPL	'Wilson's Plover'	(Charadrius wilsonia)
197 KILL	'Killdeer'	(Charadrius vociferus)
198 MOPL	'Mountain Plover'	(Charadrius montanus)
199 LGPL	'Lesser Golden-Plover'	(Pluvialis dominica)
200 BBPL	'Black-bellied Plover'	(Pluvialis squatarola)
201 SURF	'Surfbird'	(Aphriza virgata)
202 RUTU	'Ruddy Turnstone'	(Arenaria interpres)
203 BLTU	'Black Turnstone'	(Arenaria melanocephala)
UNPL	'Unidentified Plover'	

SANDPIPERS

204 AMWO	'American Woodcock'	(Scolopax minor)
205 COSN	'Common Snipe'	(Gallinago gallinago)
206 LBCU	'Long-billed Curlew'	(Numenius americanus)
207 WHIM	'Whimbrel'	(Numenius phaeopus)
208 BTCU	'Bristle-thighed Curlew'	(Numenius tahitiensis)
209 UPSA	'Upland Sandpiper'	(Bartramia longicauda)
210 SPSA	'Spotted Sandpiper'	(Actitis macularia)
211 SOSA	'Solitary Sandpiper'	(Tringa solitaria)

213	WILL	'Willet'	(Catoptrophorus semipalmatus)
214	GRYE	'Greater Yellowlegs'	(Tringa melanoleuca)
215	LEYE	'Lesser Yellowlegs'	(Tringa flavipes)
216	REKN	'Red Knot'	(Calidris canutus)
217	PUSA	'Purple Sandpiper'	(Calidris maritima)
218	ROSA	'Rock Sandpiper'	(Calidris ptilocnemis)
219	SHSA	'Sharp-tailed Sandpiper'	(Calidris acuminata)
220	PESA	'Pectoral Sandpiper'	(Calidris melanotos)
221	WRSA	'White-rumped Sandpiper'	(Calidris fuscicollis)
222	BASA	'Baird's Sandpiper'	(Calidris bairdii)
223	LESA	'Least Sandpiper'	(Calidris minutilla)
224	CUSA	'Curlew Sandpiper'	(Calidris ferruginea)
225	DUNL	'Dunlin'	(Calidris alpina)
226	SBDO	'Short-billed Dowitcher'	(Limnodromus griseus)
227	LBDO	'Long-billed Dowitcher'	(Limnodromus scolopaceus)
228	STSA	'Stilt Sandpiper'	(Calidris himantopus)
229	SESA	'Semipalmated Sandpiper'	(Calidris pusilla)
230	WESA	'Western Sandpiper'	(Calidris mauri)
231	BBSA	'Buff-breasted Sandpiper'	(Tryngites subruficollis)
232	MAGO	'Marbled Godwit'	(Limosa fedoa)
233	BTGO	'Bar-tailed Godwit'	(Limosa lapponica)
234	HUGO	'Hudsonian Godwit'	(Limosa haemastica)
235	BKGO	'Black-tailed Godwit'	(Limosa limosa)
236	RUFF	'Ruff'	(Philomachus pugnax)
237	SAND	'Sanderling'	(Calidris alba)
	UNSA	'Unidentified Sandpiper'	

AVOCET, STILT

238	AMAV	'American Avocet'	(Recurvirostra americana)
239	BNST	'Black-necked Stilt'	(Himantopus mexicanus)

PHALAROPES

240	REPH	'Red Phalarope'	(Phalaropus fulicaria)
241	WIPH	'Wilson's Phalarope'	(Phalaropus tricolor)
242	RDPH	'Red-necked Phalarope'	(Phalaropus lobatus)

JAEGERS, SKUAS

243	POJA	'Pomerine Jaeger'	(Stercorarius pomarinus)
244	PAJA	'Parasitic Jaeger'	(Stercorarius parasiticus)
245	LTJA	'Long-tailed Jaeger'	(Stercorarius longicaudus)
246	GRSK	'Great Skua'	(Catharacta skua)
247	SPSK	'South Polar Skua'	(Catharacta maccormickf)

GULLS, TERNS

248	GLGU	'Glaucous Gull'	(Larus hyperboreus)
249	ICGU	'Iceland Gull'	(Larus glaucoides)
250	KUGU	'Kumlien's Gull'	(Larus glaucoides kumlieni)
251	GWGU	'Glaucous-Winged Gull'	(Larus glaucescens)

254	LBBG	'Lesser Black-backed Gull'	(Larus fuscus)
255	HERG	'Herring Gull'	(Larus argentatus)
256	THGU	'Thayer's Gull'	(Larus thayeri)
257	CAGU	'California Gull'	(Larus californicus)
258	RBGU	'Ring-billed Gull'	(Larus delawarensis)
259	MEGU	'Mew Gull'	(Larus canus)
260	CBHG	'Common Black-headed Gull'	(Larus ridibundus)
261	LAGU	'Laughing Gull'	(Larus atricilla)
262	FRGU	'Franklin's Gull'	(Larus pipixcan)
263	BOGU	'Bonaparte's Gull'	(Larus philadelphia)
264	LIGU	'Little Gull'	(Larus minutus)
265	HMGU	'Heermann's Gull'	(Larus heermanni)
266	IVGU	'Ivory Gull'	(Pagophila eburnea)
267	BLKI	'Black-legged Kittiwake'	(Rissa tridactyla)
268	RLKI	'Red-legged Kittiwake'	(Rissa brevirostris)
269	ROGU	'Ross' Gull'	(Rhodostethia rosea)
270	SAGU	'Sabine's Gull'	(Xema sabini)
271	GBTE	'Gull-billed Tern'	(Sterna nilotica)
272	FOTE	'Forster's Tern'	(Sterna forsteri)
273	COTE	'Common Tern'	(Sterna hirundo)
274	ARTE	'Arctic Tern'	(Sterna paradisaea)
275	RSTE	'Roseate Tern'	(Sterna dougallii)
276	ALTE	'Aleutian Tern'	(Sterna aleutica)
277	SOTE	'Sooty Tern'	(Sterna fuscata)
278	BRTE	'Bridled Tern'	(Sterna anaethetus)
279	LETE	'Least Tern'	(Sterna antillarum)
280	ROTE	'Royal Tern'	(Sterna maxima)
281	ELTE	'Elegant Tern'	(Sterna elegans)
282	SATE	'Sandwich Tern'	(Sterna sandvicensis)
283	CATE	'Caspian Tern'	(Sterna caspia)
284	BLTE	'Black Tern'	(Chlidonias niger)
285	WWBT	'White-winged Black Tern'	(Chlidonias leucopterus)
286	BRNO	'Brown Noddy'	(Anous stolidus)
287	BLNO	'Black Noddy'	(Anous minutus)
288	HYTE	'Hybrid Tern'	(Sterna spp)
	UNGU	'Unidentified Gull/Tern'	

SKIMMER

289	BLSK	'Black Skimmer'	(Rynchops niger)

ALCIDS

290	RAZO	'Razorbill'	(Alca torda)
291	COMU	'Common Murre'	(Uria aalge)
292	TBMU	'Thick-billed Murre'	(Uria lomvia)
293	DOVE	'Dovekie'	(Alle alle)
294	BLGU	'Black Guillemot'	(Cepphus grylle)
295	MAGU	'Mandt's Guillemot'	(Cepphus grylle mandti)
296	PIGU	'Pigeon Guillemot'	(Cepphus columba)
297	MAMU	'Marbled Murrelet'	(Brachyramphus marmoratus)
298	K'MU	'Kittlitz's Murrelet'	(Brachyramphus brevirostris)
29[illegible]	XU	'Xantus' Murrelet'	[illegible]nthliboramphus hypoleucus)

301	ANMU	'Ancient Murrelet'	(Synthliboramphus antiquus)
302	CAAU	'Cassin's Auklet'	(Ptychoramphus aleuticus)
303	PAAU	'Parakeet Auklet'	(Cyclorrhynchus psittacula)
304	CRAU	'Crested Auklet'	(Aethia cristatella)
305	LEAU	'Least Auklet'	(Aethia pusilla)
306	WHAU	'Whiskered Auklet'	(Aethia pygmaea)
307	RHAU	'Rhinoceros Auklet'	(Cerorhinca monocerata)
308	ATPU	'Atlantic Puffin'	(Fratercula arctica)
309	HOPU	'Horned Puffin'	(Fratercula corniculata)
310	TUPU	'Tufted Puffin'	(Fratercula cirrhata)

PIGEONS, DOVES

311	WCPI	'White-crowned Pigeon'	(Columba leucocephala)
312	BTPI	'Band-tailed Pigeon'	(Columba fasciata)
313	RBPI	'Red-billed Pigeon'	(Columba flavirostris)
314	ZEDO	'Zenaida Dove'	(Zenaida aurita zenaida)
315	WWDO	'White-winged Dove'	(Zenaida asiatica)
316	MODO	'Mourning Dove'	(Zenaida macroura)
317	RODO	'Rock Dove'	(Columba livia)
318	SPDO	'Spotted Dove'	(Streptopelia chinensis)
319	RTDO	'Ringed Turtle Dove'	(Streptopelia risoria)
320	CGDO	'Common Ground Dove'	(Columbina passerina)
321	RGDO	'Ruddy Ground Dove'	(Columbina talpacoti)
322	INDO	'Inca Dove'	(Columbina inca)
323	WTDO	'White-tipped Dove'	(Leptotila verreauxi)
324	KWQD	'Key West Quail Dove'	(Oreopeleia chrysia)
325	RQDO	'Ruddy Quail Dove'	(Oreopeleia montana montana)
	UNDO	'Unidentified Dove'	

PARROT

326	CWPA	'Canary-winged Parakeet'	(Brotogeris versicolurus)

CUCKOOS, ROADRUNNER, ANIS

327	MACU	'Mangrove Cuckoo'	(Coccyzus minor)
328	YBCU	'Yellow-billed Cuckoo'	(Coccyzus americanus)
329	BBCU	'Black-billed Cuckoo'	(Coccyzus erythropthalmus)
330	GRRO	'Greater Roadrunner'	(Geococcyx californianus)
331	SBAN	'Smooth-billed Ani'	(Crotophaga ani)
332	GBAN	'Groove-billed Ani'	(Crotophaga sulcirostris)

BARN OWL

333	CBOW	'Common Barn Owl'	(Tyto alba)

TYPICAL OWLS

334	ESOW	'Eastern Screech-Owl'	(Otus asio)
335	WSOW	'Western Screech-Owl'	(Otus kennicottii)
336	WHOW	'Whiskered Owl'	(Otus trichopsis)
337	FLOW	'Flammulated Owl'	(Otus flammeolus)

339	SNOW	'Snowy Owl'	(Nyctea scandiaca)
340	NHOW	'Northern Hawk-Owl'	(Surnia ulula)
341	NPOW	'Northern Pygmy-Owl'	(Glaucidium gnoma)
342	FPOW	'Ferruginous Pygmy-Owl'	(Glaucidium brasilianum)
343	ELOW	'Elf Owl'	(Micrathene whitneyi)
344	BUOW	'Burrowing Owl'	(Athene cunicularia)
345	BDOW	'Barred Owl'	(Strix varia)
346	SPOW	'Spotted Owl'	(Strix occidentalis)
347	GGOW	'Great Gray Owl'	(Strix nebulosa)
348	LEOW	'Long-eared Owl'	(Asio otus)
349	SEOW	'Short-eared Owl'	(Asio flammeus)
350	BOOW	'Boreal Owl'	(Aegolis funereus)
351	NSWO	'Northern Saw-whet Owl'	(Aegolius acadicus)
	UNOW	'Unidentified Owl'	

GOATSUCKERS

352	CWWI	'Chuck-will's-widow'	(Caprimulgus carolinensis)
353	WPWI	'Whip-poor-will'	(Caprimulgus vociferus)
354	CPWI	'Common Poor-will'	(Phalaenoptilus nuttallii)
355	COPA	'Common Pauraque'	(Nyctidromus albicollis)
356	CONI	'Common Nighthawk'	(Chordeiles minor)
357	LENI	'Lesser Nighthawk'	(Chordeiles acutipennis)
	UNGO	'Unidentified Goatsucker'	

SWIFTS

358	BLSW	'Black Swift'	(Cypseloides niger)
359	CHSW	'Chimney Swift'	(Chaetura pelagica)
360	VASW	'Vaux's Swift'	(Chaetura vauxi)
361	WTSW	'White-throated Swift'	(Aeronautes saxatalis)

HUMMINGBIRDS

362	LUHU	'Lucifer Hummingbird'	(Calothorax lucifer)
363	RTHU	'Ruby-throated Hummingbird'	(Archilochus colubris)
364	BCHU	'Black-chinned Hummingbird'	(Archilochus alexandri)
365	COHU	'Costa's Hummingbird'	(Calypte costae)
366	ANHU	'Anna's Hummingbird'	(Calypte anna)
367	BRHU	'Broad-tailed Hummingbird'	(Selasphorus platycercus)
368	RUHU	'Rufous Hummingbird'	(Selasphorus rufus)
369	ALHU	'Allen's Hummingbird'	(Selasphorus sasin)
370	CAHU	'Calliope Hummingbird'	(Stellula calliope)
371	RIHU	'Rivoli's Hummingbird'	(Eugenes fulgens)
372	BUHU	'Blue-throated Hummingbird'	(Lampornis clemenciae)
373	BFHU	'Buff-bellied Hummingbird'	(Amazilia yucatanensis)
374	BBHU	'Broad-billed Hummingbird'	(Cynanthus latirostis)
	UNHU	'Unidentified Hummingbird'	

TROGON

KINGFISHERS

376	BEKI	'Belted Kingfisher'	(Ceryle alcyon)
377	RIKI	'Ringed Kingfisher'	(Ceryle torquata)
378	GNKI	'Green Kingfisher'	(Chloroceryle americana)

WOODPECKERS

379	NOFL	'Northern Flicker'	(Colaptes auratus)
380	PIWO	'Pileated Woodpecker'	(Dryocopus pileatus)
381	RBWO	'Red-bellied Woodpecker'	(Melanerpes carolinus)
382	GFWO	'Golden-fronted Woodpecker'	(Melanerpes aurifrons)
383	GIWO	'Gila Woodpecker'	(Melanerpes uropygialis)
384	RHWO	'Red-headed Woodpecker'	(Melanerpes erythrocephalus)
385	ACWO	'Acorn Woodpecker'	(Melanerpes formicivorus)
386	LEWO	'Lewis' Woodpecker'	(Melanerpes lewis)
387	YBSA	'Yellow-bellied Sapsucker'	(Sphyrapicus varius)
388	RBSA	'Red-breasted Sapsucker'	(Sphyrapicus ruber)
389	WISA	'Williamson's Sapsucker'	(Sphyrapicus thyroideus)
390	HAWO	'Hairy Woodpecker'	(Picoides villosus)
391	DOWO	'Downy Woodpecker'	(Picoides pubescens)
392	LBWO	'Ladder-backed Woodpecker'	(Picoides scalaris)
393	NUWO	'Nuttall's Woodpecker'	(Picoides nuttallii)
394	ARWO	'Arizona Woodpecker'	(Picoides stricklandi)
395	RCWO	'Red-cockaded Woodpecker'	(Picoides borealis)
396	WHWO	'White-headed Woodpecker'	(Picoides albolarvatus)
397	BBWO	'Black-backed Woodpecker'	(Picoides arctus)
398	TTWO	'Three-toed Woodpecker'	(Picoides tridactylus)
399	IBWO	'Ivory-billed Woodpecker'	(Campephilus principalis)
	UNWO	'Unidentified Woodpecker'	

COTINGA

400	RTBE	'Rose-throated Becard'	(Pachyramphus aglaiae)

TYRANT FLYCATCHERS

401	EAKI	'Eastern Kingbird'	(Tyrannus tyrannus)
402	GRKI	'Gray Kingbird'	(Tyrannus dominicensis)
403	TRKI	'Tropical Kingbird'	(Tyrannus melancholicus)
404	WEKI	'Western Kingbird'	(Tyrannus verticalis)
405	CAKI	'Cassin's Kingbird'	(Tyrannus vociferans)
406	TBKI	'Thick-billed Kingbird'	(Tyrannus crassirostris)
407	FTFL	'Fork-tailed Flycatcher'	(Tyrannus savana)
408	STFL	'Scissor-tailed Flycatcher'	(Tyrannus forficatus)
409	KIFL	'Greater Kiskadee'	(Pitangus sulphuratus)
410	SBFL	'Sulfur-bellied Flycatcher'	(Myiodynastes luteiventris)
411	GCFL	'Great Crested Flycatcher'	(Myiarchus crinitus)
412	BCFL	'Brown Crested Flycatcher'	(Myiarchus tyrannulus)
413	ATFL	'Ash-throated Flycatcher'	(Myiarchus cinerascens)
414	DCFL	'Dusky-capped Flycatcher'	(Myiarchus tuberculifer)
415	EAPH	'Eastern Phoebe'	(Sayornis phoebe)

417	SAPH	'Say's Phoebe'	(Sayornis saya)
418	YBFL	'Yellow-bellied Flycatcher'	(Empidonax flaviventris)
419	ACFL	'Acadian Flycatcher'	(Empidonax virescens)
420	WIFL	'Willow Flycatcher'	(Empidonax traillii)
421	ALFL	'Alder Flycatcher'	(Empidonax alnorum)
422	LEFL	'Least Flycatcher'	(Empidonax minimus)
423	HAFL	'Hammond's Flycatcher'	(Empidonax hammondii)
424	DUFL	'Dusky Flycatcher'	(Empidonax oberholseri)
425	GRFL	'Gray Flycatcher'	(Empidonax wrightii)
426	WEFL	'Western Flycatcher'	(Empidonax difficilis)
427	BBFL	'Buff-breasted Flycatcher'	(Empidonax fulvifrons)
	EMFL	'Unidentified Empidonax Flycatcher'	
428	COFL	'Cowes' Flycatcher'	(Contopus pertinax)
429	EWPE	'Eastern Wood-Pewee'	(Contopus virens)
430	WWPE	'Western Wood-Pewee'	(Contopus sordidulus)
431	OSFL	'Olive-sided Flycatcher'	(Contopus borealis)
432	VEFL	'Vermillion Flycatcher'	(Pyrocephalus rubinus)
433	NBTY	'Northern Beardless Tyrannulet'	(Camptostoma imberbe)
434	UNFL	'Unidentified Flycatcher'	

LARKS

435	EUSK	'Eurasian Skylark'	(Alauda arvensis)
436	HOLA	'Horned Lark'	(Eremophila alpestris)

SWALLOWS

437	VGSW	'Violet-green Swallow'	(Tachycineta thalassina)
438	TRSW	'Tree Swallow'	(Tachycineta bicolor)
439	BKSW	'Bank Swallow'	(Riparia riparia)
440	NRWS	'Northern Rough-winged Swallow'	(Stelgidopteryx serripennis)
441	BASW	'Barn Swallow'	(Hirundo rustica)
442	CLSW	'Cliff Swallow'	(Hirundo pyrrhonota)
443	CASW	'Cave Swallow'	(Hirundo fulva)
444	PUMA	'Purple Martin'	(Progne subis)
	UNSW	'Unidentified Swallow'	

JAYS, MAGPIES, CROWS

445	GRJA	'Gray Jay'	(Perisoreus canadensis)
446	BLJA	'Blue Jay'	(Cyanocitta cristata)
447	STJA	'Steller's Jay'	(Cyanocitta stelleri)
448	SCJA	'Scrub Jay'	(Aphelocoma coerulescens)
449	GBJA	'Gray-breasted Jay'	(Aphelocoma ultramarina)
450	GEJA	'Green Jay'	(Cyanocorax yncas)
451	BBMA	'Black-billed Magpie'	(Pica pica)
452	YBMA	'Yellow-billed Magpie'	(Pica nuttalli)
453	CORA	'Common Raven'	(Corvus corax)
454	CHRA	'Chihuahua Raven'	(Corvus cryptoleucus)
455	AMCR	'American Crow'	(Corvus brachyrhynchos)
456	NOCR	'Northwestern Crow'	(Corvus caurinus)
457	FICR	'Fish Crow'	(Corvus ossifragus)
	UNCR	'Unidentified Crow'	

459	CLNU	'Clark's Nutcracker'	(Nucifraga columbiana)

TITMICE, VERDIN, BUSHTIT

460	BCCH	'Black-capped Chickadee'	(Parus atricapillus)
461	CACH	'Carolina Chickadee'	(Parus carolinensis)
462	MECH	'Mexican Chickadee'	(Parus sclateri)
463	MOCH	'Mountain Chickadee'	(Parus gambeli)
464	SITI	'Siberian Tit'	(Parus cinctus)
465	BOCH	'Boreal Chickadee'	(Parus hudsonicus)
466	CBCH	'Chestnut-backed Chickadee'	(Parus rufescens)
467	TUTI	'Tufted Titmouse'	(Parus bicolor)
468	BCTI	'Black-crested Titmouse'	Tufted race
469	PLTI	'Plain Titmouse'	(Parus inornatus)
470	BRTI	'Bridled Titmouse'	(Parus wollweberi)
471	VERD	'Verdin'	(Auriparus flaviceps)
472	BUSH	'Bushtit'	(Psaltriparus minimus)

NUTHATCHES

473	WBNU	'White-breasted Nuthatch'	(Sitta carolinensis)
474	RBNU	'Red-breasted Nuthatch'	(Sitta canadensis)
475	BHNU	'Brown-headed Nuthatch'	(Sitta pusilla)
476	PYNU	'Pygmy Nuthatch'	(Sitta pygmaea)

CREEPER

477	BRCR	'Brown Creeper'	(Certhia americana)

WRENTIT

478	WREN	'Wrentit'	(Chamaea fasciata)

BULBUL

479	RWBU	'Red-whiskered Bulbul'	(Pycnonotus jocosus)

DIPPER

480	AMDI	'American Dipper'	(Cinclus mexicanus)

WRENS

481	HOWR	'House Wren'	(Troglodytes aedon)
482	WIWR	'Winter Wren'	(Troglodytes troglodytes)
483	BEWR	'Bewick's Wren'	(Thryomanes bewickii)
484	CAWR	'Carolina Wren'	(Thryothorus ludovicianus)
485	CTWR	'Cactus Wren'	(Campylorhynchus brunneicapillus)
486	MAWR	'Marsh Wren'	(Cistothorus palustris)
487	SEWR	'Sedge Wren'	(Cistothorus platensis)
488	CNWR	'Canyon Wren'	(Catherpes mexicanus)
489	ROWR	'Rock Wren'	(Salpinctes obsoletus)
	UNWR	'Unidentified Wren'	

MOCKINGBIRDS, THRASHERS

490 NOMO	'Northern Mockingbird'	(Mimus polyglottus)
491 GRCA	'Gray Catbird'	(Dumetella carolinensis)
492 BRTH	'Brown Thrasher'	(Toxostoma rufum)
493 LBTH	'Long-billed Thrasher'	(Toxostoma longirostre)
494 GRTH	'Gray Thrasher'	(Toxostoma cinereum)
495 BETH	'Bendire's Thrasher'	(Toxostoma bendirei)
496 CBTH	'Curve-billed Thrasher'	(Toxostoma curvirostre)
497 CATH	'California Thrasher'	(Toxostoma redivivum)
498 LCTH	'LeConte's Thrasher'	(Toxostoma lecontei)
499 CRTH	'Crissal Thrasher'	(Toxostoma dorsale)
500 SATH	'Sage Thrasher'	(Oreoscoptes montanus)
UNTH	'Unidentified Thrasher'	

THRUSHES

501 AMRO	'American Robin'	(Turdus migratorius)
502 FIEL	'Fieldfare'	(Turdus pilaris)
503 CCRO	'Clay-colored Robin'	(Turdus grayi)
504 VATH	'Varied Thrush'	(Ixoreus naevius)
505 WOTH	'Wood Thrush'	(Hylocichla mustelina)
506 HETH	'Hermit Thrush'	(Catharus guttatus)
507 SWTH	'Swainson's Thrush'	(Catharus ustulatus)
508 GCTH	'Gray-cheeked Thrush'	(Catharus minimus)
509 VEER	'Veery'	(Catharus fuscescens)
510 EABL	'Eastern Bluebird'	(Sialia sialis)
511 WEBL	'Western Bluebird'	(Sialia mexicana)
512 MOBL	'Mountain Bluebird'	(Sialia currucoides)
513 EMBL	'Eastern/Mountain Blueb.Hybrid'	(Sialia spp)
514 NOWH	'Northern Wheatear'	(Oenanthe oenanthe)
515 BLUE	'Bluethroat'	(Luscinia svecica)
516 TOSO	'Townsend's Solitaire'	(Myadestes townsendi)
UNTU	'Unidentified Thrush'	

OLD WORLD WARBLERS, GNATCATCHERS, KINGLETS

517 ARWA	'Arctic Warbler'	(Phylloscopus borealis)
518 BGGN	'Blue-gray Gnatcatcher'	(Polioptila caerulea)
519 BCGN	'Black-capped Gnatcatcher'	(Polioptila nigriceps)
520 BTGN	'Black-tailed Gnatcatcher'	(Polioptila melanura)
UNGN	'Unidentified Gnatcatcher'	
521 GCKI	'Golden-crowned Kinglet'	(Regulus satrapa)
522 RCKI	'Ruby-crowned Kinglet'	(Regulus calendula)

WAGTAILS, PIPITS

523 WHWA	'White Wagtail'	(Motacilla alba)
524 YLWA	'Yellow Wagtail'	(Motacilla flava)
525 WAPI	'Water Pipit'	(Anthus spinoletta)
526 RTPI	'Red-throated Pipit'	(Anthus cervinus)
527 SPPI	'Sprague's Pipit'	(Anthus spragueii)

WAXWINGS

528 BOWA	'Bohemian Waxwing'	(Bombycilla garrulus)
529 CEWA	'Cedar Waxwing'	(Bombycilla cedrorum)

SILKY FLYCATCHER

530 PHAI	'Phainopepla'	(Phainopepla nitens)

SHRIKES

531 NRSH	'Northern Shrike'	(Lanius excubitor)
532 LOSH	'Loggerhead Shrike'	(Lanius ludovicianus)

STARLING

533 EUST	'European Starling'	(Sturnus vulgaris)

VIREOS

534 BCVI	'Black-capped Vireo'	(Vireo atricapillus)
535 WEVI	'White-eyed Vireo'	(Vireo griseus)
536 HUVI	'Hutton's Vireo'	(Vireo huttoni)
537 BEVI	'Bell's Vireo'	(Vireo bellii)
538 GRVI	'Gray Vireo'	(Vireo vicinior)
539 YTVI	'Yellow-throated Vireo'	(Vireo flavifrons)
540 SOVI	'Solitary Vireo'	(Vireo solitarius)
541 BWVI	'Black-whiskered Vireo'	(Vireo altiloquus)
542 REVI	'Red-eyed Vireo'	(Vireo olivaceus)
543 YGVI	'Yellow-green Vireo'	(Vireo olivaceus flavoviridis)
544 PHVI	'Philadelphia Vireo'	(Vireo philadelphicus)
545 WAVI	'Warbling Vireo'	(Vireo gilvus)
UNVI	'Unidentified Vireo'	

WOOD WARBLERS

546 BAWW	'Black-and-white Warbler'	(Mniotilta varia)
547 POWA	'Prothonotary Warbler'	(Protonotaria citrea)
548 SWWA	'Swainson's Warbler'	(Limnothlypis swainsonii)
549 WEWA	'Worm-eating Warbler'	(Helmitheros vermivorus)
550 GWWA	'Golden-winged Warbler'	(Vermivora chrysoptera)
551 BWWA	'Blue-winged Warbler'	(Vermivora pinus)
552 BRWA	'Brewster's Warbler'	Golden-winged/Blue-winged Hybrid
553 LAWA	'Lawrence's Warbler'	Golden-winged/Blue-winged Hybrid
554 BAWA	'Bachman's Warbler'	(Vermivora bachmanii)
555 TEWA	'Tennessee Warbler'	(Vermivora peregrina)
556 OCWA	'Orange-crowned Warbler'	(Vermivora celata)
557 NAWA	'Nashville Warbler'	(Vermivora ruficapilla)
558 VIWA	'Virginia's Warbler'	(Vermivora virginiae)
559 CLWA	'Colima Warbler'	(Vermivora crissalis)
560 LUWA	'Lucy's Warbler'	(Vermivora luciae)
561 NOPA	'Northern Parula'	(Parula americana)

563	OLWA	'Olive Warbler'	(Peucedramus taeniatus)
564	YEWA	'Yellow Warbler'	(Dendroica petechia)
565	MAWA	'Magnolia Warbler'	(Dendroica magnolia)
566	CMWA	'Cape May Warbler'	(Dendroica tigrina)
567	BTBW	'Black-throated Blue Warbler'	(Dendroica caerulescens)
568	YRWA	'Yellow-rumped Warbler'	(Dendroica coronata)
569	BGWA	'Black-throated Gray Warbler'	(Dendroica nigrescens)
570	TOWA	'Townsend's Warbler'	(Dendroica townsendi)
571	BTGW	'Black-throated Green Warbler'	(Dendroica virens)
572	GCWA	'Golden-cheeked Warbler'	(Dendroica chrysoparia)
573	HEWA	'Hermit Warbler'	(Dendroica occidentalis)
574	CRWA	'Cerulean Warbler'	(Dendroica cerulea)
575	BLWA	'Blackburnian Warbler'	(Dendroica fusca)
576	YTWA	'Yellow-throated Warbler'	(Dendroica dominica)
577	SUWA	'Sutton's Warbler'	(Dendroica potomac)
578	GRWA	'Grace's Warbler'	(Dendroica graciae)
579	CSWA	'Chestnut-sided Warbler'	(Dendroica pensylvanica)
580	BBWA	'Bay-breasted Warbler'	(Dendroica castanea)
581	BPWA	'Blackpoll Warbler'	(Dendroica striata)
582	PIWA	'Pine Warbler'	(Dendroica pinus)
583	KIWA	'Kirtland's Warbler'	(Dendroica kirtlandii)
584	PRWA	'Prairie Warbler'	(Dendroica discolor)
585	PAWA	'Palm Warbler'	(Dendroica palmarum)
586	OVEN	'Ovenbird'	(Seiurus aurocapillus)
587	NOWA	'Northern Waterthrush'	(Seiurus noveboracesis)
588	LOWA	'Louisiana Waterthrush'	(Seiurus motacilla)
589	KEWA	'Kentucky Warbler'	(Oporornis formosus)
590	COWA	'Connecticut Warbler'	(Oporornis agilis)
591	MOWA	'Mourning Warbler'	(Oporornis philadelphia)
592	MGWA	'MacGillivray's Warbler'	(Oporornis tolmiei)
593	COYE	'Common Yellowthroat'	(Geothlypis trichas)
594	GRCH	'Ground Chat'	(Geothlypis poliocephala)
595	YBCH	'Yellow-breasted Chat'	(Icteria virens)
596	RFWA	'Red-faced Warbler'	(Cardellina rubrifrons)
597	HOWA	'Hooded Warbler'	(Wilsonia citrina)
598	WIWA	'Wilson's Warbler'	(Wilsonia pusilla)
599	CAWA	'Canada Warbler'	(Wilsonia canadensis)
600	AMRE	'American Redstart'	(Setophaga ruticilla)
601	PARE	'Painted Redstart'	(Myioborus pictus)
	UNWA	'Unidentified Warbler'	

WEAVER FINCHES

602	HOSP	'House Sparrow'	(Passer domesticus)
603	ETSP	'Eurasian Tree Sparrow'	(Passer montanus)

MEADOWLARKS, BLACKBIRDS, ORIOLES

604	BOBO	'Bobolink'	(Dolichonyx oryzivorus)
605	EAME	'Eastern Meadowlark'	(Sturnella magna)
606	WEME	'Western Meadowlark'	(Sturnella neglecta)
[illegible]	YHBL	'Yellow-headed Blackbird'	(Xanthocephalus xanthocephalus)
608	RWBL	'Red-winged Blackbird'	(Agelaius phoeniceus)

610	OROR	'Orchard Oriole'	(Icterus spurius)
611	AUOR	'Audubon's Oriole'	(Icterus graduacauda)
612	SBOR	'Spotted-breasted Oriole'	(Icterus pectoralis)
613	STOR	'Streak-backed Oriole'	(Icterus pustulatus)
614	HOOR	'Hooded Oriole'	(Icterus cucullatus)
615	ALOR	'Altamira Oriole'	(Icterus gularis)
616	SCOR	'Scott's Oriole'	(Icterus parisorum)
617	NOOR	'Northern Oriole'	(Icterus galbula)
	UNOR	'Unidentified Oriole'	
618	RUBL	'Rusty Blackbird'	(Euphagus carolinus)
619	BRBL	'Brewer's Blackbird'	(Euphagus cyanocephalus)
620	GTGR	'Great-tailed Grackle'	(Quiscalus mexicanus)
621	BTGR	'Boat-tailed Grackle'	(Quiscalus major)
622	COGR	'Common Grackle'	(Quiscalus quiscula)
623	BHCO	'Brown-headed Cowbird'	(Molothrus ater)
624	BRCO	'Bronzed Cowbird'	(Molothrus aeneus)
625	UNBL	'Unidentified Blackbird'	

TANAGERS

626	WETA	'Western Tanager'	(Piranga ludoviciana)
627	SCTA	'Scarlet Tanager'	(Piranga olivacea)
628	HETA	'Hepatic Tanager'	(Piranga flava)
629	SUTA	'Summer Tanager'	(Piranga rubra)
	UNTA	'Unidentified Tanager'	

GROSBEAKS, FINCHES, SPARROWS, BUNTINGS

630	NOCA	'Northern Cardinal'	(Cardinalis cardinalis)
631	PYRR	'Pyrrhuloxia'	(Cardinalis sinuatus)
632	RBGR	'Rose-breasted Grosbeak'	(Pheucticus ludovicianus)
633	BHGR	'Black-headed Grosbeak'	(Pheucticus melanocephalus)
634	BLGR	'Blue Grosbeak'	(Guiraca caerulea)
	UNGR	'Unidentified Grosbeak'	
635	INBU	'Indigo Bunting'	(Passerina cyanea)
636	LABU	'Lazuli Bunting'	(Passerina amoena)
637	VABU	'Varied Bunting'	(Passerina versicolor)
638	PABU	'Painted Bunting'	(Passerina ciris)
639	DICK	'Dickcissel'	(Spiza americana)
640	EVGR	'Evening Grosbeak'	(Coccothraustes vespertinus)
641	PUFI	'Purple Finch'	(Carpodacus purpureus)
642	CAFI	'Cassin's Finch'	(Carpodacus cassinii)
643	HOFI	'House Finch'	(Carpodacus mexicanus)
644	WCSE	'White-collared Seedeater'	(Sporophila torqueola)
645	PIGR	'Pine Grosbeak'	(Pinicola enucleator)
646	ROFI	'Rosy Finch'	(Leucosticte arctoa)
647	EUGO	'European Goldfinch'	(Carduelis carduelis)
648	HORE	'Hoary Redpoll'	(Carduelis hornemanni)
[illegible]	[illegible]	[illegible]	[illegible]
650	PISI	'Pine Siskin'	(Carduelis pinus)

653 LAGO	Lawrence's Goldfinch	(Carduelis lawrencei)
654 RECR	Red Crossbill	(Loxia curvirostra)
655 WWCR	'White-winged Crossbill'	(Loxia leucoptera)
656 OLSP	'Olive Sparrow'	(Arremonops rufivirgatus)
657 GTTO	'Green-tailed Towhee'	(Pipilo chlorurus)
658 RSTO	'Rufous-sided Towhee'	(Pipilo erythrophthalmus)
659 BRTO	'Brown Towhee'	(Pipilo fuscus)
660 ABTO	'Abert's Towhee'	(Pipilo aberti)
661 LKBU	'Lark Bunting'	(Calamospiza melanocorys)
662 SASP	'Savannah Sparrow'	(Passerculus sandwichensis)
663 GRSP	'Grasshopper Sparrow'	(Ammodramus savannarum)
664 BDSP	'Baird's Sparrow'	(Ammodramus bairdii)
665 LCSP	'LeConte's Sparrow'	(Ammodramus leconteii)
666 HESP	'Henslow's Sparrow'	(Ammodramus henslowii)
667 STSP	'Sharp-tailed Sparrow'	(Ammodramus caudacutus)
668 SESP	'Seaside Sparrow'	(Ammodramus maritimus)
669 VESP	'Vesper Sparrow'	(Pooecetes gramineus)
670 LASP	'Lark Sparrow'	(Chondestes grammacus)
671 FSSP	'Five-striped Sparrow'	(Amphispiza quinquestriata)
672 RWSP	'Rufous-winged Sparrow'	(Aimophila carpalis)
673 RCSP	'Rufous-crowned Sparrow'	(Aimophila ruficeps)
674 BASP	'Bachman's Sparrow'	(Aimophila aestivalis)
675 BOSP	'Botteri's Sparrow'	(Aimophila botterii)
676 CASP	'Cassin's Sparrow'	(Aimophila cassinii)
677 BTSP	'Black-throated Sparrow'	(Amphispiza bilineata)
678 SGSP	'Sage Sparrow'	(Amphispiza belli)
679 DEJU	'Dark-eyed Junco'	(Junco hyemalis)
680 YEJU	'Yellow-eyed Junco'	(Junco phaeonotus)
681 ATSP	'American Tree Sparrow'	(Spizella arborea)
682 CHSP	'Chipping Sparrow'	(Spizella passerina)
683 CCSP	'Clay-colored Sparrow'	(Spizella pallida)
684 BRSP	'Brewer's Sparrow'	(Spizella breweri)
685 FISP	'Field Sparrow'	(Spizella pusilla)
686 BCSP	'Black-chinned Sparrow'	(Spizella atrogularis)
687 HASP	'Harris' Sparrow'	(Zonotrichia querula)
688 WCSP	'White-crowned Sparrow'	(Zonotrichia leucophrys)
689 GCSP	'Golden-crowned Sparrow'	(Zonotrichia atricapilla)
690 WTSP	'White-throated Sparrw'	(Zonotrichia albicollis)
691 FOSP	'Fox Sparrow'	(Passerella iliaca)
692 LISP	'Lincoln's Sparrow'	(Melospiza lincolnii)
693 SWSP	'Swamp Sparrow'	(Melospiza georgiana)
694 SOSP	'Song Sparrow'	(Melospiza melodia)
695 MCLO	'McCown's Longspur'	(Calcarius mccownii)
696 LALO	'Lapland Longspur'	(Calcarius lapponicus)
697 SMLO	'Smith's Longspur'	(Calcarius pictus)
698 CCLO	'Chestnut-collared Longspur'	(Calcarius ornatus)
699 SNBU	'Snow Bunting'	(Plectrophenax nivalis)
700 MCBU	'McKay's Bunting'	(Plectrophenax hyperboreus)
701 UNSP	'Unidentified Sparrow'	

Appendix B
Field Activities Procedures
Kress Creek Site

CONTENTS

CONTENTS (Continued)

Title: Chemical Field Screening

Revision No.: 0

Date: 1/11/93

Chemical Field Screening

1.0 HNu MONITORING

1.1 References

HNu Model PI101 Portable Photoionization Analyzer Instruction Manual, December 1985, HNu Systems, Inc., Newton, Massachusetts

HNu Model ISP1 101 Intrinsically Safe Portable Photoionization Analyzer Instruction Manual, January 1986, HNu Systems, Inc., Newton, Massachusetts

1.2 Sensitivity

0 to 20 parts per million (ppm) at full-scale detection at span = 9.8 ppm; 10.2 eV Probe.

1.3 Range

0.1 to 2,000 ppm.

1.4 Calibration Details

1.4.1 Calibration Gas

Isobutylene at 100 ppm.

1.4.2 Calibration

By analyzing a gas of known concentration, the HNu is calibrated. Isobutylene is typically used as the calibration gas with the instrument calibrated to benzene equivalents. When calibrating the HNu, remember to deliver the calibration gas at ambient temperature and pressure, handle gas cylinders with care, and calibrate every day. Also, the calibration gas must be stable during the period of use; gas cylinders must have regulators.

1.4.3 Calibration Procedure

1. Identify the probe by lamp label.

2. Attach the probe to the readout unit. Twist connector clockwise until locked.

3. Affirm the relative photoionization sensitivity (PS) calibration gas. [The correct reading for isobutylene to read in benzene equivalents is equal to isobutylene ppm × PS (Isob.)/PS (benzene).]

4. Turn the function switch to battery check position. The indicator should read within the green arc. If indicator is below the green arc or if red LED comes on, battery must be charged.

5. Zero the instrument by turning function switch to standby and rotate potentiometer until the meter reads zero.

6. Connect sampling hose to regulator outlet and the other end to sampling probe of HNu.

7. Crack regulator valve.

8. Adjust span potentiometer to obtain proper reading.

9. If calibration cannot be achieved, clean the UV light source window using lens paper and HNu cleaning compound.

10. If still unable to calibrate, perform preventive maintenance. Return to factory if those procedures do not work.

1.4.4 Calibration Frequency

Daily or after maintenance. Recharge battery after each use. Factory checkout and calibration shall be yearly or when malfunctioning.

1.5 Preventive Maintenance (Frequency)

The HNu is inspected and calibrated for correct operations in the warehouse prior to delivery to the field.

1.5.1 Battery (Daily)

Check the battery charge during each period of operation. When the meter needle falls below the green zone or the low-battery indicator light illuminates, recharge battery. Do not use the instrument when light is on. When not operating, leave the analyzer assembled and connected to the battery charger. In case of emergency, the analyzer may be used with a low-battery charge.

1.5.2 Gas Cylinders and Valves (After installation)

Gas supply lines must be leak-tested. Leakage can be determined by testing line connections and valve stems with a commercially available leak test solution. Leaks are generally stopped by tightening the fitted surfaces.

1.5.3 Air Sampling Stream (Initially, then as needed)

Leaks that develop in this system may result in dilution or loss of sample, causing erroneous vapor concentrations and slow response. A fan draws gas in through the probe and ion chamber. Small fluctuations in the flow rate will not affect the measurement. A major obstruction to the flow rate will prevent operation and lengthen response time. Refer to the manufacturer's instrument manual for specific procedures.

1.6 Quality Control Requirements

Precision of ± 30 percent. Daily calibration.

2.0 OVA MONITORING

2.1 Reference

Model OVA 128 Century Organic Vapor Analyzer, Instruction, December 1985, Foxboro, New Haven, Connecticut

2.2 Sensitivity

0.1 ppm (methane).

2.3 Range

0 to 1,000 ppm.

2.4 Calibration Details

2.4.1 Calibration Gas

Methane gas at 100 ppm.

2.4.2 Calibration

By analyzing a gas of known concentration, the OVA is calibrated. Methane in air at a concentration of 100 ppm is typically used as the calibration mixture, although the OVA can be calibrated to many other compounds. Primary calibration of an OVA is performed at the factory. When calibrating the OVA, remember to deliver the calibration gas at ambient temperature and pressure, handle the gas cylinders with care, and calibrate every day. Also, the calibration gas must be stable during the period of use, and gas cylinders must have regulators.

2.4.3 Calibration Procedure

1. Connect probe readout assembly to sidepack unit.
2. Check battery condition by moving INSTR Switch to BATT.
3. Turn INSTR to ON and allow 5 min to warm up.
4. Use calibration adjust knob to set needle to level desired for activating alarm. If alarm level is not zero, the calibration switch must be set to appropriate level.
5. Turn volume knob fully clockwise.
6. Turn the alarm level adjust knob until the audible alarm is activated.
7. Move calibration switch to 1X̂ and adjust meter reading to zero using zero calibration adjustment.
8. Turn pump switch on.

9. Open hydrogen tank valve and hydrogen supply valve. Wait 1 min.

10. Depress ignitor button until burner lights (not more than 6 sec).

11. Set calibration switch to 10X.

12. Connect sampling hose to regulator outlet and the other end to sampling probe of OVA.

13. Crack the regulator valve.

14. Check to see if proper reading is achieved.

15. If reading is ± 10 percent from expected value, return to factory for recalibration.

2.4.4 Calibration Frequency

Daily or after maintenance, recharge battery after each use. Factory checkout and calibration shall be yearly or when malfunctioning.

2.5 Preventive Maintenance

The OVA is inspected and calibrated in the warehouse prior to delivery to the field.

2.5.1 Battery (Daily)

Check battery condition by moving the INSTR switch to the BATT position. Recharge the battery if a low charge is indicated. Do not use the instrument with a low battery charge. When not operating, leave the analyzer connected to a battery charger. Never recharge battery in a hazardous environment.

2.5.2 Particle Filter (As needed)

Particle filters (primary and secondary) remove foreign matter (> 10 microns) from the sample stream. These filters must be in the sample line whenever the instrument is operating. A decrease in flow rate may indicate a plugged filter.

2.5.3 Sampling Fixtures (As needed)

Sampling fixtures should be periodically cleaned with an air hose and/or detergent water to eliminate foreign particulate matter.

2.5.4 Hydrogen Fuel, Calibration Gas, and Valves (Initially, after changes)

Use prepurified or zero-grade hydrogen (certified total hydrocarbons as methane < 0.5 ppm recommended). Fuel and calibration gas supply lines should be leak tested. Leakage can be checked by testing line connections and valve stems with a commercially available leak test solution. Leaks are usually stopped by tightening the fitted surfaces.

2.5.5 Air Sampling Pump System (Initially, as needed)

Leaks that develop in this system may result in dilution or loss of sample, causing erroneous vapor concentrations and slow response. The OVA is equipped with a flow gauge that provides a method to check for air leaks. Refer to the manufacturer's instrument manual for specific procedures.

2.5.6 Contaminated Control (As needed)

Background readings may be relatively high under normal ambient conditions. The sources of high background are normal methane background, contaminated hydrogen supply gas, and contamination in the air sample line. Background readings less than 1 ppm are generally accepted since sample measurement is additive to that background. However, the low background values are more desirable. High background is commonly corrected by running the OVA for an extended time in a clean area. Refer to the manufacturer's instrument manual for specific analysis and correction procedures.

2.6 Quality Control Requirements

Precision of ± 30 percent. Daily calibration.

Title: Soil Boring Log Guidelines

Revision No.: 0

Date: 1/11/93

Soil Boring Log Guidelines

1.0 SOIL BORING LOG POLICY

This soil boring guideline will be used for CH2M HILL projects in which soil boring techniques are used during field exploration. The purpose of the guideline is to assist CH2M HILL staff in accurately recording and presenting field data to describe, label, and package recovered soil samples in a consistent manner. The guideline establishes the minimum kinds of information that must be recorded in the field to adequately characterize recovered soil samples from borings greater than 1 ft.

2.0 RECORDING SOIL BORING FIELD DATA

CH2M HILL Standard Form D1586, the Soil Boring Log form, will be used on CH2M HILL projects for field logging (see Figure 1). Adherence to a standard format for recording data will help streamline project efforts and provide a consistent presentation of factual subsurface data. Heading information must be completely filled out on each log sheet used, and technical items in each column must be addressed in the field.

The boring log should be completed in the field according to the attached instructions. Forms should be filled out neatly and completely. Laboratory testing, if required, should be initiated immediately after completion of the fieldwork. Field classifications of samples should be checked against the laboratory test results, and corrections should be noted, initialed, and dated on the field log.

3.0 INSTRUCTIONS FOR COMPLETING SOIL BORING LOG, FORM D1586

Form D1586 is a standard CH2M HILL form that is available in weatherproof paper from regional form distributors.

Following are instructions for completing the log forms in the field.

Field personnel should review logs on completion for accuracy, clarity, and thoroughness of detail. Samples should also be checked to see that information is correctly recorded on both jar lids and labels and on the log sheets.

If changes to the soil classification are made on the log forms after completion of the fieldwork, they should be done in red, then initialed and dated.

3.1 Heading Information

3.1.1 Project Number

Use standard region code, contract ID (five-digit), and point number designated for field exploration or geotechnical services.

3.1.2 Boring Number

Enter the boring number. Number the sheets consecutively for each boring.

CH2M HILL

PROJECT NUMBER | BORING NUMBER | SHEET | OF

SOIL BORING LOG

PROJECT _____ LOCATION _____

ELEVATION _____ DRILLING CONTRACTOR _____

DRILLING METHOD AND EQUIPMENT _____

WATER LEVELS _____ START _____ FINISH _____ LOGGER _____

DEPTH BELOW SURFACE (FT)	SAMPLE			STANDARD PENETRATION TEST RESULTS	SOIL DESCRIPTION	COMMENTS
	INTERVAL	NUMBER AND TYPE	RECOVERY (FT)	6"-6"-6" N	SOIL NAME, USCS GROUP SYMBOL, COLOR, MOISTURE CONTENT, RELATIVE DENSITY OR CONSISTENCY, SOIL STRUCTURE, MINERALOGY	DEPTH OF CASING, DRILLING RATE, DRILLING FLUID LOSS, TESTS AND INSTRUMENTATION

8.301

REV 11.89 FORM 01586

Figure 1
CH2M HILL Standard Soil Boring Log Form
Kress Creek RI/FS Field Procedures

3.1.3 Project

Fill in the name of the project or client [e.g., Kress Creek Remedial Investigation/Feasibility Study (RI/FS)].

3.1.4 Location

If stationing, coordinates, mileposts, or similar project layout information is available, indicate the position of the boring with regard to that system, using modifiers such as "approximate" or "estimated." If this information is not available, identify the facility (e.g., Kress Creek—with relation to a crossroad).

3.1.5 Date of Start and Finish

Enter the dates when the boring was begun and completed. Time of day will be added if several borings are performed on the same day.

3.1.6 Logger/Sampler

Enter first initial and full last name.

3.2 Technical Data

3.2.1 Depth Below Surface

Use a depth scale that is appropriate for the sample spacing and for the complexity of subsurface conditions.

3.2.2 Sample Interval

Draw horizontal lines at the top and bottom depth of each sample interval. These lines should extend to the soil description column. For a very short sample interval, the bottom line can be lowered after the interval column to provide room for writing the information. Enter the depth at the top and bottom of the sample interval.

3.2.3 Sample Type and Number

Enter the sample type and number as presented in Section 3.0 of the Field Sampling Plan (FSP) (Appendix A).

3.2.4 Soil Description

The soil classification should follow the format described in the section below entitled Field Classification of Soil.

3.2.5 Symbolic Log

Leave this column blank during fieldwork.

3.3 Field Classification of Soil

Soil descriptions should be precise and comprehensive without being verbose. The correct overall impression of the soil should not be distorted by excessive emphasis on

insignificant details. In general, similarities between consecutive samples should be stressed rather than differences.

Soil descriptions shall be recorded in the Soil Description column for every soil sample collected. The format and order for soil descriptions should be:

1. Soil name [synonymous with American Society for Testing and Materials (ASTM) D 2487-85 Group Name)] with appropriate modifiers

2. Color

3. Moisture content

4. Relative density or consistency

5. Soil structure or mineralogy

6. Group symbol

This order follows, in general, the format described in ASTM D 2488-84. Examples of soil descriptions are provided in Table 1.

3.3.1 Soil Name

The basic name of a soil shall be identical to the ASTM D 2487-85 Group Name based on visual estimates of gradation and plasticity. The soil name should be capitalized. The only acceptable soil names are those from ASTM D 2487-85.

Examples of acceptable soil names are illustrated by the following:

> A soil sample is visually estimated to contain 15 percent gravel, 55 percent sand, and 30 percent fines (passing No. 200 sieve). The fines are estimated as either low or highly plastic silt. This sample is SILTY SAND WITH GRAVEL, with a Group Symbol of (SM).
>
> Another soil sample has the following visual estimate: 10 percent gravel, 30 percent sand, and 60 percent fines (passing the No. 200 sieve). The fines are estimated as low plastic silt. This sample is SANDY SILT. The gravel portion is not included in the soil name because the gravel portion was estimated as less than 15 percent. The Group Symbol is (ML).

The gradation of coarse-grained soil (more than 50 percent retained on No. 200 sieve) is included in the specific soil name in accordance with ASTM D 2487-85. The maximum size and angularity or roundedness of gravel and sand-sized particles should be recorded. For fine-grained soil (50 percent or more passing the No. 200 sieve), the name is modified by the appropriate plasticity/elasticity term in accordance with ASTM D 2487-85.

Interlayered soil should each be described starting with the predominant type. An introductory name should be used such as "Interlayered Sand and Silt." Also, the relative proportion of each soil type should be indicated (see Table 1 for example).

Table 1
Exampie Soil Descriptions

POORLY GRADED SAND, fine, well-rounded, light brown, moist, loose (SP)

FAT CLAY, dark gray, moist, stiff (CH)

SILT, light greenish gray, wet, very loose, some mica, lacustrine (ML)

WELL-GRADED SAND WITH GRAVEL, subangular gravel to 0.6 in. maximum, reddish brown, moist, dense (SW)

POORLY GRADED SAND WITH SILT, white, wet, medium dense (SP-SM)

ORGANIC SILT WITH SAND, dark brown to black, wet, firm to stiff but spongy undisturbed, becomes soft and sticky when remolded, many fine roots, trace of mica (OH)

SILTY GRAVEL WITH SAND, subrounded gravel to 1.2 in. maximum, brownish red, moist, very dense (GM)

INTERLAYERED SILT (60 percent) AND CLAY (40 percent): SILT WITH SAND, nonplastic, sudden reaction to shaking, medium greenish gray, layers mostly 1.5 to 8.3 in. thick; CLAY, dark gray, firm and brittle undisturbed, becomes very soft and sticky when remolded, layers 0.2 to 1.2 in. thick (ML and CH)

SILTY SAND WITH GRAVEL, weak gravel to 1.0 in. maximum, light yellowish brown, compact, moist, very few small particles of coal, fill (SM)

SANDY ELASTIC SILT, very light gray to white, wet, stiff, weak calcareous cementation (MH)

SILTY CLAY WITH SAND, dark brownish gray, moist, stiff (ML-CL)

WELL-GRADED GRAVEL WITH SILT, rounded gravel to 1.0 in. maximum, brown, moist, very dense (GW-GM)

Where helpful, the evaluation of plasticity/elasticity can be justified by describing results from the visual-manual procedures for identifying fine-grained soils, such as reaction to shaking, toughness of a soil thread, or dry strength as described in ASTM D 2488-84.

3.3.2 Color

The basic color of a soil, such as brown, gray, or red, shall be given. The color term can be modified by adjectives such as light, dark, or mottled. The color description should be kept simple and should not emphasize unimportant color aspects or shades.

3.3.3 Moisture Content

The degree of moisture present in a soil sample should be defined as dry, moist, or wet. Moisture content can be estimated as follows:

- Dry—Requires addition of considerable moisture to obtain optimum moisture content.
- Moist—Near optimum moisture content.
- Wet—Requires drying to obtain optimum moisture content

3.3.4 Relative Density of Consistency

Relative density of a coarse-grained (cohesionless) soil will be qualitatively estimated based on experience. If the presence of large gravel or disturbance of the sample makes evaluation of the in situ relative density or consistency difficult, then this item should be left out of the description and explained in the comments column of the soil boring log.

Consistency of fine-grained (cohesive) soil will be based on qualitative evaluation using techniques outlined in Table 2.

3.3.5 Soil Structure or Mineralogy

Discontinuities and inclusions are important and should be described. Such features include slick-ensides, bedding or laminations, root holes, and wood debris.

Significant mineralogical information should be noted. Cementation or unusual mineralogy should be described, as well as other information such as organic debris or odor.

3.3.6 Group Symbol

Each soil description is concluded with the appropriate group symbol from ASTM D 2487-85. The group symbol should be placed in parentheses at the end of the description to indicate that the classification has been estimated.

In accordance with ASTM D 2488-84, dual symbols (e.g., GP-GM or SW-SC) can be used to indicate that a soil is estimated to have between 5 and 12 percent fines. Borderline symbols (e.g., GM/SM or SW/SP) can be used to indicate that

Table 2
Consistency of Fine-Grained Soil
(Developed from Sowers, 1979)

Consistency	Field Test
Very soft	Easily penetrated several inches by fist
Soft	Easily penetrated several inches by thumb
Firm	Can be penetrated several inches by thumb with moderate effort
Stiff	Readily indented by thumb, but penetrated only with great effort
Very stiff	Readily indented by thumbnail
Hard	Indented with difficulty by thumbnail

a soil sample has been identified as having properties that do not distinctly place the soil into a specific group. Generally, the group name assigned to a soil with a borderline symbol should be the group name for the first symbol. The use of a borderline symbol should not be used indiscriminately. Effort should be made to first place the soil into a single group.

Title: Sample Documentation and Packing and Shipping Instructions

Revision No.: 0

Date: 1/11/93

Sample Documentation and Packing and Shipping Instructions

1.0 SAMPLE DOCUMENTATION INSTRUCTIONS

1.1 Sample Identification Matrix (Figure 1)

1. Enter site name.

2. Enter project number.

3. Enter the case number and/or SAS number.

4. Enter the Central Regional Laboratory (CRL) log number or NAREL equivalent.

5. Specify the sample matrix using the two-digit codes listed below:

   - SS – Surface Soil
   - SB – Subsurface Soil
   - SW – Surface Water
   - SWL – Surface Water for Leachability
   - SD – Sediment
   - SDL – Sediment for Leachability
   - FT – Fish Tissue
   - AQ – Blank Samples
   - WL – Liquid Investigation-Derived Waste

6. Enter the sample number.

7. Enter the organic traffic label number or the SAS sample number.

8. Enter the inorganic traffic label number.

9. Enter the radiologic traffic label numbers.

10. Enter the chain-of-custody (COC) number.

11. Indicate the laboratory to be doing the analysis (abbreviations may be used as they are shown on the current laboratory list).

12. Enter the date the sample was taken: month, day, year (no hyphen or slash, e.g., 081292).

13. Enter the shipping date.

14. Enter the airbill number of the shipment.

15. List sample tag numbers corresponding to sample containers shipped under the traffic label number listed in either Box 7 or 8.

16. List the quality control (QC) lot numbers of the containers matching the tag numbers listed in Item 14.

Note: The date recorded on this form must be suitable for computer entry. Each entry must be flush left and must not exceed the number of digits allowed in each section. If portions of samples are to be sent to more than one laboratory for analysis, allow an entire line for each laboratory to accommodate for the additional traffic report, COC, and airbill numbers.

1.2 Sample Tag (Figure 2)

1. Enter date of sampling.

2. Enter time of sampling (military time only).

3. Specify "grab" or "composite" sample with an "X."

4. Enter CH2M HILL sample identification code.

5. Obtain signature of the sampler.

6. Indicate preservative used (if any) with an 'X."

7. Specify all parameters for analysis by placing an "X" to the right of each one.

8. Indicate the sample number [for Contract Laboratory Program (CLP) laboratory] or CRL log number (for CRL).

9. Indicate case number and/or SAS number (e.g., Case No. 1234 and/or SAS No. 5678E).

10. Leave BLANK (for laboratory use only).

11. Enter any desired analyses not listed on menu [e.g., polychlorinated biphenyls (PCBs), ammonia, sulfide] and mark box with an "X."

1.3 Inorganic Traffic Report (Figure 3)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Check the code that describes the activity being performed.

4a. Enter site name.

4b. Enter city and state of site.

4c. Enter spill ID No. [obtained from the Project Manager (PM) or Data Coordinator].

5. Enter Environmental Protection Agency (EPA) region number (e.g., V).

6. Enter sampler's company/office.

7. Enter sampler's name.

8. Enter laboratory name, address, and laboratory contact.

9. Indicate date of shipment.

10. Indicate airbill number corresponding to sample shipment.

11. Indicate the shipment carrier (i.e., Federal Express).

12. Enter the ITR Label Number.

13. Indicate sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7, 8) from Box 5 on ITR.

14. Specify sample concentration with an L, M, or H indicating contamination level, if applicable.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a matrix spike/matrix spike duplicate (MS/MSD).

17. Enter CH2M HILL sample number.

18. Enter the date/time of sample collection.

19. Leave BLANK (for laboratory use only).

20. Write at bottom of form if shipment is **complete** or is **not complete**.

1.4 Organic Traffic Report (Figure 4)

1. Enter assigned laboratory case number.

2. Enter assigned laboratory SAS number, if applicable.

3. Check the code that describes the activity being performed.

4a. Enter site name.

4b. Enter site city and state.

4c. Enter spill ID No. (obtained from the PM or Data Coordinator).

5. Enter EPA region number (e.g., V).

6. Enter sampler's company/office.

7. Enter sampler's name.

8. Enter laboratory name, address, and laboratory contact.

9. Indicate date of shipment.

10. Indicate airbill number corresponding to sample shipment.

11. Indicate the shipment carrier (i.e., Federal Express).

12. Enter the OTR Label Number.

13. Specify sample description with a number (e.g., 1, 2, 3, 4, 5, 6, 7, 8) from Box 5 on OTR.

14. Specify the sample concentration with an L, M, or H indicating contamination level, if applicable.

15. Check required analyses.

16. Specify special handling to notify laboratory if sample is a MS/MSD.

17. Enter CH2M HILL sample number.

18. Enter the date/time of sample collection.

19. Leave BLANK (for laboratory use only).

20. Write at bottom of form if shipment is **complete** or is **not complete**.

1.5 SAS Packing List (Figure 5)

1. Enter assigned SAS case number.

2. Enter EPA region number (e.g., V).

3. Enter STL's name.

4. Enter STL's company/office and phone number.

5. Enter date sample was taken.

6. Enter date of shipment.

7. Enter site name.

8. Enter laboratory name and address.

9. Enter name of laboratory contact.

10. List SAS sample numbers, which should include the SAS number.

11. Specify sample matrix, concentration, tag number, and analysis to be performed (e.g., low concentration soil sample for TCL analysis, Tag No. 5-48246).

12. Leave BLANK (for laboratory use only).

1.6 COC Record (Figure 6)

1. Enter first six digits of the CRL sample identification code.

2. Leave blank.

3. Obtain full signature of sampler and signed initials of active team members (including person responsible for paperwork).

4. Enter last three digits of the CRL sample identification code.

5. List sampling dates for all samples.

6. List sampling times for all samples (military time only).

7. Indicate "grab" or "composite" sample with an "X."

8. List CH2M HILL sample numbers.

9. Enter number of containers per sample.

10. List analyses individually.

11. Enter column heading for traffic label number and list serial numbers for corresponding sample identification codes.

12. Write in the words "CASE No.:" or "SAS No.:" and enter the correct number.

13. Enter column heading for "tag number" and list tag numbers for each sample container.

14. Obtain signature of sample team leader and carry out COC procedures.

15. State carrier service and airbill number, lab service, and custody seal numbers.

1.7 Combined COC and Traffic Report Forms (Figure 7)

A. Project Code: Leave blank.

B. Account Code: Leave blank.

C. Regional Information: If sampling is in support of oversight activities, indicate here. If not, leave blank.

D. Non-Superfund Program: If sampling is not done under the Superfund program, enter the name of the program [e.g., Resource Conservation and Recovery Act (RCRA)]. Not applicable to Kress Creek site.

E. Site Name, City, State: Complete as instructed.

F. Site Spill ID: Enter ID code provided by the project manager.

G. Region No.: Enter "Region V."

H. Sampling Company: Enter "CH2M HILL."

I. Sampler Information: Complete as instructed.

J. Type of activity: RI/FS – Remedial Investigation/Feasibility Study

K. Shipping Information: Complete as instructed.

L. Ship To: Enter laboratory name, address, and sample recipient/custodian.

M. SAS/Case No.: Complete as instructed.

N. Sample Numbers: For routine organic/inorganic samples, enter the CLP numbers from the "stick-on" labels. For SAS samples, enter the SAS sample numbers (SAS number plus a unique sequential numeric suffix).

O. Sample Information: Complete as instructed.

P. Regional Specific Tracking Number or Tag Number: Enter sample tag number(s).

Q. Station Location Number: Enter sample identifier (as defined in the FSP).

R. Time/Date: Complete as instructed. Use military time.

S. Sampler Initials: OPTIONAL.

T. Corresponding CLP Organic/Inorganic Sample Number: Enter CLP sample number (from "stick-on" labels) of corresponding sample from same location. Not applicable to SAS forms.

U. Designated Field QC: Indicate QC status when applicable (field blanks, trip blanks, duplicates, MS/MSD, etc.).

V. Sampling Status: Is the sampling for this case/SAS complete? Circle one.

W. Page 1 of __: Record number of documents enclosed in cooler.

X. MS/MSD and/or Duplicate: List samples.

Y. Additional Samplers Signatures: OPTIONAL.

Z. COC Seal No.: Enter the numbers that appear on the custody seals to be used to seal the cooler (there should be two).

AA. "Relinquished by" and "Time/Date": Complete as instructed. Use military time.

BB. Split Samples: Potentially responsible party (PRP) representative (PRP contractor) shall sign off here if work is oversight. Not applicable to Kress Creek site.

Distribution: The **Lab Copy and Lab Copy for Return to SMO** are included with the shipment. The **Region Copy and SMO Copy** are returned to the office.

1.8 Notice of Transmittal (Figure 8)

1. Enter name of team leader.
2. Enter team leader's firm name.
3. Enter CH2M HILL project number.
4. Enter case number.
5. Enter date.
6. Enter number of samples shipped.
7. Enter matrix of samples.
8. Enter the site name in words.
9. Enter the location of the site (city, state).

1.9 CRL Sample Data Report (Figure 9)

The CRL Sample Data Report is filled out by the CH2M HILL Sample Documentation Coordinator. A separate report is filled out for each laboratory that receives samples.

1. Enter the case number or SAS number.
2. Enter the site name.
3. Enter the laboratory name.
4. Enter the date shipped.
5. Enter the Superfund D.U. number.
6. Enter the EPA RPM.
7. Enter the CERCLIS number.
8. Enter the page numbers.
9. Enter the CRL numbers.
10. Enter the organic or inorganic traffic report number or the SAS packing list number.
11. Check the appropriate boxes for the analyses to be performed.

2.0 PACKAGING AND SHIPPING PROCEDURES

2.1 Low-Concentration Chemical Samples

1. Prepare coolers for shipment.

   - Drain openings. If applicable, drains shall be sealed with an epoxy compound or filled with silicone rubber to prevent accidental leakage.
   - Affix labels reading "This Side Up" on all four sides and "Fragile" on at least two sides of each cooler.
   - Place mailing label with laboratory address on top of coolers.

- Fill bottom of coolers with about 1 in. of cellulose packing material or use preformed poly-foam liner.

- Place appropriate traffic reports, SAS packing lists, or regional field sheets and COC records with corresponding custody seals on top of each cooler.

2. Arrange decontaminated sample containers in groups by sample number.

3. Use a grease pencil to mark volume levels on bottles.

4. Secure appropriate sample tags with string or wire around the lids of containers.

5. Place labels reading "RADIOACTIVE" on each sample container and secure container lids with strapping tape.

6. Arrange containers in front of assigned coolers.

7. Affix appropriate adhesive labels from assigned traffic report to each container. Protect with clear label protection tape.

8. Seal each container within a separate plastic bag.

9. Arrange containers in coolers so that they do not touch.

10. Ice used to preserve the samples should be repackaged in double ziplock-type bags and placed on and around the containers [especially on volatile organic compound (VOC) vials].

11. Fill remaining spaces with cellulose packing material or place poly-foam liner cover on top of samples.

12. Contact a health physics technician to obtain a radiological release survey. Sample shipping radiological surveys will be conducted using CH2M HILL Radiation Safety Manual (RSM) Procedures RP-06, RP-07, and RP-11. In addition, unshielded contact gamma measurements shall be taken on each sample container using an SPA-8 gamma scintillation detector.

    The dose rate at any point on the external surface of the cooler must not exceed 0.5 millirem per hour. The fixed and removable radioactive surface contamination on the external surface of the package must not exceed the following limits:

Fixed	Removable
α < 300 dpm/100 cm^2	α < 20 dpm/100 cm^2
$\beta\gamma$ < 1,000 dpm/100 cm^2	$\beta\gamma$ < 200 dpm/100 cm^2

    An unshielded contact SPA-8 reading equal to or exceeding 1.0×10^6 cpm indicates the package activity may exceed the Department of Transportation (DOT) "limited quantity" limit. This sample must be split and shipped in two or more coolers. Sample mass per cooler for a sample with SPA-8 results equal to

or greater than those shown above must be < 500 g. Each elevated activity sample that has been split into less than 500 g portions using the criteria shown above will be shipped in a cooler containing only background activity samples. If the sample cannot be split into portions < 500 g with SPA-8 readings less than 10^6 cpm, contact the Radiation Health Manager for additional instructions. Do not ship sample without further approval.

13. Sign COC form (or obtain signature) and indicate the time and date it was relinquished to Federal Express.

14. Separate copies of forms. Obtain a copy of the radiological survey results for shipment with the sample. Complete a copy of the shipper's declaration (Figure 10) for limited activity shipments. Place with other paperwork to be shipped with the cooler. Seal copies within a large ziplock-type bag and tape to inside lid of cooler. Distribute remaining copies as indicated in the following sections.

15. Close lid and latch.

16. Carefully peel custody seals from backings and place intact over lid openings (right front and left back). Cover seals with clear protection tape (Figure 11).

17. Tape cooler shut on both ends, making several complete revolutions with strapping tape. **Do not** cover custody seals (see Figure 11).

18. Relinquish to Federal Express. Place airbill (Figures 12 and 13) receipt inside the mailing envelope and send along with the other documentation to the Sample Documentation Coordinator.

19. Telephone the SMO in Alexandria, Virginia.

    (Note: This step should be omitted for samples sent to the CRL).

    Ms. Leslie Braun (subject to change)
    703/557-2490

    Provide the following information:

    - Your name
    - Project name
    - Case number
    - Number of samples sent to each laboratory for analysis
    - Airbill numbers

    This must be done IMMEDIATELY following sample shipment. If the SMO is closed at that time, call in the information as first priority the next day.

2.2 Medium- and High-Concentration Chemical Samples

Medium- and high-concentration samples are packaged using the same techniques used for packaging low-concentration samples, with several additional restrictions. First, a special airbill including a Shipper's Certification for Restricted Articles is required (Figures 12 and 13). Second, labels reading "Flammable Liquid N.O.S." or "Flammable Solid

N.O.S." must be placed on at least two sides of the cooler. Third, sample containers are packaged in metal cans with lids before being placed in the cooler, as indicated below.

1. Place approximately 1/2-in. of cellulose packing material in the bottom of the can.

2. Place labels reading "Radioactive" on each sample container and position the sample jar in the ziplock-type bag so that the sample tags can be read through the plastic bag.

3. Place the jar in the can and fill the remaining volume with cellulose packing material.

4. Close the can and secure the lid with metal clips.

5. Write the traffic report number on the lid.

6. Place labels reading "This Side Up" and "Flammable Liquid N.O.S." (or "Flammable Solid N.O.S.") on the can.

7. Place the cans in the cooler.

8. Continue to package shipment as described in Low Concentration Samples above, Steps 10 through 18.

2.3 Special Instructions for Shipping Samples by Federal Express (Figures 11 and 12)

1. Label cooler as hazardous shipment.

   - Write shipper's address on outside of cooler. If address is stenciled on, just write "shipper" above it.
   - Write or affix sticker reading "This Side Up" on two adjacent sides.
   - Write or affix sticker reading "ORM-E" (with a box around it) on two adjacent sides. Below ORM-E, write NA No. 9188.
   - Label cooler to indicate "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.

2. Complete the special shipping bill for restricted articles (Figures 10, 12, and 13).

   - Under Proper Shipping Name, write "Hazardous Substance, N.O.S." and "liquid" or "solid," as applicable.
   - Under Class, write "ORM-E."
   - Under Identification No., write NA No. 9188.

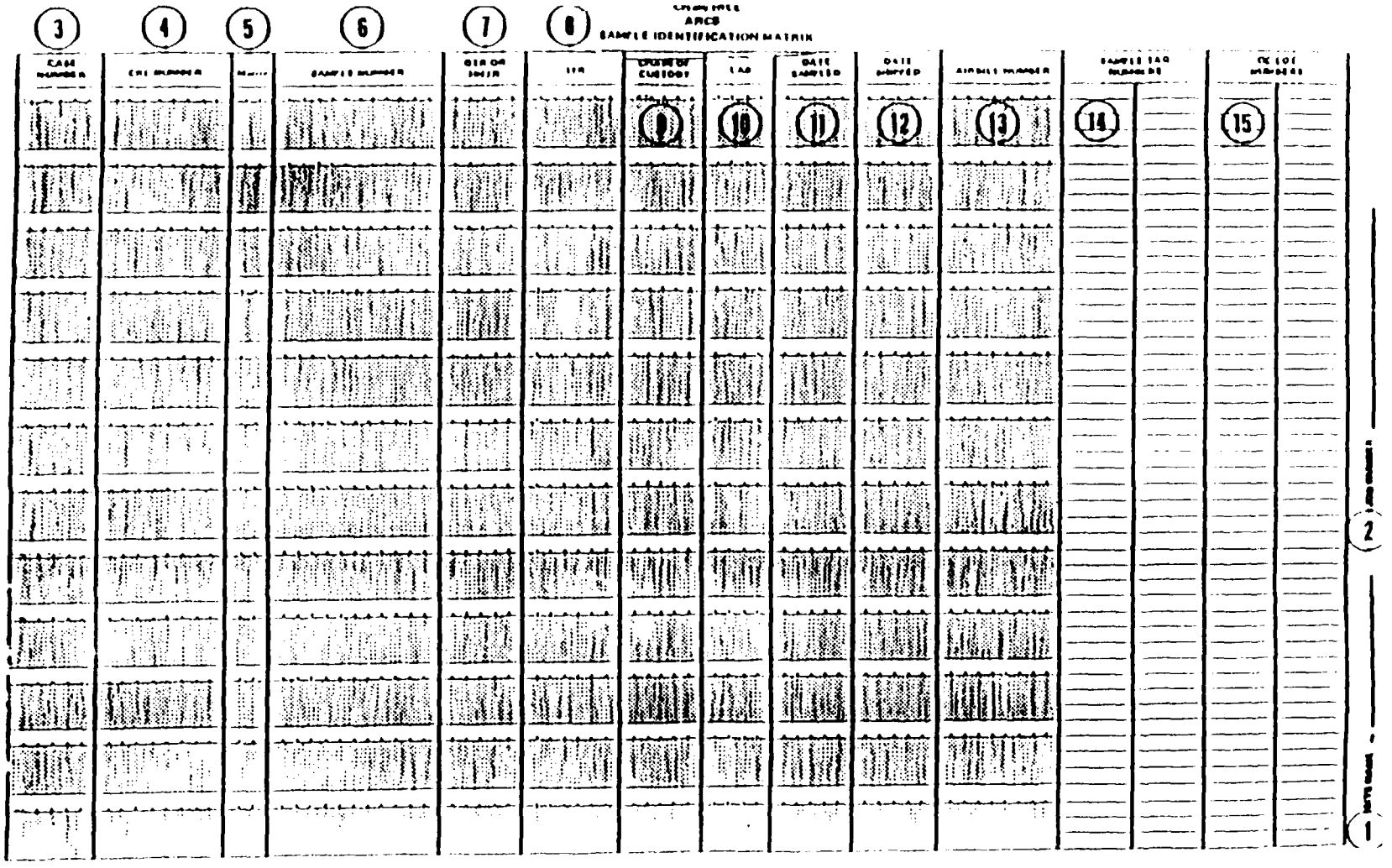


Figure 1
CH2M HILL ARCS
Sample Identification Matrix
Kress Creek RI/FS Field Procedures

Figure 2
Sample Label Tags
Kress Creek RI/FS Field Procedures

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

REGION 5
230 South Dearborn Street
Chicago, Illinois 60604

EPA

Case # or Project Code | Sample Number | Month/Day/Year | Time | DESIGNATE: Grab / Comp.

Station Number and Location | Samplers (signatures)

Tag Number 5— 077106 | Lab Sample Number

PRESERVATIVE: H_2SO_4 ☐
HCL ☐ HNO_3 ☐ NaOH ☐ Other ☐

ANALYSES			
VOA	METALS	Mercury	TOC
ABN	CYANIDE	Fluoride	BOD
PEST/PCB		Nitrate/Nitrite	COB
Pesticides		TOC	TDS
Herbicides		BOD	TSS
PCB		COB	O&G
PCDD/PCDF		TDS	Sulfate
2,3,7,8-TCDD		TSS	Chloride
Ames Mutagen		O&G	Sulfide
Asbestos		Sulfate	Ammonia
Phosphorus		Chloride	Alkalinity
TO1		Sulfide	Acidity
TO2		Ammonia	TKN
TOX		Alkalinity	
CBOD		Acidity	
Bio-Acute		TKN	
Bio-Chronic			

Remarks:

USE FOR MS/MSD ☐

EPA United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703-557-2490 FTS 557-2490

Inorganic Traffic Report

(For Inorganic CLP Analysis)

SAS No. (if applicable)

Case No.

1. Project Code | Account Code

Regional Information

Non-Superfund Program

Site Name

City, State | Site Spill ID

2. Region No. | Sampling Co.

Sampler (Name)

Sampler Signature

3. Type of Activity

ENF ☐ ER ☐ LSI ☐ NPLD ☐ PA ☐ RA ☐ RD ☐ STSI ☐ O&M ☐ RIFS ☐ SSI ☐ ST ☐ STPA ☐ Other ☐

4. Date Shipped | Carrier

Airbill Number

5. Ship To

6. Preservative (Enter in Column D)

1. HNO_3
2. NaOH
3. HCl
4. H_2SO_4
5. Ice only
6. Other (SAS) (Specify)
N. Not preserved

7. Sample Description (Enter in Column A)

1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

Double volume required for spike/duplicate analysis sample

Ship medium and high concentration samples in paint cans.

See reverse for additional standard instructions.

For total or dissolved metals, check only one RAS analysis per each sample.

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc. Low Med High	C Sample Type: Comp / Grab	D Preservative from Box 6	E - RAS Analysis: Metals Total	Metals Dissolved	Cyanide	Low Conc. Nitrate/Nitrite	Low Conc. Fluoride	High pH	High Conductivity	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Org. Samp. No

Shipment for Case complete? (Y/N)

TR

CHAIN OF CUSTODY RECORD

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)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted (Signature)
☐ Declined

EPA Form 9110-1 (Rev. 11-90) Replaces EPA Form (2075-6), previous edition which may be used

DISTRIBUTION:
Green - Region Copy Pink - SMO Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

Figure 3
Inorganic Traffic Report
Kress Creek RI/FS Field Procedures

EPA

United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703 557 2490 FTS 557 2490

Organic Traffic Report

(For Organic CLP Analysis)

SAS No (if applicable)

Case No

1. Project Code | Account Code

Regional Information

Non Superfund Program

Site Name

City, State | Site Spill ID

2. Region No. | Sampling Co

Sampler (Name)

Sampler Signature

3. Type of Activity

ENF ☐ PA ☐ O&M ☐
ER ☐ RA ☐ RIFS ☐
LSI ☐ RD ☐ SSI ☐
NPLD ☐ STSI ☐ ST ☐
STPA ☐
Other ☐

4. Date Shipped | Carrier

Airbill Number

5. Ship To

6. Preservative (Enter in Column D)

1. HCl
2. HNO3
3. Na_2SO_4
4. H_2SO_4
5. Other (SAS) (Specify)
6. Ice only
N. Not preserved

7. Sample Description (Enter in Column A)

1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

Triple volume required for matrix spike/matrix spike duplicate analysis sample.
Ship medium and high concentration samples in paint cans.
See reverse for additional standard instructions.
Please indicate sample to spike/duplicate

CLP Sample Numbers (from labels)	A Enter # from Box 7	B Conc Low Med High	C Sample Type Comp / Grab	D Preservative from Box 6	E RAS Analysis VOA	BNA	Pest/ PCB	High ARO/ TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/ Year/Time Sample Collection	I Sampler Initials	J Corresp. CLP Inorg Samp. No.	
Shipment for Case complete? (Y/N)												TR		

CHAIN OF CUSTODY RECORD

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)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	Is custody seal intact? Y/N/none

Split Samples ☐ Accepted (Signature)
☐ Declined

EPA Form 9110-2 (Rev. 10-90) Replaces EPA Form (2075-7), previous edition which may be used

DISTRIBUTION:
Blue - Region Copy Pink - SMO Copy White - Lab Copy for Return to SMO Yellow - Lab Copy

Figure 4
Organic Traffic Report
Kress Creek RI/FS Field Procedures

EPA United States Environmental Protection Agency
Contract Laboratory Program Sample Management Office
PO Box 818 Alexandria, VA 22313
703 557 2490 FTS 557 2490

Special Analytical Service

Packing List/Chain of Custody

SAS No

1 Project Code | Account Code

Regional Information

Non Superfund Program

Site Name

City, State | Site Spill ID

2 Region No | Sampling Co

Sampler *(Name)*

Sampler Signature

3 Type of Activity

	Lead	Pre Remedial	Remedial		Removal	
SF			RIFS		CLEM	
PRP		PA	RD		REMA	
ST		SSI	RA		REM	
FED		LSI	O&M		OIL	
			NPL D		UST	

4 Date Shipped | Carrier

Airbill Number

5 Ship To

6 Sample Description *(Enter in Column A)*
1 Surface Water
2 Ground Water
3 Leachate
4 Rinsate
5 Soil/Sediment
6 Oil
7 Waste
8 Other *(Specify)*

7 Preservative *(Enter in Column C)*
1 HCl
2 HNO3
3 $NAHSO_4$
4 H_2SO_4
5 NAOH
6 Other (SAS) *(Specify)*
7 Ice only
N Not preserved

Sample Numbers	A Matrix Enter from Box 6	B Conc Low Med High	C Preservative Used from Box 7	D Analysis	E Sample used for spike and/or duplicate	F Regional Specific Tracking Number or Tag Number	G Station Location Identifier	H Mo/Day/Year/Time Sample Collection	I Sampler Initials	J Designated Field QC
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										

Shipment for SAS complete? (Y/N)

CHAIN OF CUSTODY RECORD

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)*
Received by: *(Signature)*	Date / Time	Received for Laboratory by: *(Signature)*	Date / Time	Remarks Is custody seal intact? Y/N/none	

Split Samples ☐ Accepted *(Signature)*
☐ Declined

EPA Form 9110-3 (4-91)

DISTRIBUTION:
White - Region Copy Yellow - SMO Copy Pink - Lab Copy for Return to SMO Gold - Lab Copy

Figure 5
Special Analytical Service Packing List
Kress Creek RI/FS Field Procedures

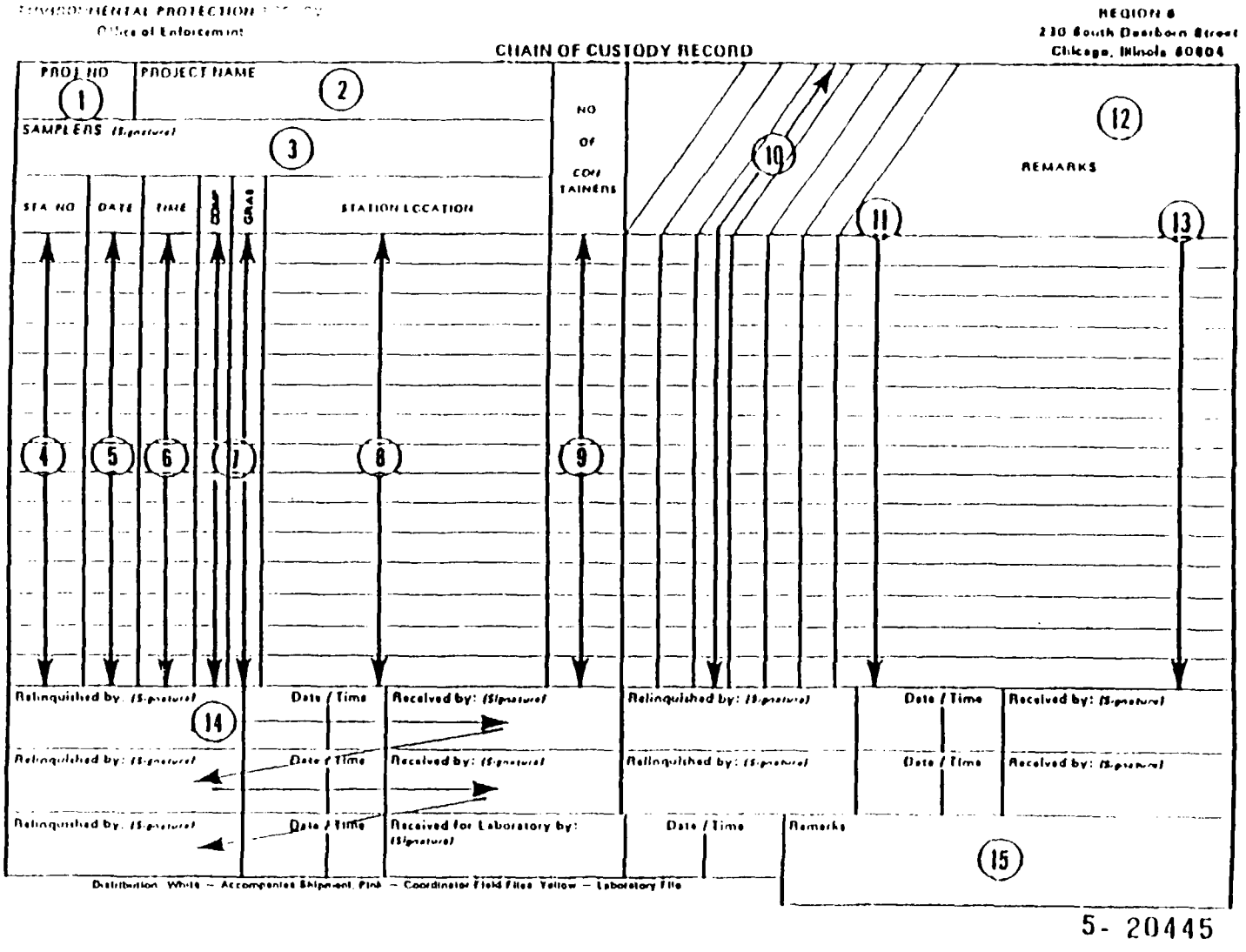
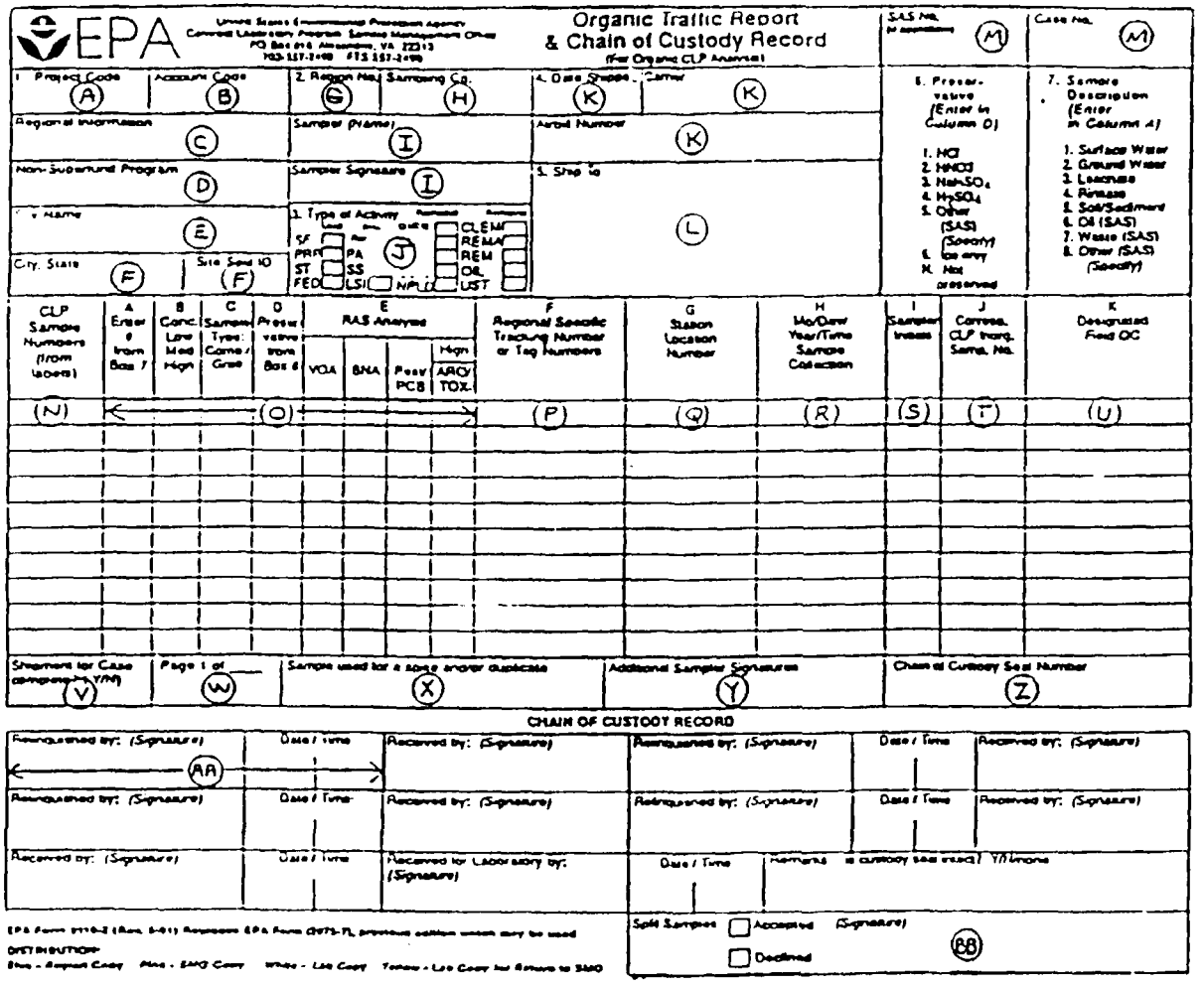


Figure 6
Chain of Custody Record
Kress Creek RI/FS Field Procedures



EPA

Organic Traffic Report & Chain of Custody Record

(For Organic CLP Analysis)

SAS No. (M) | Case No. (M)

1. Project Code (A) | Account Code (B)

2. Region No. (G) | Sampling Co. (H)

4. Date Shipped (K) | Carrier (K)

Regional Information (C)

Sampler (Printed) (I)

Airbill Number (K)

Non-Superfund Program (D)

Sampler Signature (I)

5. Ship to (L)

Site Name (E)

3. Type of Activity (J): CLEM, REMA, REM, OIL, UST; SF, PRP, PA, ST, SS, FED, LSI, NPL

City, State (F) | Site Spill ID (F)

6. Preservative (Enter in Column D)
1. HCl
2. HNO3
3. NaHSO4
4. H2SO4
5. Other (SAS) (Specify)
6. Ice only
N. Not preserved

7. Sample Description (Enter in Column A)
1. Surface Water
2. Ground Water
3. Leachate
4. Rinsate
5. Soil/Sediment
6. Oil (SAS)
7. Waste (SAS)
8. Other (SAS) (Specify)

CLP Sample Numbers (from labels)	A Error # from Box 7	B Conc. Low Med High	C Sample Type: Comp / Grab	D Preservative from Box 6	E RAS Analyses: VOA	BNA	Pest/PCB	High ARCV/TOX	F Regional Specific Tracking Number or Tag Numbers	G Station Location Number	H Mo/Day/Time Sample Collection	I Sampler Initials	J Corresp. CLP Inorg. Samp. No.	K Designated Field QC
(N)	←			(O)	→				(P)	(Q)	(R)	(S)	(T)	(U)

Shipment for Case complete? Y/N (V) | Page 1 of (W) | Samples used for a lower another duplicate (X) | Additional Sampler Signatures (Y) | Chain of Custody Seal Number (Z)

CHAIN OF CUSTODY RECORD

Relinquished by: (Signature) (AA)	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)
Received by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	Is custody seal intact? Y/N/None

EPA Form 9110-2 (Rev. 6-91) Replaces EPA Form 9175-7L, previous edition version may be used

DISTRIBUTION: White - Lab Copy; Yellow - Lab Copy for Return to SMO; Pink - Region Copy; Blue - SMO Copy

Split Samples ☐ Accepted (Signature) ☐ Declined (BB)

Figure 7
Combined Chain of Custody and Traffic Report Forms
Kress Creek RI/FS Field Procedures

NOTICE OF TRANSMITTAL

DATE:

TO: CH2M HILL - REM/FIT Office, Reg. V-X (WI)
310 West Wisconsin Avenue, Suite 700
P.O. Box 2090
Milwaukee, Wisconsin 53201

Attn.: Shirley Stringer

FROM: (1) (name) / (2) (firm)

CH2M HILL PROJECT #: (3)

Enclosed are appropriate copies of the sample documentation forms completed under Case # (4) for the (5), 19(5) shipment of (6) (qty) (7) (matrix) samples from the (8) site located in (9), (9).

GLT85/15

Figure 8
Notice of Transmittal
Kress Creek RI/FS Field Procedures

CENTRAL REGIONAL LABORATORY SAMPLE DATA REPORT

ORGANICS/INORGANICS

THIS FORM IS TO BE USED FOR SAMPLES SENT TO CONTRACT ONLY

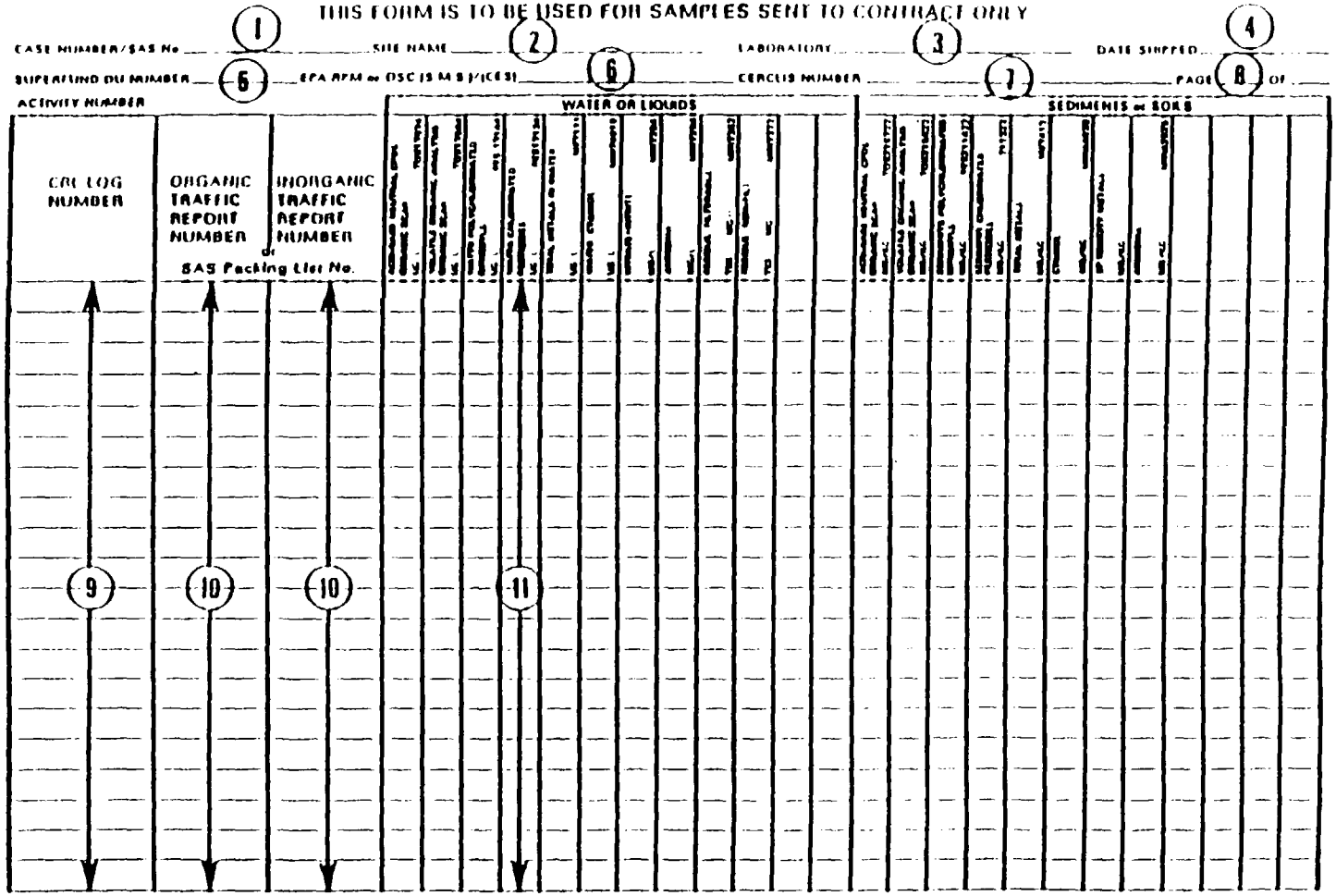


Figure 9
CRL Organic / Inorganic Sample Data Report
Kress Creek RI/FS Field Procedures

Engineers
Planners
Economists
Scientists

Date

Shipper's Declaration for Excepted Radioactive Materials, Instruments, and Articles

This package conforms to the conditions and limitations specified in 49 CFR 173.421 for excepted radioactive material, limited quantity, N.O.S., UN2910.

The materials are packaged in a strong, tight package; the radiation level on the surface of the package does not exceed 0.5 mR/hr; the outer surfaces of the package do not have transferrable contamination exceeding the limits specified in 49 CFR 173.443(a); and the outside of the inner package is labeled with a sticker that reads "Radioactive."

No other labels are required.

Shipped to:

_____
_____
_____
_____

Contact for Information:

George Stephens
Radiation Health Manager
CH2M HILL, Inc.
Oak Ridge Office
615/483-9032

ORORR38/035.51

Figure 10
Shipper's Declaration Form
Kress Creek RI/FS Field Procedures

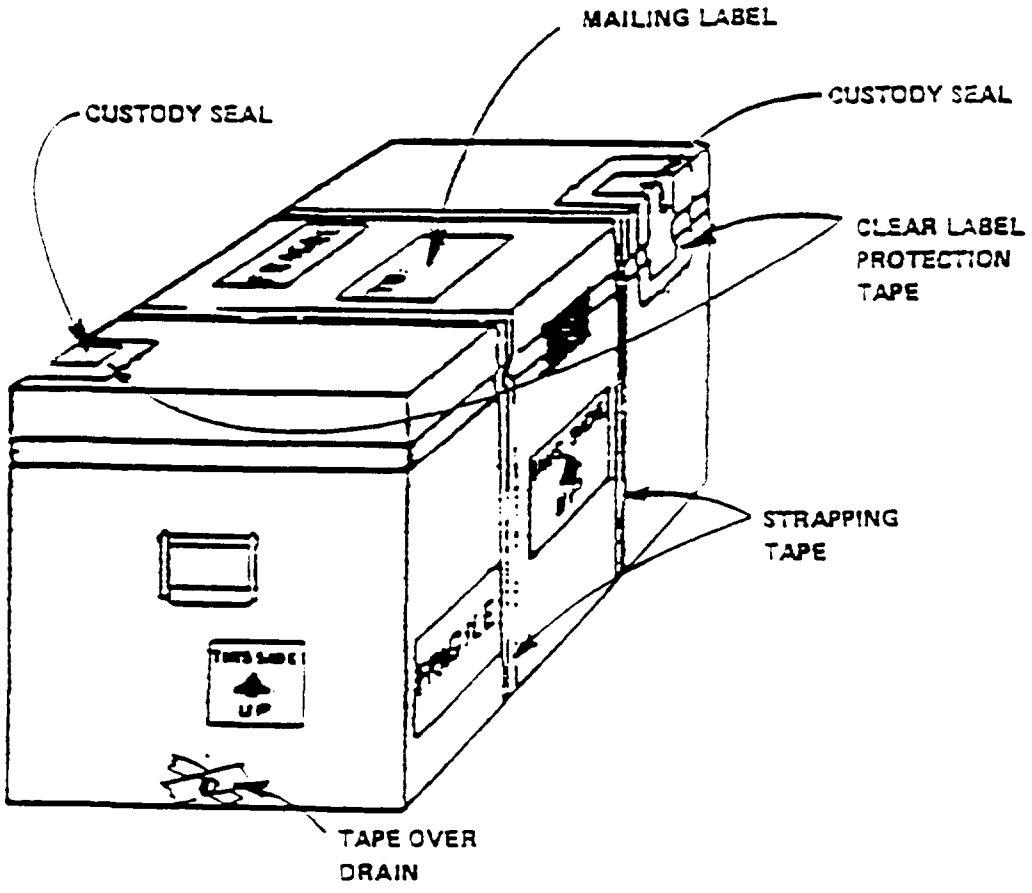


Figure 11
Packaging Diagram
Kress Creek RI/FS Field Procedures

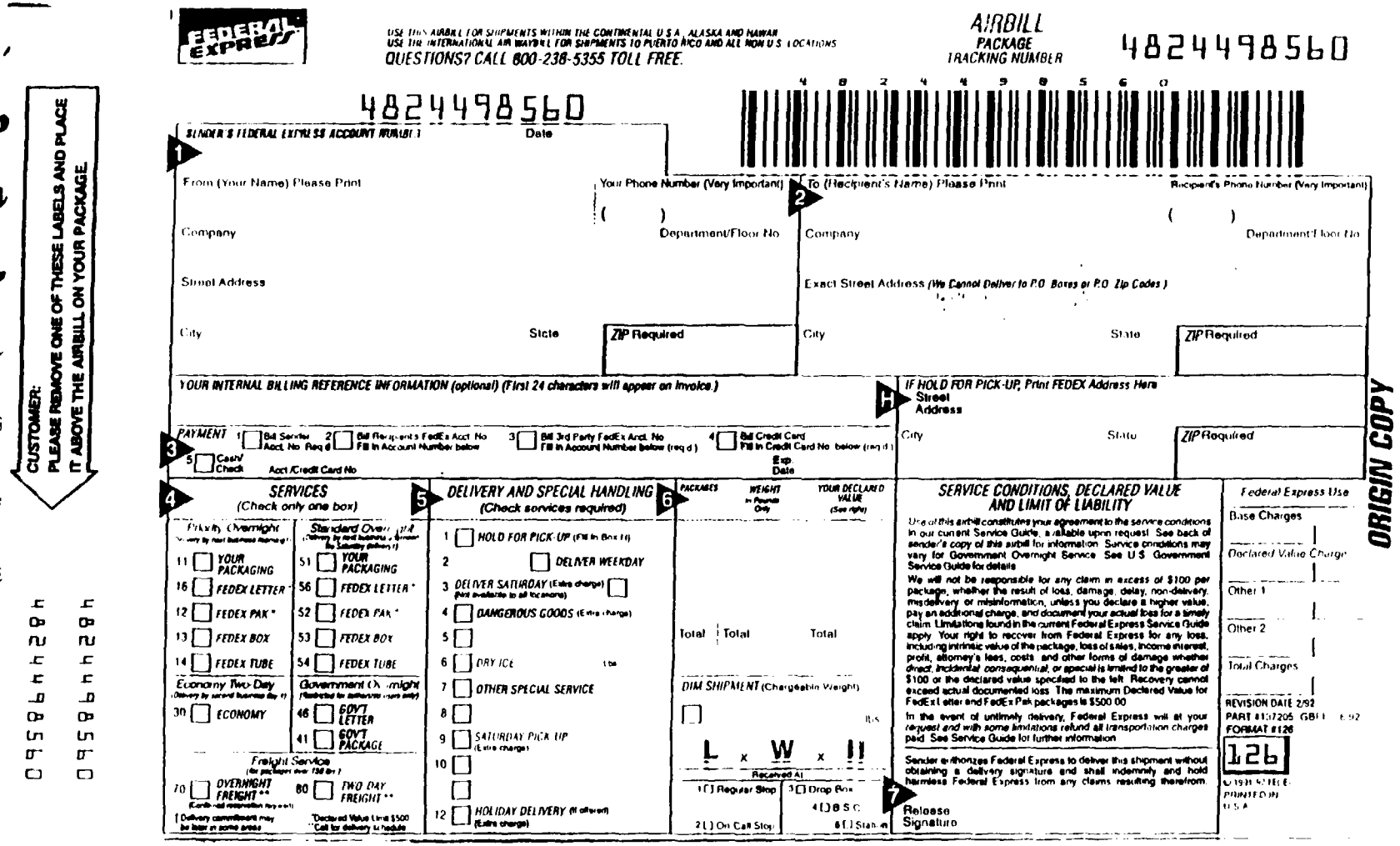


Figure 12
Sample Federal Express Airbill
Kress Creek RI/FS Field Procedures

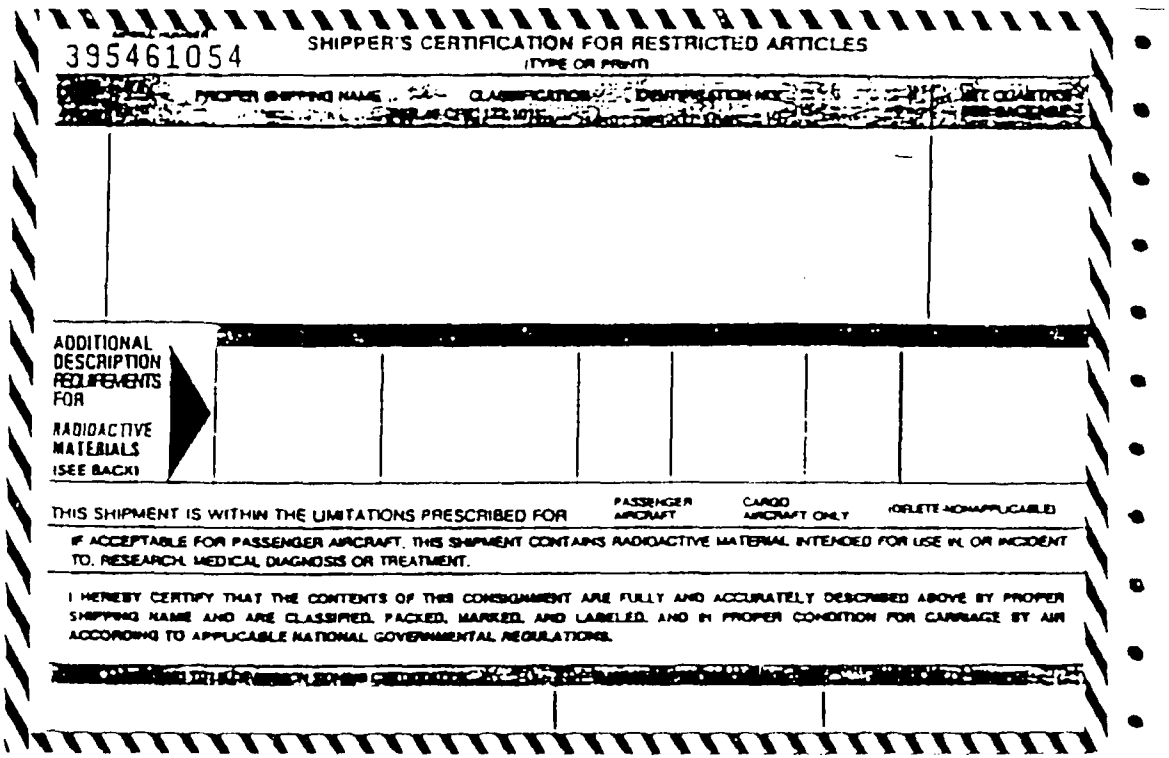


Figure 13
Certification for Restricted Articles
Kress Creek RI/FS Field Procedures

Title: Fixed and Removable Surface Contamination Surveys

Document No.: SOP: RP-06

Revision No.: 0

Date: 12/18/92

Approved by: George Sterling
Radiation Health Manager

Date: 12/18/92

RP-06
Fixed and Removable Surface Contamination Surveys

1.0 PURPOSE

The procedure describes general methods and techniques to be used when performing contamination surveys. For this project, this procedure's primary use will be for the release of equipment after decontamination when used in conjunction with RP-07.

2.0 SCOPE

2.1 Contamination surveys are performed and documented to demonstrate compliance with DOE Order 5480.11 and 10 CFR 20 and to help maintain exposures from surface radioactivity as low as reasonably achievable (ALARA). Surface contamination measurements are made for the following reasons:

1. to provide data for characterization of site radiological contamination
2. to verify the adequacy of personal protective equipment (PPE)
3. to verify the effectiveness of contamination control and decontamination
4. to meet criteria for release of articles, equipment, and personnel from Controlled Areas

3.0 REFERENCES

3.1 Department of Energy (DOE) Order 5480.11, *Radiation Protection of Occupational Workers*, Attachment 2, Surface Radioactivity Guides

3.2 Nuclear Regulatory Commission (NRC), *Guidelines for Decontamination of Facilities and Equipment Prior to Release for Unrestricted Use or Termination of Licenses for Byproduct, Source, or Special Nuclear Material*, Appendix I, Nl.RE5/CR-7082, ORN/HASR-95, June 1981

3.3 CH2M HILL Radiation Safety Manual, RP-06, November 1989

3.4 Oak Ridge Institute for Science and Education (ORISE) Environmental Survey and Site Assessment Program Survey Procedures Manual

4.0 EQUIPMENT AND MATERIALS

4.1 Survey Maps (plan view of area to be surveyed)

4.2 Envelopes or Plastic Bags

4.3 Smear Paper/Cloth

4.4 Gloves

4.5 Eberline Instrument Corporation AC-3 Detector or equivalent

4.6 Eberline Instrument Corporation HP-210 Detector or equivalent [Pancake Geiger-Mueller (GM) Detector]

4.7 Eberline Instrument Corporation PRS-1 or equivalent readout instrument (ratemeter/scaler)

4.8 Low Background Laboratory Alpha and Beta Counters

5.0 INSTRUCTION

5.1 General Guidance

5.1.1 Surveyor should review previous surveys, if available, to evaluate radiation and contamination types and levels in the areas to be surveyed.

5.1.2 Smears taken on a surface thought to be contaminated to levels exceeding 5,000 dpm/100 cm^2 beta/gamma and 100 dpm/100 cm^2 alpha may be evaluated using a portable ratemeter/scaler and pancake GM probe.

5.1.3 Smears taken on surfaces thought to be contaminated to levels less than 5,000 dpm/100 cm^2 beta/gamma and 100 dpm/100 cm^2 alpha should be evaluated with low background alpha and beta counters.

5.1.4 For surveys of equipment, record readings on Direct Surface Contamination Survey or Transferrable Surface Contamination Survey forms. Provide sketches of surveyed equipment and results in logbook as necessary.

5.2 Survey Locations

5.2.1 Smear and direct contamination surveys should be taken on floors, walls, and on representative items contacted by personnel when surveying areas outside the contamination area.

5.2.2 Smears should be taken on floors, walls, ledges, overhead surfaces, and floor drains to establish general transferrable contamination levels within the contamination area.

5.2.3 Smears and/or direct contamination readings shall be taken on all equipment removed from the contamination area.

5.2.4 All smear locations shall be recorded on the survey map using consecutive numbers enclosed within a square.

5.2.5 At the end of each day, survey maps and survey records shall be returned to the Site Safety Coordinator (SSC) for review and evaluation.

5.3 Direct Surface Contamination Surveys

5.3.1 Verify that the portable survey instrument(s) are calibrated and operating properly.

5.3.2 Pass the detector slowly over the surface to be surveyed holding the detector within 1 cm (1/2-in.) of the surface. Locate the area of maximum count rate by using the audible response of the instrument. Measure for at least 1/2-min, record the count rate at this position, and take a smear if applicable (see Section 5.4).

5.3.3 Record the required direct surface contamination readings in the appropriate columns of Form 6.1, for gross alpha and gross beta-gamma as appropriate.

5.3.4 Calculate the alpha emitter surface contamination level (C, dpm/100 cm^2) by subtracting the background cpm (B) from the gross cpm (G), divide the difference by the instrument's efficiency factor (E, cpm/dpm), and then divide the result by the detector surface area (A, cm^2). See Equation 1. Record the result.

(Equation 1)

$$C = (G-B)(1/E)(100/A)$$

5.3.5 Calculate the direct beta-gamma surface contamination level (C, dpm/100 cm^2) by subtracting the background reading (B, cpm) from the gross reading (G, cpm), dividing the difference by the instrument's efficiency factor (E, cpm/dpm), and dividing the result by the detector's surface area (A, cm^2). See Equation 1.

5.3.6 To measure beta-gamma surface contamination levels on surfaces that have high levels of alpha contamination, take an ordinary direct reading (G, cpm) with the GM pancake detector and a direct reading (P, cpm) with the pancake GM through a layer of 2-mil polyethylene. Calculate the beta-gamma emitter surface contamination level (C, dpm/100 cm^2) by subtracting the background reading (B, cpm) from the direct reading through the layer of polyethylene (P, cpm), divide the difference by the instrument's efficiency factor (E, cpm/dpm), and then divide the result by the detector surface area (A, cm^2). See Equation 2. Record the result.

(Equation 2)

$$C = (P-B)(1/E)(100/A)$$

5.3.7 Go to Section 5.5 of this procedure; calculate the minimum detectable activity (MDA).

5.4 Transferrable Contamination Surveys

5.4.1 Wipe smear paper/cloth over an area of approximately 100 cm^2. The smeared area can be a square 4 in. × 4 in. or an "S" pattern 16 in. long.

5.4.2 Place smears in coin envelopes.

Each smear should be identified by labeling it with a number that corresponds to the smear location number that is marked on the survey map.

5.4.3 Complete the survey and counting information on the heading of Form 6.2 using current calibration and background determinations posted on the laboratory counter(s).

5.4.4 Count each smear for at least 0.5 min and record the following:

Gross counts
Smear count time in minutes
Background

5.4.5 Calculate the transferrable alpha emitter or beta-gamma surface contamination level (C, dpm/100 cm^2) by subtracting the background cpm (B) from the gross cpm (G), then divide the difference by the instrument's efficiency factor (E, cpm/dpm). See Equation 3. Record the result.

(Equation 3)

$$C = (G-B)(1/E)$$

5.4.6 Go to Section 5.5 of this procedure and calculate the MDA.

5.5 Statistical Considerations for Contamination Surveys

5.5.1 Direct Contamination Surveys

(a) Calculate the standard deviation of the surface contamination count rate, sigma. See Equation 4.

(Equation 4)

$$\Sigma = \left[\frac{G}{T_s} + \frac{B}{T_b} \right]^{f/2}$$

where:

Σ = the standard deviation of the surface contamination count rate
G = the sample count rate (cpm)
T_s = the sample count time (min)
B = the background count rate (cpm)
T_b = the background count time (min)

(b) Calculate the MDA in units of dpm/100 cm^2. See Equation 5.

(Equation 5)

$$MDA = \frac{2.71 + 4.65\ (B \cdot t)^{1/2}}{(t) \cdot (E) \cdot \left[\frac{A}{100} \right]}$$

where:

MDA = minimum detectable activity (activity level in disintegrations/minute/100 cm^2)
B = background count rate in cpm
t = counting time in minutes
E = detector efficiency in cpm/dpm
A = active probe area in cm^2 (HP-260, 210 = 15.5 cm^2 and AC-3 = 59.0 cm^2)
2.71 = constant

The constant 2.71 may be dropped when the total background counts equal or exceed 10. When this occurs, Equation 5 may be reduced to:

$$MDA = \frac{4.65\ (B/t)^{1/2}}{(E)\left[\frac{A}{100}\right]}$$

MDA is the minimum activity a sample must contain before it is said to exhibit any radioactive material above background. The MDA as defined above represents that concentration above which there is less than a 5 percent probability that radioactivity will be reported present when it is really absent (Type I error), or reported absent when it is really present (Type II error).

(c) When the surface contamination level (Equations 1, 2, or 3) is greater than MDA, report the surface contamination level.

(d) When the surface contamination level is less than MDA, report the result as "less than" the MDA value (e.g., < X dpm/100 cm^2 where X is the calculated MDA).

5.5.2 Transferrable Contamination Surveys

(a) Calculate sigma as indicated in Equation 4.

(b) Calculate MDA as indicated in Equation 5, with A = the surface area smeared (e.g., 100 cm^2).

(c) When the surface contamination level (Equations 1, 2, or 3) is greater than MDA, report the surface contamination level.

(d) When the surface contamination level is less than MDA, report the result as "less than" the MDA value (i.e., < × dpm, where × is the calculated MDA).

5.6 Quality Control

5.6.1 Obtain a duplicate count for approximately one in every ten measurements for both alpha and beta-gamma direct readings. The locations will be chosen by the Health Physics Technician. Record the duplicate results with a (D) after the measurement value.

5.6.2 Duplicate counts will be performed on 10 percent of all transferrable contamination samples and recorded as (D) on the CH2M HILL Transferrable Surface Contamination Survey Sheets.

CH2M HILL

Transferable Surface Contamination Survey

Survey Date: _____ **Survey No.:** _____

Surveyor's Name: _____ **Project/Site:** _____

Area Surveyed: _____ **Project Number:** _____

ALPHA SURVEY INSTRUMENT

Scaler Model: _____

Serial Number: _____

Probe Model: _____

Serial Number: _____

Calibration Date: _____

Efficiency: _____ cpm/dpm

Background: _____ cpm

MDA: _____ dpm

BETA-GAMMA SURVEY INSTRUMENT

Scaler Model: _____

Serial Number: _____

Probe Model: _____

Serial Number: _____

Calibration Date: _____

Efficiency: _____ cpm/dpm

Background: _____ cpm

MDA: _____ dpm

SMEAR NO.	LOCATION	ALPHA		BETA-GAMMA	
		GROSS cpm	dpm/100 cm^2 a)	GROSS cpm	dpm/100 cm^2 a)

a) dpm/100cm^2 Results are the total per swipe (1 swipe covers 100cm^2

_____ _____

Surveyed by **Reviewed by**

CH2M HILL

Direct Surface Contamination Survey

Survey Date: _____ **Survey No.:** _____

Surveyor s Name: _____ **Project/Site Name:** _____

Area Surveyed: _____ **Project Number:** _____

ALPHA SURVEY INSTRUMENT

Scaler Model: _____

Serial Number: _____

Probe Model: _____

Serial Number: _____

Calibration Date: _____

Efficiency: _____ cpm/dpm

Background: _____ cpm

MDA: _____ dpm

BETA-GAMMA SURVEY INSTRUMENT

Scaler Model: _____

Serial Number: _____

Probe Model: _____

Serial Number: _____

Calibration Date: _____

Efficiency: _____ cpm/dpm

Background: _____ cpm

MDA: _____ dpm

LOCATION	ALPHA			BETA-GAMMA		
	Gross cpm	dpm	dpm/100 cm^2	Gross cpm	dpm	dpm/100 cm^2

_____ _____

Surveyed by *Reviewed by*

Title: Evaluation of Surface Contamination on Articles to be Released for Unrestricted Use

Document No.: SOP: RP-07

Revision No.: 0

Date: 12/18/92

Approved by: George Stevens
Radiation Health Manager

Date: 12/18/92

RP-07
Evaluation of Surface Contamination on Articles to be Released for Unrestricted Use

1.0 PURPOSE

This procedure describes the methods of surveying equipment or materials for release to unrestricted use from work sites.

2.0 SCOPE

This procedure applies to work sites and to materials, equipment, etc., that are subject to release for unrestricted use.

3.0 REFERENCES

3.1 U.S. Department of Energy (DOE) Order 5480.11, Radiation Protection of Occupational Workers, Attachment 2, *Surface Radioactivity Guides*

3.2 CH2M HILL Radiation Safety Manual, November 1989

3.3 CH2M HILL Radiation Protection Procedure 6.0, Fixed and Removable Contamination Surveys, RP-06, November 1, 1989

4.0 EQUIPMENT AND MATERIALS

4.1 Alpha Scintillation Detector (Eberline AC-3 or equivalent)

4.2 Pancake-Geometry Beta-Gamma Detector (Eberline HP-210, HP-260, or equivalent)

4.3 Portable Ratemeter/Scaler (Eberline PRS-1 or equivalent)

4.4 Scintillation Alpha Counter (Eberline SAC-4 or equivalent)

4.5 Equipment and Materials Release, Form 7.1

4.6 Cloth swipes and envelopes (NuCon smears or equivalent)

5.0 INSTRUCTIONS

5.1 Release of Items for Unrestricted Use

5.1.1 Both direct and transferable surveys shall be made on equipment, materials, or vehicles that are to be released for work sites for unrestricted use. This shall include any item that has entered a Controlled Area or items that may have come in direct contact with contamination. The potential for objects to have become

contaminated by airborne dust particles must also be considered. The potential for contamination should be considered prior to any release of equipment or materials.

5.1.2 All items to be released shall be surveyed in such a manner as to fully demonstrate that external and internal areas (of items being released) comply with surface contamination limits specified in Table 1 of this procedure.

5.1.3 The Site Safety Coordinator (SSC) shall review survey data and inspect items prior to release.

In the event the SSC has any question about internal contamination potentials, this item shall not be released until a complete review of pertinent information has been completed by the health physics technician. Upon completion of this review, determinations of equipment release requirements will be quickly resolved and the Radiation Health Manager (RHM) shall be advised.

5.1.4 Release limits specified in NRC Regulatory Guide 1.86 are shown in Table 1. CH2M HILL release criteria are shown in Table 2. Considering NRC, CH2M HILL, and the Illinois Administrative Code, Section 340, CH2M HILL criteria use the most restrictive criteria.

5.1.5 When items to be released have met the release limits specified in Table 2, the SSC shall sign the release form. Items released shall have a completed and signed copy of Form 7.1 attached to its exterior surface. No item shall be considered releasable without this form attached. Vehicles are not required to have this form attached unless the vehicle is to remain at the site for later removal.

5.1.6 Any large item to be released that does not have a copy of Form 7.1 attached shall be considered potentially contaminated and will require a release survey. Small items (hand tools, etc.) that are to be released may be placed in a clean carton or box with a copy of Form 7.1 attached to the container prior to release. Small items that are to be released to individuals do not require the attached form if such items are immediately removed from the site after the release surveys.

5.2 Surface Measurements

5.2.1 Perform a direct surface contamination survey initially to determine the gross amount of activity on the item surveyed. See Procedure RP-06.

5.2.2 Surfaces must be dry for effective alpha contamination monitoring.

5.2.3 Vehicles hauling contaminated materials in their contained truck beds need only the exterior portions of the vehicle surveyed until they are released from the project.

Table 1
NRC Regulatory Guide 1.86
Surface Radioactivity Contamination Limits
for Release for Unrestricted Use

Nuclide[a]	Average[bc]	Maximum[bd]	Removable[be]
U-nat, U-235, U-238, and associated decay products	5,000 dpm α/100 cm^2	15,000 dpm α/100 cm^2	1,000 dpm α/100 cm^2
Transuranics, Ra-226, Ra-228, Th-230, Th-228, Pa-231, Ac-227, I-125, I-129	100 dpm/100 cm^2	300 dpm/100 cm^2	20 dpm/100 cm^2
Th-nat, Th-232, Sr-90, Ra-223, Ra-224, U-232, I-126, I-131, I-133	1,000 dpm/100 cm^2	3,000 dpm/100 cm^2	200 dpm/100 cm^2
Beta-gamma emitters (nuclides with decay modes other than alpha emission or spontaneous fission) except Sr-90 and others noted above.	5,000 dpm $\beta\gamma$/100 cm^2	15,000 dpm $\beta\gamma$/100 cm^2	1,000 dpm $\beta\gamma$/100 cm^2

[a]Where surface contamination by both alpha- and beta-gamma-emitting nuclides exists, the limits established for alpha- and beta-gamma-emitting nuclides should apply independently.
[b]As used in this table, dpm (disintegrations per minute) means the rate of emission by radioactive material as determined by correcting the counts per minute observed by an appropriate detector for background, efficiency, and geometric factors associated with the instrumentation.
[c]Measurements of average contaminant should not be average over more than 1 square meter. For objects of less surface area, the average should be derived for each such object.
[d]The maximum contamination level applies to an area of not more than 100 cm^2.
[e]The amount of removable radioactive material per 100 cm^2 of surface area should be determined by wiping that area with dry filter or soft absorbent paper, applying moderate pressure, and assessing the amount of radioactive material on the wipe with an appropriate instrument of known efficiency. When removable contamination on objects of less surface area is determined, the pertinent levels should be reduced proportionally and the entire surface should be wiped.

Table 2
CH2M HILL Surface Radioactivity
Action Levels* for Unrestricted Release
of Equipment and Materials

Direct Survey – DPM/100 cm^2		Transferable Survey – DPM/100 cm^2	
Alpha	**Beta-Gamma**	**Alpha**	**Beta-Gamma**
300	1000	20	200

*No 100 cm^2 area greater than this value.

5.3 Direct Surface Contamination Survey

5.3.1 Beta/Gamma Measurement

A. Pass the Geiger-Mueller (GM) detector about 1 cm from the surface or item to be surveyed.

B. Scan the item, covering the potentially contaminated areas, at the rate of 1 to 2 cm per second.

C. Record and identify all locations surveyed. If practical, sketch the item and identify the measurement locations on the sketch.

D. All measurements shall be reported in units of dpm per 100 cm^2.

5.3.2 Direct Alpha Measurements

A. Place the alpha scintillation detector on or near the surface to be surveyed.

B. Scan the item using the fixed point measurement technique and allowing 30 sec per measurement.

C. Record and identify locations surveyed using a sketch, if practical.

D. All measurements shall be reported in units of dpm per 100 cm^2.

5.3.3 Evaluation of Direct Measurements

If the activity for either alpha or beta/gamma is greater than the criteria for direct surface contamination, then the item should be considered for decontamination.

5.4 Transferrable Surface Contamination Survey

5.4.1 Transferrable Surface Contamination Surveys are described in Procedure RP-06.

5.4.2 Counting Transferrable Contamination Smears

Additional instructions for counting smear samples are provided in Procedure RP-06.

A. Gross Alpha Counting

1. Using the scintillation alpha counter, count each smear for 1.0 min.

2. Record the results on the survey form.

3. Compute the dpm/100 cm^2.

B. Gross Beta/Gamma Counting

1. Using the gross beta/gamma counting system, count each smear for 1.0 min.

2. Record the results on the survey form.

3. Compute the dpm/100 cm^2.

C. Quality Control

1. Take one duplicate smear for every 20 smears obtained.

2. The duplicate reading will be recorded on the same survey form with the original measurement and identified as a duplicate reading (D).

CH2M HILL
EQUIPMENT AND MATERIALS RELEASE FORM

DATE _____ SITE _____ SURVEY NUMBER _____ SURVEYORS NAME _____	BETA-GAMMA: DIRECT PROBE _____ BKG. _____ (CPM) EFF. _____	ALPHA: DIRECT PROBE _____ BKG. _____ (CPM) EFF. _____	ALPHA: TRANSFERABLE DETECTOR _____ BKG. _____ (CPM) EFF. _____	BETA-GAMMA: TRANSFERABLE DETECTOR _____ BKG. _____ (CPM) EFF. _____

ITEM SURVEYED FOR RELEASE	DIRECT: ALPHA		DIRECT: BATA-GAMMA		TRANSFERABLE: ALPHA		TRANSFERABLE: BETA-GAMMA		CERTIFIED AS MEETING CRITERIA (%)			COMMENTS
	CPM	DPM/100 cm ^{2}	CPM	DPM/100 cm ^{2}	CPM	DPM/100 cm ^{2}	CPM	DPM/100 cm ^{2}	YES	NO	INITIALS	

APPROVAL

FORM 7

I HAVE REVIEWED THE ABOVE DATA AND, EXCEPT: WHERE NOTED UNDER COMMENTS, ALL MEASUREMENTS CONFORM TO GUIDELINES FOR UNRESTRICTED RELEASE.

_____

SSC/SITE MANAG

Title: Radiological Dose and Exposure Rate Measurements

Document No.: SOP: RP-11

Revision No.: 0

Date: 12/18/92

Approved by: _____ Date: 12/18/92
Radiation Health Manager

RP-11
Radiological Dose and Exposure Rate Measurements

1.0 PURPOSE

The purpose of this procedure is to describe the methods for performing exposure and dose rate measurements during radiological investigations.

2.0 SCOPE

This procedure describes the technique used to identify a gamma-ray exposure rate and beta dose rate for areas under assessment, and for establishing radiological controls for such work areas. The results will determine the need for establishing restricted areas, use of protective clothing and equipment, requiring the use of the Radiation Work Permit, etc.

3.0 REFERENCES

3.1 CH2M HILL Radiation Protection Procedure 1.0, Radiation Protection Terminology and Posting of Areas, RP-01, November 1989

3.2 CH2M HILL Radiation Safety Manual, November 1989

4.0 EQUIPMENT

4.1 Maps and/or drawings

4.2 Ionization Chamber Detector (Eberline RO-2 or equivalent), NBS Traceable Check Source

4.3 Grid markers and measuring devices

Form 11.1, Radiation Dose and Exposure Rate Survey

4.5 Form 11.2, General Radiation Survey Data

4.6 Clipboard and pen

5.0 INSTRUCTIONS

5.1 Prerequisites

5.1.1 Select a beta-gamma dose rate survey instrument and verify that it is calibrated and operating properly in accordance with its specific procedures.

5.1.2 Review past radiological conditions, if available, for the area to be investigated.

5.1.3 Use a map or sketch to show measurement locations for the radiation survey.

5.1.4 The area to be surveyed should be divided into a system that is identified by intervals of length or that includes specific landmarks, allowing for a future duplication of measurements.

5.2 Procedure

5.2.1 Beta measurements are taken as follows:

A. Place the survey instrument with the beta window open on the surface to be measured. Record the measurement in the column for open window (OW).

B. Without moving the instrument, close the beta window and make another measurement at the same location. Record this measurement in the column for closed window (CW).

C. Subtract closed window from open window readings and multiply result by the beta calibration factor (posted on the instrument).

D. The result of the above is the beta dose rate (see Example 7.0).

5.2.2 Gamma measurements are taken as follows:

A. At a distance of 1 m from the surface, obtain another measurement with the beta window closed. Record this measurement in the column for closed window (CW).

5.2.3 Record gamma readings in mR/hr and beta readings in mrad/hr on survey forms in the appropriate columns.

5.3 Quality Control

5.3.1 Perform a duplicate measurement for 1 in every 20 measurements.

5.3.2 If the survey included less than 20 measurement locations, at least one location will be chosen at random.

5.3.3 The duplicate reading will be recorded on the same survey form with the original readings and will be identified as a duplicate measurement (D).

6.0 DOCUMENTATION AND INTERPRETATION OF RESULTS

Measurements and other pertinent information for routine surveys shall be recorded on Radiation Dose and Exposure Rate Survey, Form 11.1. Where documentation is required for unusual or non-routine radiation surveys, General Radiation Survey Data, Form 11.2, shall be completed. Results shall be reviewed by the Radiation Protection Site Supervisor.

7.0 EXAMPLE

Beta dose rate calculation

Calculate the beta dose rate (R_b) given:

Open window reading R_o = 10 mr/hr
Closed window reading R_c = 2 mr/hr
Beta dose correction factor (K) = 3.2

Solution

$R_b = (R_o - R_c) \times K$
$R_b = (10-2) \times 3.2 = 26$ mrad/hr

CH2M HILL
RADIATION DOSE RATE SURVEY FORM

DATE _____ AREA SURVEYED _____

SURVEYOR'S NAME _____ SURVEY NO.* _____

INSTRUMENT MODEL NUMBER _____

INSTRUMENT SERIAL NUMBER _____

INSTRUMENT CALIBRATION DATE _____

INSTRUMENT CALIBRATION FACTOR(S) _____

LOCATION**	AT ONE METER GAMMA		SURFACE CONTAMINATION				
	GROSS	CORRECTED	(a) β +7 (OPEN β)	(b) 7 ONLY (CLOSED β)	β ONLY (a-b)	GAMMA CORRECTED mR/hr	BETA CORRECTED mrad/hr

* FROM SITE SURVEY LOG

** LOCATION: USE LOCATION CODE WHEN APPLICABLE, OTHERWISE DESCRIBE AREA SURVEYED

CH2M HILL
GENERAL RADIATION SURVEY DATA

Form 11.2

Area Surveyed: _____ Date: _____

Surveyor's Name: _____ Survey No.[1]: _____

RWP No.: _____ Time Required to Perform Survey: _____

Instrument No. 1

Ratemeter/Scaler: Model No. _____ Serial No. _____
Detector Probe: Type _____ Model No. _____
Serial No. _____

Instrument No. 2

Ratemeter/Scaler: Model No. _____ Serial No. _____
Detector Probe: Type _____ Model No. _____
Serial No. _____

Air Samples Collected: _____ Yes _____ No
Type Collected: _____ Area _____ Breathing Zone
Results: _____

Transferrable Contamination: Average _____ Maximum _____

Description of Survey (attach additional sheets and sketch, if necessary)

_____

_____

_____

_____

_____

_____

_____

_____

Surveyed by: _____ Reviewed by: _____

_____

[1]From Site Survey Log.

Title: Eberline ESP-2 Digital Ratemeter/Scaler General Setup and Operation

Document No.: SOP: RP-22

Revision No.: 0

Date: 12/18/92

Approved by: _____ Date: 2/18/92
Radiation Health Manager

RP-22
Eberline ESP-2 Digital Ratemeter/Scaler
General Setup and Operation

1.0 PURPOSE

This general procedure describes the Eberline Model ESP-2 digital ratemeter/ scaler. Included are operational specifications, calibration details, and operational and maintenance information. This procedure is not intended to take the place of the ESP-2 Technical Manual that describes the complete capabilities, uses, and operational/maintenance details of the instrument, but should be utilized as a reference guide for field use. Details for using specific detectors with the ESP-2 are described in Standard Operating Procedures (SOPs) RP-23 through RP-26.

2.0 DESCRIPTION OF INSTRUMENT

The ESP-2 (Eberline Smart Portable) is a microcomputer-based portable radiation survey meter that allows the user to detect and measure alpha, beta, and gamma radiation. The ESP-2 is manufactured by TMA/Eberline Instrument Corporation of Santa Fe, New Mexico, and is capable of storing the informational parameters for up to three separate detectors. This information consists of calibration constants, high voltage settings, dead time, units of measure, and alarm setpoints, and has already been preset into the ESP-2 for each detector.

The ESP-2 can function as a ratemeter or scaler and will display the appropriate radiation units along with the data. Ratemeter readings are both analog and digital. In the ratemeter mode, the microcomputer calculates counts per second, divides by the calibration constant, and displays the digital value along with the appropriate units selected. A moving bar graph representation is also shown on the liquid crystal display (LCD) in the ratemeter mode. This allows the user to instantly observe an increase or decrease in count rate; the length of the bar graph is proportional to the activity at the detector.

In the scaler mode, the detector signal is integrated for a selected count time (1 sec to 4 hr), with the time remaining shown on the top line. When the integrated counts exceed the selected alarm setpoint, the alarm sounds. This feature can be used to tell a work party when to leave the radiation area.

The four detectors contained in each CH2M HILL field kit are each designed to measure a specific type of radiation and are compatible with the ESP-2. Each detector sends (provides) a pulse signal to the electronics package within the ESP-2 for counting purposes. This pulse rate from the detector is proportional to the radiation field intensity at the detector face.

2.1 Specifications

2.1.1 Mechanical (with batteries, excluding probe)

A. Overall case dimensions (including all protrusions):
L = 10.5 in. (26.7 cm), H = 5.0 in. (12.7 cm), W = 5.2 in. (13.2 cm)

B. Moisture Resistance: Low, case seal not waterproof.

C. Weight: Approximately 3.8 lb (1.73 kg).

2.1.2 Temperature

Operating:	-20°C to +50°C (-4°F to 122°F)
Storage:	-30°C to +80°C (-22°F to 176°F)

2.1.3 Voltages

A. Low Voltage: 5 Vdc

B. High Voltage (detector bias voltage): 500 to 2450 Vdc, adjustable from the keypad

C. Battery Supply Voltage: 5.8 to 10.0 Vdc

2.1.4 Detectors

Most Eberline GM, proportional, or scintillation detectors for alpha, beta, gamma, or neutron activity may be used on the ESP-2. The detectors connect to the ESP-2 via an MHV-series coaxial connector located on the front of the instrument.

2.1.5 Readout

Two lines of 16 alphanumeric characters presented on LCD.

2.1.6 Alarm

A 2,000-Hz audio tone is generated from the speaker for the Ratemeter and Scaler Operating Modes.

2.1.7 External Controls

A single row of seven 3/8-in.-square pushbutton switches on 1/2-in. centers across the face of the instrument.

2.1.8 Internal Controls

The internal controls consist of four potentiometers and five switches accessible by removing the side access panel.

2.1.9 Power Supply

The ESP-2 uses six C-cell batteries providing 120 and 300 hr of continuous use with carbon-zinc and alkaline batteries, respectively. The unit signals the user by blinking the first character on the display to indicate a low battery condition.

2.1.10 Data

Date-time, location, reading, instrument status

2.1.11 Operating Modes

Ratemeter:

A. Normal (fast or slow time constant)
B. Peak trap (fast or slow time constant)

Scaler:

A. Fixed precision 2, 5, 8, or 10%
B. Integrating
C. Average rate

2.1.12 Multiple Detectors

Allows user to preset operating parameters for up to three detectors. Detectors can be switched, and operation with a new detector is as simple as selecting the detector by user-defined identification (up to 13 characters).

3.0 CALIBRATION

The ESP-2 is calibrated on a biyearly basis by the manufacturer, i.e, TMA/Eberline. The type of calibration source used is dependent upon the detector chosen. Below is a list of detectors and the respective sources used by TMA/Eberline for ESP-2 calibration.

Detector	Source	Activity/Intensity[a]
AC3-7	Th-230	22,400 d/m
HP-260	Sr/Y-90/Tc-99	13,600 d/m/7,840 d/m
HP-270	Ra-226	SN: E554

Detector	Source	Activity/Intensity[a]
SPA-3	Am-241	152,000 d/m
SPA-8	Am-241	152,000 d/m

[a]The activity/intensity values vary according to TMA/ Eberline selection. Listed values are for calibrations conducted on April 10, April 27, April 28, and April 30, 1992.

The ESP-2 and the detectors shown above are calibrated to a primary standard traceable to the National Institute of Standards and Technology (NIST) during each biyearly calibration.

4.0 OPERATION

The ESP-2 field kit contains the radiation survey instrument, four different detector probes, three detector cables, three check sources, a shippers declaration (for transportation purposes), and an instruction manual. Headphones for the ESP-2 are available from ORO. Operational instructions for the ESP-2 are included in Procedures RP-23 through RP-26 covering operation with the AC-3, HP-260, HP-270, and SPA-3 detectors. The instrument source check techniques are included in each applicable detector procedure. Tc-99, Cs-137, and Th-230 low-level check sources are normally contained within the field kit for operational checks.

5.0 MAINTENANCE

The ESP-2 digital ratemeter/scaler requires little user maintenance. Unit should be wiped clean with a moist towel if it becomes soiled. Unit must not be immersed in any liquid. Power supply batteries should be replaced every 6 months, or more often under circumstances of temperature extremes or extensive use. If the ESP-2 unit fails to operate for any reason, contact the CH2M HILL instrument coordinator at 1-615-483-9032 for advice or instructions.

Title: Eberline Model ESP-2, Digital Ratemeter/Scaler and SPA-3 Detector

Document No.: SOP: RP-23

Revision No.: 0

Date: 12/18/92

Approved by: George Stanley
Radiation Health Manager

Date: 12/18/92

RP-23
Eberline Model ESP-2 Digital Ratemeter/Scaler and SPA-3 Detector

1.0 PURPOSE

This procedure describes the operation of the ESP-2 digital ratemeter/scaler and SPA-3 scintillation detector for evaluation of gamma fields and surface contamination. A description of the detector, specifications, calibration information, a step-by-step operating section, and maintenance requirements are included.

2.0 DESCRIPTION

2.1 General

The Eberline Model SPA-3 scintillation detector is a cylindrical aluminum tube containing a 2-in.-diameter × 2-in.-long NaI (T1) crystal, a 10-stage photomultiplier tube, tube socket with a dynode resistor string, and a magnetic shield. The SPA-3 may be used in ratemeter or pulse-height applications.

2.2 Specifications

2.2.1 Crystal: NaI (T1), 2-in.-diameter × 2-in.-long (5.1 cm × 5.1 cm)

2.2.2 Photomultiplier Tube: ≅2-in.-diameter, 10-dynode, end-window with S-11 photocathode

2.2.3 Operating Voltage: Variable dependent upon application

2.2.4 Maximum Voltage: +1,600 V

2.2.5 Sensitivity: ≅1,200 k cpm per mR/h with Cs-137

2.2.6 Wall Material: Aluminum

2.2.7 Wall Thickness: 1/8-in. (0.32 cm), 1/16-in. (0.16 cm) at crystal

2.2.8 Size: 2-5/8-in.-diameter × 11-1/8-in.-long (6.7 cm × 28.3 cm)

2.2.9 Weight: 3.25 lb (1.5 kg)

3.0 CALIBRATION

The Eberline SPA-3 detector is calibrated on a biyearly basis by the manufacturer, TMA/Eberline. Calibration data are maintained on file in ORO and are readily accessible.

4.0 SETUP AND SOURCE CHECK (RATEMETER MODE)

Instrument source checks will be performed once per day prior to the start of field activities, at a minimum.

4.1 Remove the SPA-3 detector probe from the kit.

4.2 Remove the detector cable identified with green tape from the kit and install on the SPA-3 probe.

4.3 Press the ON/OFF ESP-2 control panel key. A few seconds will elapse before the digital display is activated. Set up for this probe usually requires reprogramming the ESP-2.

4.4 Press MODE/STR=ORE key. The display should read "LOG THIS VALUE?".

4.5 Press -/CRSR key two times. The name of a detector should be displayed. If detector is the SPA-3, go to Step 9. If the detector name is not SPA-3, then press RESET and -/CRSR. Note the name of next detector. If SPA-3, go to Step 9; if not, press -/CRSR. If display indicates that the next detector is the SPA-3, go to Step 9. If the third detector is not the SPA-3, then press the -/CRSR key until the display shows the name of the detector not to be used for your job. The name and parameters for this detector will be erased and the SPA-3 detector parameters will be installed in its place.

4.6 Press the +/INC key. "EDIT DET.INFO?" will be displayed. Press +/INC key again. Display will read "RESET WHEN DONE" and a cursor will underline the first digit of the old detector name.

4.7 Press the +/INC key until the "S" (in SPA-3) is displayed. When correct, move the cursor one space to the right by pressing the -/CRSR key once. As before, press the +/INC key until the "P" (in SPA-3) is displayed. Move right with the -/CRSR key and continue with this technique until "SPA-3" is spelled out on the display. Press the RESET key.

4.8 The detector number and SPA-3 should be displayed in the upper left- hand corner. Press the -/CRSR key to begin parameter adjustments. "HV=? (same value)" should be displayed. For general SPA-3, a high voltage value of 850 V may be used. Press and hold down the RESET button. Now press the +/INC key (to increase the voltage value) or the -/CRSR key (to decrease the voltage), as applicable, to obtain 850 V. It may not be possible to get exactly 850, so $\pm$10 V is acceptable. Release the RESET key and press the -/CRSR key. By holding down the RESET and pressing the + or = keys, adjust the DT (dead time) parameter to approximately 1×10^{-6} sec. When complete, press the -/CRSR key and adjust, as before, the CC (correction coefficient) to 1.00. Press the -/CRSR and adjust the ALM (alarm setpoint) to 1.00×10^{6}. Press -/CRSR. Units should be expressed in count/min. If not, press RESET key and the + or - key, as applicable, to adjust the units. When correct, go to Step 9. Unit has now been reprogrammed for use with the SPA-3 probe.

4.9 Press the MODE/STORE key.

4.10 The ESP-2 is now set for ratemeter use with output displayed in count/min. To source check the instrument and probe, first obtain one of the field kit Cs-137, 1 μCi, check sources. Observe and record the general gamma background count value. In a normal office or other low background area, the value should be approximately 3 to 7K count/min. Next, balance the Cs-137 source, serial number up, on the uppermost end of the probe near the small ledge. Allow the reading to stabilize and record the value as gross source count. Now determine the net source count by subtracting the gamma background count value from the gross source count value. The derived net source count value should be about 263,000 $\pm$ 5,200 count/min.[1] The instrument should not be used if response is not within the range specified above.

5.0 OPERATION

5.1 To scan for contaminated areas or hot spots, the SPA-3 detector is kept as close as possible to the surface and is moved back and forth. Walking speed over the surface should be about 0.5 m per second. For optimum detection, sensitivity changes in the instrument response are monitored via the audible output rather than by the digital meter reading. Locations of direct radiation, discernible above the ambient level, should be marked on facility maps and field marked with tape, paint, pin flags, etc., and identified for further measurements and/or sampling.

5.2 If the SPA-3 data are to be converted to exposure rate, then measurements should be conducted at 1 m from the surface.

6.0 MAINTENANCE

Maintenance on the SPA is performed by the manufacturer. If the SPA-3 does not respond to a source check, as required in Section 4.0, the detector should be taken out of service and returned to the manufacturer for maintenance and recalibration.

[1]This value is experimentally derived. See ESP-2 calibration data logbook, SPA-3 section.

Title: Eberline Model ESP-2 Digital Ratemeter/Scaler and AC-3 Detector

Document No.: SOP: RP-24

Revision No.: 0

Date: 12/18/92

Approved by: _____ Date: 12/18/92
Radiation Health Manager

RP-24
Eberline Model ESP-2 Digital Ratemeter/Scaler and AC-3 Detector

1.0 PURPOSE

This procedure describes the operation of the ESP-2 digital ratemeter/scaler and AC-3 detector for evaluation of alpha surface contamination. Included are a description of the detector, specifications, calibration information, a step-by-step operating section, and maintenance techniques.

2.0 DESCRIPTION

2.1 General

The Eberline AC-3 alpha scintillation detector is designed for use with the ESP-2 and other Eberline radiation detection instruments. The AC-3 is a rugged unit developed for general alpha surveys and personnel alpha monitoring. There are two versions of the AC-3, differing only in the window assembly. The AC-3-7 designates a maximum open window area for alpha surveys, and the AC-3-8 designates a rugged window that has a fine mesh protector over the Mylar for personnel monitoring. The CH2M HILL field kits contain the AC-3-7 version for surveys of maximum sensitivity and detection capability. The Mylar window is a "sandwich" assembly that can be replaced by the removal of six screws. A clean plastic probe face cover is supplied to protect the window when the probe is not in use.

2.1 Specifications

2.2.1 Active Area: 9.1-in.^{2} (59 cm^2) within 5.75-in. × 2-in. (14.6 cm × 5.1 cm) sampling area

2.1.2 Window Thickness: 0.85 mg/cm^2 aluminized Mylar

2.1.3 Efficiency: From a 1-in.-diameter source or from 59 cm^2 of a large distributed area Pu-239 source (2π geometry). AC-3-7 window: 28 percent minimum, 31 percent typical.

2.1.4 Sensitivity: For a large area Pu-239 source, AC-3-7 window: typically 2×10^7 counts per minute per μCi/cm^2 (9 cpm per dpm/cm^2)

2.1.5 Gamma Sensitivity: Insensitive to gamma radiation

2.1.6 Scintillator: ZnS(Ag) power embedded on tape

2.1.7 Operating Voltage: Optimum voltage depends on phototube characteristics, cable length, input impedance, and sensitivity of counter

2.1.8 Maximum Voltage: +1,600 V

2.1.9 Temperature Range: -40°F to 140°F (-40°C to +60°C)

2.1.10 Weight: 1 lb, 6 oz (0.62 kg)

3.0 CALIBRATION

The AC-3 detector is calibrated by the manufacturer on a biyearly basis. Calibration data are maintained on file in the CH2M HILL ORO Office and are readily available.

4.0 SETUP AND SOURCE CHECK (SCALER MODEL)

Instrument source checks shall be performed once per day prior to the start of field activities at a minimum.

4.1 Remove the AC-3 detector probe from the kit. Remove the clear plastic protector from the probe face.

4.2 Remove the detector cable (identified with green tape) from the kit and install on the AC-3 probe (also identified with green tape).

4.3 Connect the opposite end of the cable to the front connector on the ESP-2 unit.

4.4 Press the ON/OFF ESP-2 control panel key. A few seconds will elapse before the digital display is activated. To verify that the ESP-2 is set up for the detector probe, Steps 5 through 7 are required.

4.5 Press MODE/STORE key. The display should read "LOG THIS VALUE?".

4.6 Press -/CRSR key once or twice until the detector name (AC-3-7) is displayed. If incorrect detector name, go to Step 8.

4.7 Press the MODE/STORE key to return to normal display mode. Go to Step 12.

4.8 Press the RESET key. The display will show "USE DET. #__?" and the name of the detector for which the operating parameters are currently set.

4.9 Press the -/CRSR key until the name of the appropriate detector probe is displayed.

4.10 Press the +/INC key to select the detector probe.

4.11 Press the MODE/STORE key to return to the normal display mode.

4.12 If the unit is already set up for the scaler mode of operation, go to Step 16.

4.13 Press MODE/STORE, and then press -/CRSR four times. The display will indicate "RATEMETER MODE."

4.14 Press RESET, then +/INC key. Unit is now set for the scaler mode.

4.15 Press -/CRSR three times, then MODE/STORE. Unit is ready for a background count.

4.16 Place the AC-3 detector probe face down on a clean flat surface.

4.17 Press RESET. At the end of a 1-min count, the ESP-2 will beep. Record the 1-min background count as "BKG." By pressing RESET, obtain a total of three background measurements. Average the three "BKG" values and record as "AVE.BKG."

4.18 Remove the envelope containing the alpha check source from the kit (labeled K1C or K2C).

4.19 Remove the source from the envelope and place the source label down on a clean flat surface.

4.20 Place the detector probe face squarely over the check source. Place a small weight on the back of the probe if necessary.

4.21 Press RESET, as in Step 17, and record each 1-min source count as "D/M." Average the three "D/M" and record as "AVE.D/M."

4.22 Subtract "AVE.BKG" from "AVE.D/M" to obtain the source activity in disintegrations per minute. The "AVE.D/M" should be within the range listed below:

ESP-2 Serial Number	Source Serial Number	Source Activity in D/M
847	K1C	17,554 $\pm$ 3,510[a]
848	K2C	16,234 $\pm$ 3,247[a]
[a]February 21, 1990.		

If the "AVE.D/M" value does not fall within the above range, do not use the instrument. Return the AC-3 to ORO for recalibration.

4.23 If the source check is acceptable, then the unit is ready for use in the scaler mode.

5.0 OPERATION

5.1 Surface being surveyed shall be dry.

5.2 Remove plastic face cover. Extra care should be taken not to damage the aluminized mylar face.

5.3 The detector should be held as close as possible to (without contacting) the surface being surveyed.

5.4 Fixed point measurements shall have a minimum count time of 0.5 min.

5.5 Survey scanning speed should not be more than 1 in. per second. Thorough monitoring requires detector to remain immobile or in a fixed position for approximately 5 sec.

5.6 Caution should be taken not to contaminate the detector when obvious surface contamination is present.

5.7 Collect count data using either the scaler or ratemeter mode. Record the results of the count in a field logbook or on the appropriate survey form.

5.8 Replace the plastic face cover on the detector face and turn instrument off when not in use.

6.0 MAINTENANCE

In general, maintenance is conducted on the AC-3 probe by the manufacturer prior to calibration. This includes basic cleaning and inspection of the outer casing, window grating, connector, and mylar window. Internally, the scintillator material, electronic components, and electrical connections are inspected. Frequently, in the field, the mylar window is scratched or damaged causing a condition known as a "light leak." This condition renders the AC-3 probe useless for alpha measurements. Field repairs may be made to the mylar surface by covering the damaged area with a small piece of electrical tape or fast-drying black enamel paint. No more than 10 percent of the active detector surface area should be repaired in this manner. As soon as practical, the mylar should be replaced by trained serviee personnel.

Title: Eberline Model ESP-2 Ratemeter/Scaler and HP-270 Detector

Document No.: SOP: RP-25

Revision No.: 0

Date: 12/18/92

Approved by: _____ Date: 12/18/92
Radiation Health Manager

RP-25
Eberline Model ESP-2 Ratemeter/Scaler and HP-270 Detector

1.0 PURPOSE

This procedure describes the operation of the ESP-2 digital ratemeter/scaler and HP-270 detector for evaluation of beta/gama exposure rates and surface contamination. A description of the detector, specifications, calibration information, a step-by-step operating section, and maintenance techniques are included.

2.0 DESCRIPTION

2.1 General

The Eberline Model HP-270 detector contains an energy compensated cylindrical GM probe and integral beta shield. The energy compensation feature permits reliable exposure rate measurement from background to 200 mR/h.

2.2 Specifications

2.2.1 Operating Voltage: 900 $\pm$ 50 V

2.2.2 Temperature Range: -40°F to + 167°F

2.2.3 Wall Thickness: 30 mg/cm^2 (tube only)

2.2.4 Wall Material: Stainless steel

2.2.5 Gamma Sensitivity: Approximately 1,200 cpm/mR/h (Cs-137)

2.2.6 Energy Response: Nonlinear

2.2.7 Size: 1.38-in. diameter, 6-in. long (3.5 cm $\times$ 15.2 cm)

2.2.8 Weight: 5 oz (142 g)

3.0 CALIBRATION

The HP-270 is calibrated biyearly by TMA/Eberline. Calibration data are maintained on file and are readily available.

4.0 SETUP AND SOURCE CHECK (RATEMETER MODE)

Instrument source checks will be performed once per day prior to the start of field activities, at a minimum.

4.1 Remove the HP-270 detector probe from the kit. Slide the black rubber probe shield to the open position.

4.2 Remove the detector cable (identified with yellow tape) from the kit and install on the HP-270 probe (also identified with yellow tape).

4.3 Connect the opposite end of the cable to the front connector on the ESP-2 unit.

4.4 Press the ON/OFF ESP-2 control panel key. A few seconds will elapse before the digital display is activated. To verify that the ESP-2 is set up for the detector probe, Steps 5 through 7 are required.

4.5 Press MODE/STORE key. The display should read "LOG THIS VALUE?".

4.6 Press -/CRSR key once or twice until the detector name (HP-270) is displayed. If incorrect detector name, go to Step 8.

4.7 Press the MODE/STORE key to return to normal display mode. Go to Step 12.

4.8 Press the RESET key. The display will show "USE DET. #__?" and the name of the detector for which the operating parameters are currently set.

4.9 Press the -/CRSR key until the name of the appropriate detector probe is displayed.

4.10 Press the +/INC key to select the detector probe.

4.11 Press the MODE/STORE key to return to the normal display mode.

4.12 If the unit is already set up for the scaler mode of operation, go to Step 16.

4.13 Press MODE/STORE, and then press -/CRSR four times. The display will indicate "RATEMETER MODE."

4.14 Press RESET, then +/INC key. Unit is now set for the scaler mode.

4.15 Press -/CRSR three times, then MODE/STORE. Unit is ready for a background measurement.

4.16 Place the HP-270 detector probe on a clean flat surface.

4.17 Press RESET. At the end of a 1-min count, the ESP-2 will beep. Record the 1-min background count as "BKG." By pressing RESET, obtain a total of three background measurements. Average the three "BKG" values and record as "AVE.BKG."

4.18 Remove the envelope containing the Cs-137 alpha check source from the kit (labeled K1B or K2B).

4.19 Remove the source from the envelope and place the source label down on a clean flat surface.

4.20 Place the detector probe face squarely over the check source. Place a small weight on the back of the probe if necessary.

4.21 Press RESET, as in Step 17, and record each 1-min source count as "mR/h." Average the three "mR/h and record as "AVE.mR/h."

4.22 Subtract "AVE.BKG" from "AVE.mR/h" to obtain the source activity in disintegrations per minute. The "AVE.mR/h" should be within the range listed below:

ESP-2 Serial Number	Source Serial Number	Source Activity in mR/h
847	K1B	3.7 $\pm$ 0.37
848	K2B	3.7 $\pm$ 0.37

If the "AVE.mR/h" value does not fall within the above range, do not use the instrument. *Report high or low counts to appropriate personnel.*

4.23 Press MODE/STORE key, then -/CRSR five times. The display will read "SCALER MODE - RESET TO CHG."

4.24 Press RESET, then -/CRSR two times.

4.25 Press MODE/STORE key. Unit is now set up for use in the ratemeter mode.

5.0 OPERATION

5.1 The HP-270 detector can be used in two modes:

A. Window open – beta/gamma measurement
B. Window closed – gamma measurement

5.2 Typically, the detector is used with the window closed to reduce over-response to low-energy gamma radiation ($\leq$200 keV).

5.3 With the high voltage set at the calibrated value, measurements can be obtained adjacent to or at a distance (1 m) from the surface to be measured.

5.4 *Collect exposure rate data and record the results of the measurement in a field logbook or* on the appropriate survey form.

5.5 Count rate from this instrument may be used to measure gamma-ray exposure rates and is user selectable for mR/h or μR/h.

6.0 MAINTENANCE

Maintenance is conducted during the biyearly calibration by the manufacturer. There are no user serviceable components within the HP-270.

Title: Eberline Model ESP-2 Digital Ratemeter/Scaler and HP-210/HP-260 Detectors

Document No.: SOP: RP-26

Revision No.: 0

Date: 12/18/92

Approved by: George Stanley
Radiation Health Manager

Date: 12/18/92

RP-26
Eberline Model ESP-2 Digital Ratemeter/Scaler and HP-210/HP-260 Detectors

1.0 PURPOSE

This procedure describes the operation of the ESP-2 digital ratemeter/scaler and HP-210/ HP-260 detectors for evaluation of beta/gamma surface contamination. Included is a description of the detector, specifications, calibration information, a step-by-step operating section, and maintenance requirements.

2.0 DESCRIPTION

The Eberline Model HP-210AL and HP-260 series hand probes provide a sensitive beta detector featuring a "Pancake" GM tube with a thin mica window. The open window, which is protected by a sturdy wire screen, permits useful beta sensitivities down to 40 keV. The detector is alpha sensitive (above 3 MeV).

Both detectors are designed for contamination surveys on personnel, table tops, floors, equipment, etc. The HP-210AL with an aluminum housing is especially suited for low-level beta monitoring in a low background area. The HP-210AL may also be used with an Eberline SH-4A source holder for beta/gamma smear counting.

2.1 Specifications

	HP-210AL	HP-260
2.1.1 Operating Voltage:	900 ± 50 V	900 ± 50 V
2.1.2 Temperature:	-22°F to +167°F (-30°C to +75°C)	-22°F to +167°F (30°C to +75°C)
2.1.3 Mica Window Thickness:	1.4 to 2.0 mg/cm^2	1.4 to 2.0 mg/cm^2
2.1.4 Mica Window Size:	1.75 in. diameter (4.45 cm) 2.4 in.^{2}	1.75 in. diameter (4.45 cm) 2.4 in.^{2}
2.1.5 Gamma Sensitivity:	≅3,600 cpm/mR/h (^{137}Cs) (into window)	≅3,600 cpm/mR/h (^{137}Cs)
2.1.6 Beta Efficiency*	≅45% ^{90}Sr-^{90} Y ≅30% ^{99}Tc ≅10% ^{14}C	≅45% ^{90}Sr-^{90} ≅30% ^{99}Tc ≅10% ^{14}C
2.1.7 Alpha Sensitivity:	3 MeV at window	3 MeV at window

	HP-210AL	HP-260
2.1.8 Size:	6.5 in. long × 3.5 in. wide × 3.8 in. high (16.2 cm × 8.8 cm × 9.6 cm)	10 in. long × 2.7 in. wide × 2.5 in. high (25.4 cm × 7 cm × 6.4 cm)
2.1.9 Weight:	1.5 lb (0.7 kg)	1.25 lb (0.57 kg)
*Efficiencies with screen in place. Screen removal will increase efficiency by 45% of stated value. Efficiencies listed as percentage of 2π emission rate from a 1-in.-diameter source.		

3.0 CALIBRATION

The HP-210AL and HP-260 is calibrated biyearly by the manufacturer to Sr-90 and Tc-99 standards. Calibration data are maintained on file in ORO and are readily accessible.

4.0 SETUP AND SOURCE CHECK (SCALER MODE)

Source check must be performed, once, at the beginning of each shift or prior to use each day. The following describes the setup and source check procedure.

4.1 Remove detector probe from the kit. Remove the red plastic protector from the probe face (if applicable).

4.2 Remove the detector cable (identified with blue tape) from the kit and install on the probe (also identified with blue tape).

Note: The cable should not be connected to the ESP-2 until the correct detector is verified.

4.3 Press the ON/OFF ESP-2 control panel key. A few seconds will elapse before the digital display is activated. To verify that the ESP-2 is set up for the proper detector probe, Steps 4 through 6 are required.

4.4 Press MODE/STORE key. The display should read "LOG THIS VALUE?".

4.5 Press -/CRSR key twice until the detector name is displayed. If incorrect detector name, go to Step 7. Correct detector name is HP-260. This detector name is used when either the HP-260 or the HP-210AL probe is installed.

4.6 Press the MODE/STORE key to return to normal display mode. Connect the probe cable to the ESP-2 after the detector name (HP-260) has been verified. Go to Step 11.

4.7 Press the RESET key. The display will show "USE DET. # __?" and the name of the detector for which the operating parameters are currently set.

4.8 Press the -/CRSR key until the name of the appropriate detector probe is displayed.

4.9 Press the +/INC key to select the detector probe.

4.10 Press the MODE/STORE key to return to the normal display mode.

4.11 If the unit is already set up for the scaler mode of operation, go to Step 15.

4.12 Press MODE/STORE, and then press -/CRSR four times. The display will indicate "RATEMETER MODE."

4.13 Press RESET, then +/INC key. Unit is now set for the scaler mode.

4.14 Press -/CRSR three times, then MODE/STORE. Unit is ready for a background count.

4.15 Place the detector probe face down on a clean flat surface.

4.16 Press RESET. At the end of a 1-min count, the ESP-2 will beep. Record the 1-min background count as "BKG." By pressing RESET, obtain a total of three background measurements. Average the three "BKG" values and record as "AVE.BKG."

4.17 Remove the envelope containing the beta check source from the kit (labeled K1A or K2A).

4.18 Remove the source from the envelope and place the source label down in an Eberline SH-4A source holder.[1]

4.19 Place the detector probe face squarely over the check source. Place a small weight on the back of the probe if necessary.

4.20 Press RESET as in Step 16 and record each 1-min source count as "CTS." Average the three "CTS" and record as "AVE.CTS."

4.21 Subtract "AVE.BKG" from "AVE.CTS" to obtain the source activity in counts per minute "ACT.CTS." The efficiency of the ESP-2 units with HP-260 probes installed is as follows:

Serial No. 846 (Kit 1) EFF* = 9.8% (TC-98)
Serial No. 848 (Kit 2) EFF* = 9.496% 10.3% (Tc-99)

[1] If an Eberline SH-4A is not available, place the source as close to the detector face as possible.

ESP-2 Serial Number	Source Serial Number	Source Activity in DPM
847	K1A	43,217 ± 10%*
848	K2A	28,476 ± 10%
*EFF value good until October 27, 1992. After October 27, refer to calibration data sheets on file.		

If the AVE.DPM value does not fall within the above range, do not use the instrument. Report high or low counts to appropriate personnel.

4.22 If the source check is acceptable, then the unit is ready for use in the scaler mode.

4.23 To change instrument to ratemeter mode, press MODE/STORE, then -/CRSR five times, then RESET, then -/CRSR twice, then MODE/STORE.

5.0 OPERATION

5.1 The surface being surveyed should be dry.

5.2 Determine background count rate and source check the HP-260/ESP-2.

5.3 Remove plastic cover.

5.4 The detector should be held as close as possible to the surface being surveyed.

5.5 Fixed-point measurements shall have a minimum count time of 0.5 min (30 sec).

5.6 Survey scanning speed should not be more than 1 in. per second. Thorough monitoring requires the detector to remain immobile or in a fixed position for approximately 5 sec.

5.7 Caution should be taken not to contaminate the detector when obvious surface contamination is present.

5.8 Always replace the plastic cover on the detector face to prevent puncturing the GM tube, and turn instrument off when not in use.

6.0 MAINTENANCE

Maintenance, if required, is conducted during the biyearly calibration by the manufacturer. There are no user serviceable components within the HP-210AL or HP-260.

Title: Setup and Operation of a Pressurized Ion Chamber

Document No.: SOP: RP-27

Revision No.: 0

Date: 12/18/92

Approved by: _____ Date: 12/18/92
Radiation Health Manager

RP-27
Setup and Operation of a Pressurized Ion Chamber

1.0 PURPOSE

To describe the operation, calibration, and maintenance of a pressurized ion chamber (PIC).

2.0 REFERENCES

2.1 Oak Ridge Institute for Science and Education (ORISE), Environmental Survey and Site Assessment Program, Survey Procedures Manual.

3.0 CALIBRATION AND SETUP

3.1 The PIC is used as a secondary standard for cross-calibration of other gamma measuring instruments. Calibration of the PIC to a primary standard traceable to the National Institute of Standards and Technology (NIST) is performed by the manufacturer. The PIC shall be recalibrated twice each year by the manufacturer.

3.2 An initial checkout of the PIC shall be performed as soon after calibration as possible. The results of this initial checkout will be used for subsequent field source and operability checks of the PIC.

3.2.1 Assemble the PIC and turn the instrument on. Check each of the batteries and allow the instrument to stabilize for 5 min.

3.2.2 Determine the background exposure rate and record on the CH2M HILL Background and Source Check Form.

3.2.3 Determine the instrument response to a 1 μCi Cs-137 source (in contact with the ionization chamber wall) and record the results on the CH2M HILL Background and Source Check Form.

3.2.4 The readings obtained in Steps 3.2.2 and 3.2.3 should be logged in the PIC maintenance and calibration notebook for use in comparison to subsequent field source and background checks.

4.0 OPERATION

4.1 Perform a source and background check of the PIC prior to transport to a field site.

4.2 Record the results of the source and background check on the CH2M HILL Background and Source Check Form.

4.3 Compare the results of the current source and background check to the results of the source and background check performed after calibration. The current source and

background check results should be within ± 10 percent of the results obtained after calibration. If the results fall outside of this range, remove the instrument from service and ship to the manufacturer for repairs.

4.4 Move the PIC to the field location, assemble, check the battery charge, and perform a field background and source check as specified in Step 4.3 above.

4.5 Set up the PIC at the survey location and accumulate integrated exposure rate data for at least 5 min. Record the results of each measurement in the field logbook.

5.0 MAINTENANCE

5.1 Maintenance for the PIC is performed by the manufacturer. The PIC should be shipped back to the manufacturer when the source or background checks fall outside of the acceptable range or when recalibration is due.

Title: Calibration of NaI Detectors for Exposure Rate Measurements

Document No.: SOP: RP-28

Revision No.: 0

Date: 12/18/92

Approved by: _____ Date: 12/18/92
Radiation Health Manager

RP-28
Calibration of NaI Detectors for Exposure Rate Measurements

1.0 PURPOSE

The purpose of this procedure is to describe the methods for calibrating NaI detectors for use in determining exposure rates.

2.0 SCOPE

This procedure describes methods for calibrating standard 1 in. × 1 in. or 2 in. × 2 in. NaI detectors for use in exposure rate measurements using a known gamma spectrum. This method allows an approximation of μR/hr from the count rate obtained from a low-level gamma scintillation detector.

3.0 REFERENCES

3.1 Manual for Conducting Radiological Surveys in Support of License Termination, NUREG/CR-5849, ORAU-92/C57

3.2 Oak Ridge Institute for Science and Education Environmental Survey and Site Assessment Program Survey Procedures Manual

4.0 EQUIPMENT AND MATERIALS

4.1 NaI gamma scintillation detector(s) (Eberline SPA-3 or equivalent) and ratemeter(s) (Eberline ESP-2 or equivalent)

4.2 Pressurized ionization chamber (PIC) direct reading exposure rate instrument (Rueter Stokes Model RSS-111 or equivalent)

4.3 Graph paper

4.4 Hewlett Packard HP-15C scientific calculator with linear regression analysis capability (or equivalent).

4.5 Site map and logbook

4.6 Measuring tape or pole

5.0 PROCEDURE

5.1 Field Measurements

5.1.1 Move instruments to the field location where measurements will be taken as part of site characterization activities. The calibration measurements must be made in

the same area, with exposure to the same mixture of gamma energies, as the measurements made as part of the site characterization surveys.

5.1.2 Establish calibration measurement locations and mark on site map. Calibration measurement locations should be chosen to cover the entire range of expected gamma count rates at the site. At least five measurement locations should be established.

5.1.3 Source check the NaI detector and PIC using the appropriate instrument procedures, and check sources as described in the CH2M HILL Radiation Safety Manual (Procedures RP-23 and RP-27).

5.1.4 Move to the first measurement location and set up the PIC at 1 meter above the ground surface. Check the battery and allow to stabilize for at least 5 min. Determine the exposure rate. Record the results in the logbook. Remove the PIC when the measurement is complete.

5.1.5 Set up the NaI detector and ratemeter system(s) to be calibrated at the same location as the PIC. Collect at least 10,000 counts on the gamma scintillation detector (approximately 1 to 2 min at most locations) using the digital integrator function (use the ESP-2 ratemeter if possible). Record the results in the logbook.

5.1.6 Repeat Step 5.1.5 for each of the NaI detector and ratemeter systems to be calibrated.

Note: If each NaI detector and ratemeter system does not respond within 15 percent of each other, the instruments' high voltage must be adjusted prior to continuing. Send the instruments to ORO for adjustment.

5.1.7 Repeat Steps 5.1.3 through 5.1.4 for each of the calibration measurement locations.

5.2 Data Reduction/Calculations

5.2.1 Calculate the average NaI detector reading at each measurement location by summing the total counts at each location and dividing by the number of detectors used.

5.2.2 Plot the average NaI counts at each location versus the μR/hr reading from the PIC.

5.2.3 Calculate a line of best fit for the graph using the HP-15C calculator (or equivalent) linear regression analysis function.

5.2.4 Calculate the calibration equation for converting cpm to μR/hr using the results of the linear regression performed in Step 5.1.3 above. This equation should be used to convert cpm from the detectors calibrated at the site to μR/hr.

Title: Surface Gamma Scan (Walkover) Survey for Creeks and Streams

Document No.: SOP: RP-29

Revision No.: 0

Date: 12/30/92

Approved by: _____ Date: 12/30/92
Radiation Health Manager

RP-29
Surface Gamma Scan (Walkover) Survey for Creeks and Streams

1.0 PURPOSE

The purpose of this procedure is to describe the methodology used to identify localized radioactive contamination and to establish boundaries of contamination for radiological characterization projects and remedial action activities.

2.0 SCOPE

This procedure includes a description of techniques and instruments used to identify localized radioactive contamination. It also includes a description of procedures for documenting results that can be used to assess response action and determination of personal protective equipment (PPE) requirements.

3.0 REFERENCES

3.1 Manual for Conducting Radiological Surveys in Support of License Termination, NUREG/CR-5849, ORAU-92/C57

3.2 Oak Ridge Institute for Science and Education (ORISE) Environmental Survey and Site Assessment Program Survey Procedures Manual

4.0 EQUIPMENT AND MATERIALS

4.1 Maps and drawings

4.2 Gridmarkers and measuring devices (i.e., pin flags, 100-ft tape measure)

4.3 Portable ratemeter/scaler (Eberline ESP-2 or equivalent with headphones)

4.4 Gamma Scintillation Detector (NaI—Eberline SPA-3 or equivalent)

4.5 Clipboard and pen

5.0 PROCEDURES

5.1 A reference system shall be established along the stream to allow documentation of survey results using maps or overlays with aerial photographs. The reference system will usually consist of measurement of the length of the stream or area to be surveyed, and designation of locations for transects along the length of the stream.

5.2 Source check the gamma scintillation detector/ratemeter-scaler and determine the background count rate. Record these readings on the CH2M HILL Weekly Field Source Check Log.

5.3 Stream Bank Scanning

5.3.1 Turn on the calibrated and source-checked instrument. Check battery position and verify calibration dates. Turn audio response switch on.

5.3.2 The detector shall be held as close to the ground surface as possible without hitting protruding objects (rocks, stumps, etc.). (Do not drag the detector on the ground.)

5.3.3 The area shall be surveyed by swinging the gamma detector in front of the body in a pendulum-like manner, while progressing at a slow walk. The detector should move perpendicular to the ground surface at a rate of 6 to 15 in. per second. The width of the "swing" should be 3 to 4 ft.

5.3.4 Use the audio response of the instrument to locate areas of elevated activity above background radiation levels. Periodically observe the meter to obtain an estimate of the average levels measured.

5.3.5 Traverse the length of the stream bank area from the edge of the waterline of both banks outward to a predetermined distance (e.g., 10 to 20 ft), as access is available. Record scan data in fieldbooks and maps.

5.3.6 Transects will be systematically established at points along the streams. The transects are survey areas perpendicular to and extending outward from the waterline to an end point. Traverse the length and width of the transect [usually predetermined and specified in the Field Sampling Plan (FSP)] to the end point. The end point may be reached when access has been prohibited by structures or dense vegetation; when the area of concern (e.g., 100-year floodplain) has been covered; when the gamma-scan indicates no gamma-radiation greater than background for a continuous distance of 50 ft or more; or as otherwise specified in the FSP.

Additional transects may be added based on the gamma-scan survey of the banks where readings are 3 times background. Record scan data in fieldbooks and on site maps.

5.3.7 The boundary of contamination and elevated levels located during the survey may be marked using flags, spray paint, or marked stakes. The contaminated areas shall be marked and recorded on the survey map.

5.3.8 Localized and elevated gamma radiation levels identified after completion of the scan survey may be further investigated by collecting bias surface soil samples and/or by performing near-surface gamma measurements with a shielded gamma scintillation detector (i.e., coneshield apparatus), depending on the work plan for the site.

Note: For scanning purposes, a hot spot or elevated level of activity is defined as an area where the measured count rate is 1.5 to 3 times the ambient count rate of the instrument (i.e., 1.5 to 3 times the response of the instrument to background).

5.3.9 Walkover scanning data shall be documented on the Surface Gamma Scan Survey Sheet and the survey drawings to identify all localized contamination. Alternatively, the results of the gamma scan may be documented as contour plots on an overlay map of the site.

5.3.10 A typical stream bank survey diagram is shown in Figure 1.

CH2M HILL

Surface Gamma Scan Survey

Project/Site Name: _____ **Surveyor's Name:** _____

Date: _____ **Survey Number:** _____

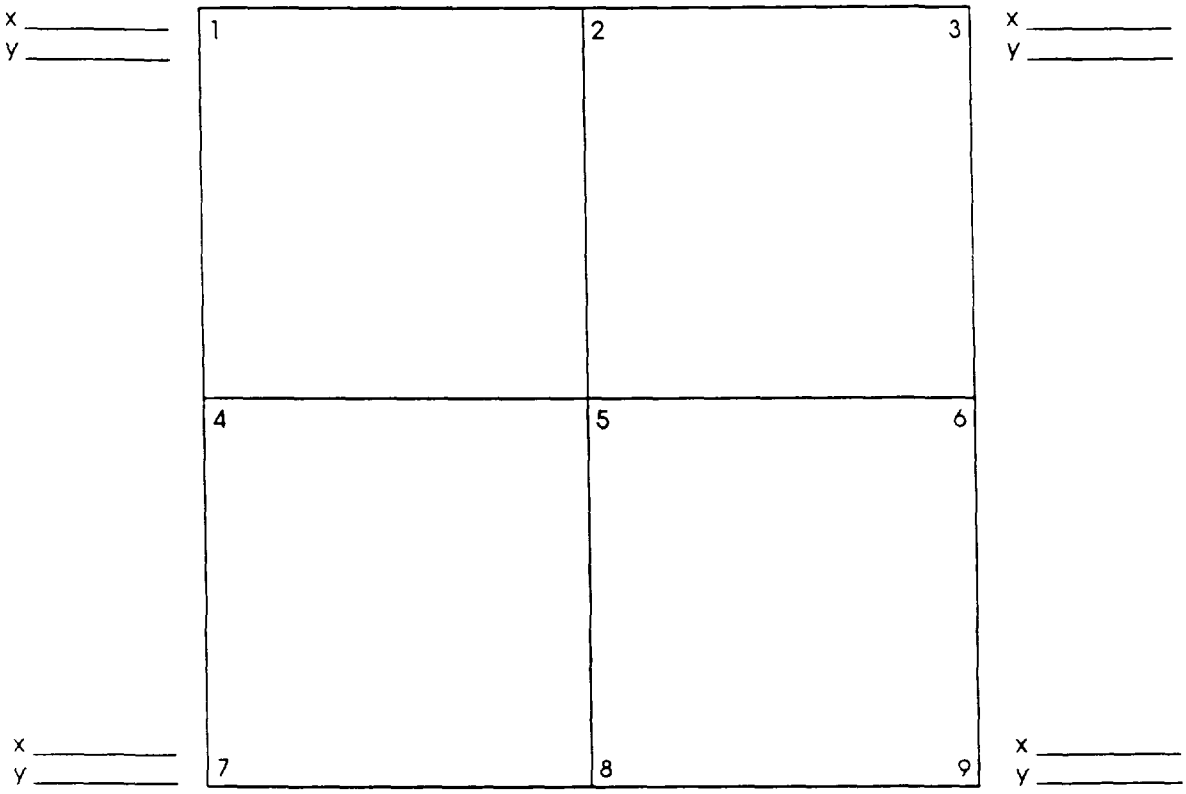
Project Number: _____

GAMMA SURVEY INSTRUMENT

Scaler Model: _____ Detector Model: _____

Serial Number: _____ Serial Number: _____

Offsite Background: _____ cpm Calibration Equation: _____

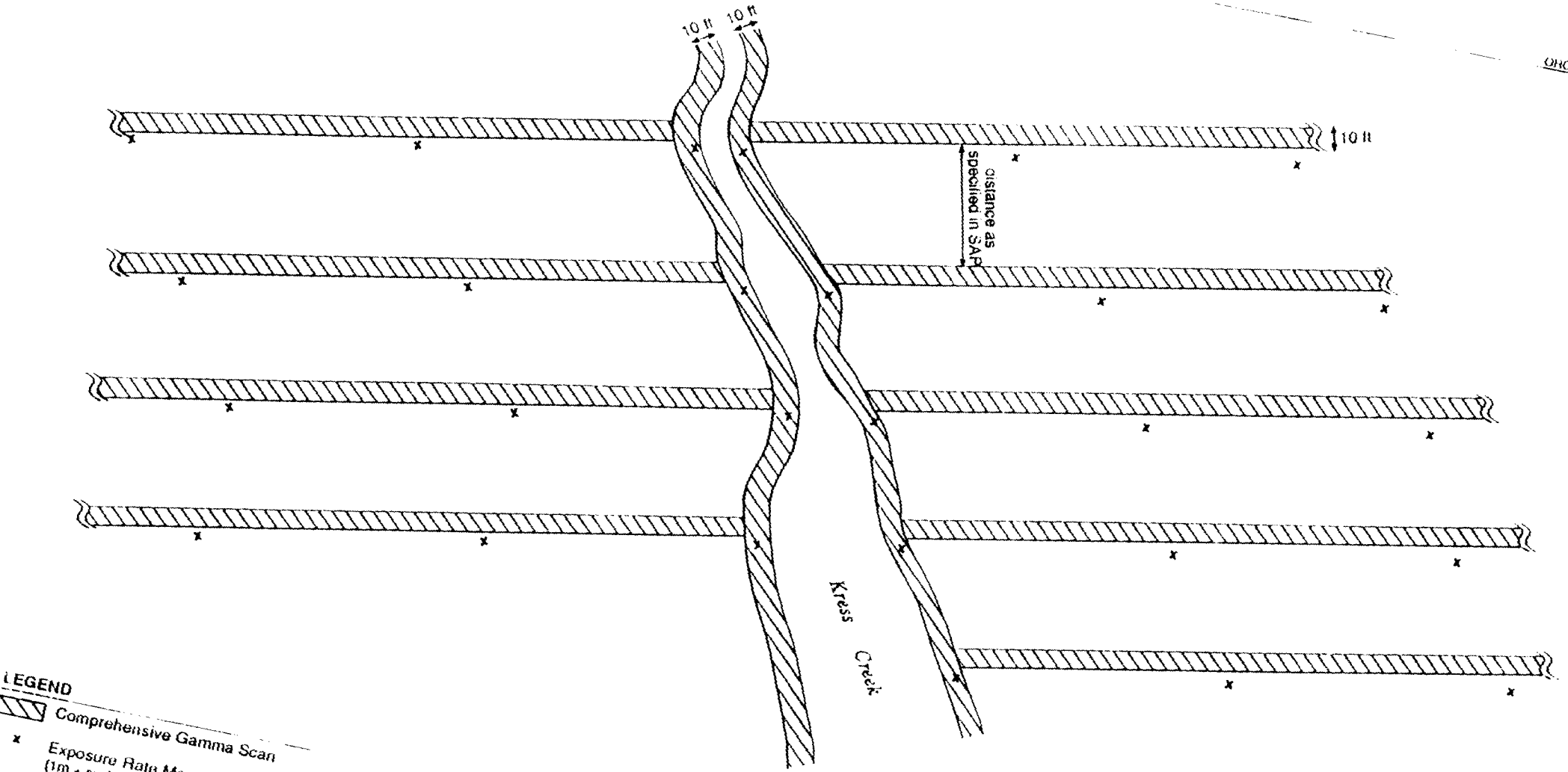


Average or Range of Levels: _____ Scale 1" = _____

	Grid Coordinates		CPM	µR/hr		Grid Coordinates		CPM	µR/hr
	x	y				x	y		
1.									
2.									
3.									
4.									
5.									
6.									
7.									
8.									
9.									

_____ _____

Surveyed by *Reviewed by*



Example Section for Radiological S

Figure 1

Title: Environmental Gamma-Ray Exposure Rate Measurements for Creeks and Streams

Document No.: SOP: RP-30

Revision No.: 1

Date: 1/11/93

Approved by: George Stevens
Radiation Health Manager

Date: 1/11/93

RP-30
Environmental Gamma-Ray Exposure Rate Measurements for Streams and Rivers

1.0 PURPOSE

The purpose of this procedure is to describe the methodology for measuring environmental gamma-ray exposure rates along creeks and streams.

2.0 SCOPE

This procedure includes a description of the techniques and the instruments used to determine environmental gamma-ray exposure rates and to document the resulting radiological survey data. The procedure does not address the measurement of worker radiation dose rates.

3.0 REFERENCES

3.1 Manual for Conducting Radiological Surveys in Support of License Termination. NUREG/CR-5849, ORAU-92/C57

3.2 Oak Ridge Institute for Science and Education (ORISE) Environmental Survey and Site Assessment Program Survey Procedures Manual

4.0 EQUIPMENT AND MATERIAL

4.1 Maps and drawings

4.2 Grid markers and measuring devices (e.g., pin flags and 100-ft tape measure)

4.3 Form for Gamma-Ray Exposure Rate Survey

4.4 Portable ratemeter/scaler (Eberline ESP-2 or equivalent)

4.5 Gamma Scintillation Detector (NaI—Eberline SPA-3 or equivalent)

4.6 Clipboard and pen

4.7 Calculator

5.0 PROCEDURES/INSTRUCTIONS

5.1 Delineation of Area

5.1.1 Transects will be systematically established at points along the streams. The transects are survey areas perpendicular to and extending outward from the waterline to an end point. The width of transects will be specified in the Field

Sampling Plan (FSP) (normally range from 10 to 20 ft). The transect will extend from each creek bank outward to an established end point, as specified in the FSP.

5.1.2 Gamma exposure rate measurements will be taken at 1-m and ground surface at intervals designated in the FSP along each transect as access permits.

5.2 Measurements

5.2.1 Determine the background count rate and source check the gamma scintillation detector and ratemeter/scaler using Procedure RP-23. Record the results on the CH2M HILL Weekly Field Source Check Log.

5.2.2 Move to the measurement location and set the ESP-2 for integrated counting. Verify that the detector-to-surface distance is maintained at 1 meter and that the count time is 0.5 min or more.

5.2.3 Collect integrated gamma counts at each location. Record the results in the Field Log.

5.3 Data Reduction

5.3.1 Raw count data will be converted to μR/hr by calibrating the gamma scintillation detectors to exposure rate, as described in RP-28 in CH2M HILL's Radiation Safety Manual (RSM). Final results will be reported in μR/hr equivalent.

5.4 Quality Control

5.4.1 Take a duplicate measurement for one in every ten readings obtained with each detector.

5.4.2 The locations for duplicate measurements will be chosen by a random generator if a non-biased survey is required.

5.4.3 Duplicate measurements will be taken after transect measurements have been made.

5.4.4 The duplicate counts will be recorded on the same form as the original survey measurements and should be identified as duplicate readings (D).

5.5 Documentation

5.5.1 Measurements and pertinent information will be recorded in CH2M HILL field notebooks.

Title:	**pH/Temperature Meter Calibration and Use**
Revision No.:	0
Date:	1/11/93

pH/Temperature Meter Calibration and Use

The pH/temperature meters used in the field are Orion Model SA 250 portable meters, which measure both pH and temperature (degrees C). The meters will be calibrated in the field by each sampling team prior to being used. The meter must be recalibrated only if:

- the meter is shut off before finishing the run, or
- the measured pH is below 7.

Otherwise, as long as the meter is left on during the sampling event, it does not have to be recalibrated unless it displays unusual readings.

1.1 CALIBRATION DETAILS

1. Connect the electrodes to the meter. Slide mode to pH.01. Choose two buffers that will bracket the expected sample range. Use pH 7 and pH 10 initially. If the sample has a pH below 7, recalibrate using the same method with pH 4 and pH 7 buffers.

2. Place both the pH and temperature electrodes into the first buffer (either one).

3. Press cal. The display will alternate between 1 and the pH value of the buffer. This indicates that the first buffer value has not yet been entered.

4. To enter the first buffer value, wait for a stable pH display, and use the up/down keys to scroll to the correct pH buffer value. Press enter. (Even if scrolling is not necessary, scroll one digit and return to correct pH buffer value before pressing enter.)

5. Rinse electrodes and place into second buffer. Wait for stable pH display. Scroll to the correct buffer pH value. Press enter.

6. The letters "pH" will be displayed. Meter is now ready for testing.

1.2 USE AND MAINTENANCE

Place both the electrodes in the sample when testing and leave the meter on throughout the sampling run. Recalibrate only if a pH value less than 7 is obtained or if the meter is accidently shut off.

The pH and temperature electrode should be rinsed with deionized water after each immersion into a sample. Also, the meter should be protected from moisture and placed in the carrying case between tests.

Title:	**Dissolved Oxygen Meter Calibration and Use**
Revision No.:	0
Date:	1/8/93

DISSOLVED OXYGEN METER CALIBRATION AND USE

1.1 MODEL

YSI 51B Dissolved Oxygen Meter

1.2 RANGE

0 to 15 mg/L – dissolved oxygen
-5 to +45 degrees Celsius – temperature

1.3 CALIBRATION

The dissolved oxygen meter is automatically temperature-compensated for permeability of the probe membrane and is adjusted manually by direct dial from fresh water to sea water of 20,000 mg/L chloride concentration. (Salinity values are obtained from a conductivity meter.)

The dissolved oxygen meters are calibrated at the field vehicle shortly before use. The Winkler titration method as specified in Standard Methods for the Examination of Water and Waste Water, 14th edition (1985), is used. The meter should not be shut off during the sampling event.

1.4 USE AND MAINTENANCE

Samples should be stirred gently with the probe during the measurement to achieve accurate readings. The probe must be rinsed fully with deionized water after every sample and capped so that the electrode will remain moist. The probe membranes should be replaced every 2 to 4 weeks. Membrane replacement is indicated when erratic readings are observed or calibration is not stable.

Title: **Conductivity/Salinity Meter Calibration and Use**

Revision No.: 0

Date: 1/8/93

CONDUCTIVITY/SALINITY METER CALIBRATION AND USE

1.1 MODEL

Hanna Instruments Model HI8033

1.2 RANGE

0 to 1,999 micromhos (on three scales)-conductivity
0 to 19,990 ppm chloride-salinity

1.3 CALIBRATION

The meter measures electrical conductivity in liquids as a function of temperature. The meter automatically compensates for temperature of the sample when the temperature is set before testing.

Calibrate the meter in the field in the conductivity mode with appropriate standard calibration solution and Temperature Versus Conductivity Calibration Chart (Table 1), as follows:

1. Place a pH meter temperature electrode and conductivity meter probe in a glass jar containing calibration solution. Make sure the four holes on the conductivity meter electrode are submerged and no air bubbles are trapped (tap a few times on the bottom and stir).

2. Set the temperature of the solution on the conductivity meter by turning the small knob in the lower left corner of the meter.

3. Find the corresponding conductivity value from the Temperature Versus Conductivity Calibration Chart (Table 1).

4. Turn knob in lower right corner of the meter until the value from the chart is obtained.

The instrument is now ready for field testing.

1.4 USE AND MAINTENANCE

For each conductivity measurement, the probe should be immersed in the sample such that the four holes in the electrode are submerged. Conductivity should be measured and recorded first. Before reading conductivity, the temperature setting on the conductivity/salinity meter should be set to the sample temperature as measured with the pH/temperature meter. The correct conductivity scale is then selected and the measurement read.

Let the meter reading stabilize before recording in the field logbook. The mode dial is then turned to the salinity mode, if needed. The salinity mode should be calibrated to read in parts per million chloride (X1000). The salinity reading is then recorded in the field logbook.

The meter should be left on throughout the sampling run. The meter must stay dry to function properly. After every test, the electrode should be rinsed with deionized water. The meter operates under temperatures from 0 to 50°C and up to 95 percent humidity. The meter should be turned off upon completion of the sampling run.

Table 1
Temperature vs. Conductivity Calibration Chart (1,413 μmhos)

For use in conductivity meter calibration at Combined Sewer Locations and Receiving Water Locations. Find Conductivity (μmhos) corresponding to sample temperature (°C).

Temperature (°C)	Conductivity (μmhos)
10	1,020
11	1,045
12	1,071
13	1,096
14	1,122
15	1,147
16	1,173
17	1,199
18	1,225
19	1,251
20	1,278
21	1,305
22	1,332
23	1,359
24	1,386
25	1,413
26	1,440
27	1,467
28	1,494

Temperature vs. Conductivity Calibration Chart (12,880 μmhos)

For use in conductivity meter calibration at Receiving Water Locations. Find Conductivity (μmhos) corresponding to sample temperature (°C).

Temperature (°C)	Conductivity (μmhos)
10	9,330
11	9,560
12	9,790
13	10,020
14	10,250
15	10,480
16	10,720
17	10,950
18	11,190
19	11,430
20	11,670
21	11,910
22	12,150
23	12,390
24	12,640
25	12,880
26	13,130
27	13,370
28	13,620

Title: **Total Hardness Titrator Calibration and Use**

Revision No.: 0

Date: 1/8/93

TOTAL HARDNESS TITRATOR USE

1.1 MODEL

Hach HA-DT

1.2 RANGE

10-4,000 mg/L

1.3 SENSITIVITY

0.1-10 mg/L

1.4 USE

1. Select the sample volume and EDTA titration cartridge corresponding to the expected calcium carbonate ($CaCO_3$) concentration. Use Table 1 for concentrations in mg/L.

2. Insert a clean delivery tube into the titration cartridge. Attach the cartridge to the titrator body.

3. Hold the digital titrator with the cartridge tip pointing up. Turn the delivery knob to eject air and a few drops of titrant. Reset the counter to zero and wipe the tip.

4. Use a graduate cylinder or pipe to measure the sample volume from Table 1. Transfer the sample into a 250-ml Erlenmeyer flask. Add deionized water to about the 100-ml mark, if necessary.

5. Add 2 ml of potassium hydroxide standard solution (8 N) and swirl to mix.

6. Add contents of one Calver 2 Calcium Indicator Powder Pillow and swirl to mix.

7. Place the delivery tube tip into the solution and swirl the flask while titrating with EDTA from pink to blue. Record the number of digits required.

8. Calculate the sample concentration using the formula below:

   digits required $\times$ digit multiplier = mg/L as $CaCO_3$

9. Record the concentration in the field logbook.

10. A method blank will be analyzed at the beginning of each day. The method blank will consist of 100 mL of deionized water being analyzed as if it contained 10 to 40 mg/L $CaCO_3$. The method blank concentration shall be recorded and used to adjust field sample results if necessary.

11. A replicate field sample will be analyzed once each day. The replicate precision results will be used as a means of assessing field and analytical precision. These results will not be used to qualify or disallow the use of any field sample results. The replicate precision control limits should be -50% and +100%. If precision results are outside these limits, the analyst shall review the analytical procedure to make sure it is being performed correctly, mix the sample thoroughly, and reanalyze the replicate. If the precision results

fall outside the control limits a second time, a note will be added to the field logbook stating that precision requirements for this procedure were not met for this day.

Table 1 Titration Calibration			
Range (mg/L $CaCO_3$)	**Sample Volume (mL)**	**Titration Cartridge (EDTA)**	**Digit Multiplier**
10-40	100	0.0800	0.1
40-160	25	0.0800	0.4
100-400	100	0.800	1.0
200-800	50	0.800	2.0
500-2,000	20	0.800	5.0
1,000-4.000	10	0.800	10.0

Title:	**Particle Size Fractioning**
Revision No.:	0
Date:	2/4/93

Field Procedure for Particle Size Fractioning

Scope

This procedure covers the field analysis of sediment samples for particle size fractioning.

Apparatus

- Sieves. A series of sieves, of square mesh woven wire cloth. The following sieves will be utilized in the field:
  - No. 10
  - No. 100
- Plastic 250-mL graduated cylinder
- Stainless-steel bowl

Sampling

1. Collect approximately 1 L of sediment and place in stainless-steel bowl if only particle size fractioning is to be conducted. If contaminant analysis is to occur after fractioning, collect approximately 4,000 g of sediment.

2. Mix the sediment and decant off extra water. Weigh the sample. For samples requiring contaminant analysis, select representative samples to be sent for total fraction Target Analyte List (TAL) metal and radionuclide analysis, and package appropriately.

3. Transfer the contents of the bowl onto the stacked sieves. The sieves should be stacked in a fashion that the largest sieve size (No. 10) is on top.

4. Using the 250-mL graduate cylinder, gently pour river water through the stack, wetting the entire sample until it appears as though all material capable of passing the No. 10 sieve has done so.

5. Repeat Step No. 4 for the No. 100 sieve.

6. After the sample has been distributed by size, record the percent of material passing each sieve by transferring the material in each sieve onto the scale and record its wet weight. For preidentified samples, each sediment fraction is then transferred to the laboratory sample bottles for additional offsite analyses.

Calculations

1. Calculate the percentage retained in the No. 10 sieve by dividing the weight retained on the No. 4 sieve by the total sediment weight, and multiplying the result by 100.

2. To determine the percent of sediment retained on the No. 100 sieve but passing the No. 10 sieve, divide the weight retained by the total weight of sediment.

3. To determine the percent of sediment passing the No. 100 sieve, divide the weight passing by the total sediment weight, and multiplying by 100.

Raw Data

The raw data should include the following:

1. Weight of sample put into top sieve (total).
2. Weight of sediment retained on the No. 10 sieve.
3. Percent of sediment retained by the No. 10 sieve.
4. Weight of sediment retained on the No. 100 sieve, but passing the No. 10 sieve.
5. Percent of sediment retained by the No. 100 sieve.
6. Weight of sediment passing the No. 100 sieve.
7. Percent of sediment passing by the No. 100 sieve.

Appendix C
Special Analytical Services Requests

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818
Alexandria, Virginia 22313
Phone: (703) 557-2490

SAS Number
[ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. **EPA Region and Site Name:** Region V/Kerr-McGee, Kress Creek Site

B. **Regional Representative:** Pat Churilla

C. **Telephone Number:** (312) 353-5210

D. **Date of Request:** January 29, 1993

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General description of analytical service requested:**

   Analysis of total suspended solids (TSS) in surface water samples. Results are reported as mg/L suspended solids.

   Due to the possibility of the samples having low levels of thorium (less than 11,000 pCi/g) and radium (less than 1,000 pCi/g), a laboratory that is set up to deal with wastes of this nature will need to be procured.

2. **Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentrations):**

   Twenty-four surface water samples will be collected for analysis during the sampling event. The aqueous samples will contain low to medium concentrations of TSS.

3. **Purpose of analysis [specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.]:**

   Superfund Remedial

4. **Estimated date(s) of collection:**

   May through July 1993

5. **Estimated date(s) and method of shipment:**

Method of shipment will be daily shipments by overnight carrier.

6. **Approximate number of days results required after lab receipt of samples:**

The laboratory will be required to provide results within 35 days of receipt of the samples.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

EPA Method 160.2, (Gravimetric, Dried 103-105°C) using glass fiber filter discs without organic binder. Use only a membrane filter apparatus with a 47-mm glass fiber filter and a coarse (40 to 60 micron) fritted disc filter support.

Samples will be kept at 4°C until sample analysis and validation of results.

Holding time is 7 days from date of sample collection.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

Sample aliquot volumes are selected on the basis of the following factors:

a) During initial sample filtration, filtration rate should not drop rapidly or require more than 5 min of filtration time. Increase the filter area or decrease the sample volume as required for reanalysis.

b) The sample aliquot filtered should provide a residue with greater than 1.0 mg for aliquots less than 200 ml in volume.

c) Sample aliquots should not exceed 200 ml in volume.

Duplicate sample aliquots should be filtered with two or more intervening samples.

Final residues are to be dried to constant weight, and the final weight is to be used for all calculations.

Constant weight is defined as:

a) Less than 0.5 mg or 4 percent weight loss from the previous weight, whichever is smaller.

b) Dried overnight (12 hr) with the single final weight used for the calculations.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, chain-of-custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Bench records of tare weights, final weights, additional weights, volumes filtered, blanks, and duplicate samples will be provided along with copies of worksheets used to calculate results.

Dates and times of determination of tare weights, sample filtration, and determination of residue weights will be part of bench records.

All records of analysis must be legible and sufficient to recalculate all sample concentrations and QA results.

10. **Other (use additional sheets or attach supplementary information, as needed):**

None.

11. **Name of sampling/shipping contact:**

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (± % or conc.)
TSS	2 to 3 mg/L for 200 mL	Difference in duplicate sample aliquots shall not exceed 0.5 mg for residues less than 5 mg. Duplicate differences shall not exceed 10% for sample values greater than 5 mg.
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

II. Quality Control Requirements

Audits Required	Frequency of Audits	Limits* (± % or conc)
Lab Duplicate	At least 1 per group of 10 or fewer samples	±10% for sample residue >5 mg and 0.5 mg residue for samples less than 5 mg
Lab Blanks (200 mL of reagent water)	At least 1 per group of 10 or fewer samples	-0.5 mg/L to +0.5 mg/L
*See Section III.		

III. Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contact Dennis Wesolowski at (312) 886-1970 for discussion of problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818
Alexandria, Virginia 22313
Phone: (703) 557-2490

SAS Number
[ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. **EPA Region and Site Name:** Region V/Kerr-McGee, Kress Creek Site

B. **Regional Representative:** Pat Churilla

C. **Telephone Number:** (312) 353-5210

D. **Date of Request:** January 29, 1993

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General description of analytical service requested:**

   Analysis of total dissolved solids (TDS) in surface water samples. Results are reported as mg/L dissolved solids.

   Due to the possibility of the samples having low levels of thorium (less than 11,000 pCi/g) and radium (less than 1,000 pCi/g), a laboratory that is set up to deal with wastes of this nature will need to be procured.

2. **Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentrations):**

   Twenty-four surface water samples will be collected for analysis during the sampling event. The aqueous samples will contain medium to high concentrations of TDS.

3. **Purpose of analysis [specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.]:**

   Superfund Remedial

4. **Estimated date(s) of collection:**

   May through July 1993

5. **Estimated date(s) and method of shipment:**

Method of shipment will be daily shipments by overnight carrier.

6. **Approximate number of days results required after lab receipt of samples:**

The laboratory will be required to provide results within 35 days of receipt of the samples.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

EPA Method 160.1, Filterable Residue, or "Standard Methods" Method 209B.

Samples will be kept at 4°C until sample analysis and validation of results.

Holding time is 7 days from date of sample collection.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

Use standard aliquots of 100 mL; however, do not use aliquots yielding more than 200 mg residue. If residue is greater than 200 mg, repeat the analysis using a smaller aliquot.

If the pH value is less than 4.0, raise the pH of the aliquot, with NaOH titrant, to between a pH of 4 and 8, and subtract the weight of sodium added from the weight of the residue.

Residue will be weighted to constant weight pursuant to Section 7.6 of Method 160.1 (weight loss is less than 0.5 mg or less than 4% weight loss from previous loss). Constant weights will also be obtainable on a single weight basis if the sample is dried for a minimum of 12 hr. The final weight is to be used for calculations.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, chain-of-custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Bench records of tare weights, final weights, additional weights to determine constant weights, volumes filtered, blanks, and duplicate samples will be provided with copies of work sheets used to calculate results. Dates and times when the following tasks are performed will be recorded as part of the bench record:

- Determination of tare weights
- Sample filtration
- Determination of constant weights
- Determination of residue weights

10. **Other (use additional sheets or attach supplementary information, as needed):**

None.

11. **Name of sampling/shipping contact:**

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Parameter	Detection Limit	Precision Desired (± % or conc.)
TDS	20 mg/L	Difference in duplicate sample aliquots shall not exceed 5 mg for residues less than 50 mg/L. Duplicate differences shall not exceed 10% for sample values greater than 5 mg/L.

II. Quality Control Requirements

Audits Required	Frequency of Audits	Limits* (± % or conc)
Lab Duplicate	At least 1 per group of 10 or fewer samples	± (10% or 5 mg residue)
Lab Blanks (100 mL of filtered reagent water)	At least 1 per group of 10 or fewer samples	-20 mg/L to +20 mg/L
*See Section III.		

III. Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contact Dennis Wesolowski at (312) 886-1970 for discussion of problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818
Alexandria, Virginia 22313
Phone: (703) 557-2490

SAS Number
[ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. **EPA Region and Site Name:** Region V/Kerr-McGee, Kress Creek Site

B. **Regional Representative:** Pat Churilla

C. **Telephone Number:** (312) 353-5210

D. **Date of Request:** January 29, 1993

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General description of analytical service requested:**

The following parameters require analysis by conventional SOWs (SOW/OLM01.8 for organics and SOW/ILM02.1 for inorganics). But, due to the possibility of the samples having low levels of thorium (less than 11,000 pCi/g) and radium (less than 1,000 pCi/g), a laboratory that is set up to deal with wastes of this nature will need to be procured.

TCL organic analysis (volatile, semivolatile, PCBs, and pesticides)
TAL metals analysis

2. **Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentrations):**

Sediment samples; eight low concentration sediment samples will be analyzed for TCL organics, and 85 sediment samples will be analyzed for TAL metals.

Soil samples; 55 floodplain soil samples will be analyzed for TAL metals.

Surface water samples; 30 surface water samples will be analyzed for TAL metals.

3. **Purpose of analysis [specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.]:**

Superfund Remedial

4. **Estimated date(s) of collection:**

May through July 1993

5. **Estimated date(s) and method of shipment:**

Method of shipment will be daily shipments by overnight carrier.

6. **Approximate number of days results required after lab receipt of samples:**

The laboratory will be required to provide results within 35 days of receipt of the samples.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

The SOW/OLM01.8 for organics and SOW/ILM02.1 for inorganics are to be followed for these analyses.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

None.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, *chain-of-custody documentation, etc.). If not completed, format of results will be left* to program discretion.**

As specified in the Statements of Work.

10. **Other (use additional sheets or attach supplementary information, as needed):**

None.

11. **Name of sampling/shipping contact:**

David Shekoski Phone: (414) 272-2426

I. Data Requirements

As required by appropriate Statements of Work.

II. Quality Control Requirements

As required by appropriate Statements of Work.

III. Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contact Dennis Wesolowski at (312) 886-1970 for discussion of problems that might result in the delay of reporting sample results.

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818
Alexandria, Virginia 22313
Phone: (703) 557-2490

SAS Number
[ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. **EPA Region and Site Name:** Region V/Kerr-McGee, Kress Creek Site

B. **Regional Representative:** Pat Churilla

C. **Telephone Number:** (312) 353-5210

D. **Date of Request:** January 29, 1993

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General description of analytical service requested:**

   Analysis of fish samples, whole fish minus internal organs, for total metal analysis.

   The method of choice for sample preparation is described in Attachment I; methods of choice for sample digestion are described in Attachment II and provided in Attachments III through VI. The sample digestates will be analyzed for the TAL metals, as described in the SOW/ILM01.0.

   Due to the possibility of the samples being radiologically contaminated, a laboratory that is set up to deal with wastes of this nature will need to be procured.

2. **Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentrations):**

   Eighteen whole fish samples will be collected during the sampling event.

3. **Purpose of analysis [specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.]:**

   Superfund Remedial

4. **Estimated date(s) of collection:**

January 1993 and again May through July 1993

5. **Estimated date(s) and method of shipment:**

Method of shipment will be daily shipments by overnight carrier.

6. **Approximate number of days results required after lab receipt of samples:**

The laboratory will be required to provide results within 35 days of receipt of the samples.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

The choice of fish digestion techniques are specified in Attachment II. More than one digestion procedure may be required for different metals. The sample digestates will be quantified according to SOW/ILM01.0.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

All metals will be analyzed using one of the attached digestion methods and quantified according to procedures described in the SOW/ILM01.0.

The laboratory must document in the case narrative any and all choices made from the options allowed within this SAS.

The ICP analyses shall use standards that are matrix matched to the final fish matrix after preparation, or use internal standardization.

Mercury spikes shall use methyl mercuric chloride for fortifications.

If Attachment III is used for mercury sample digestion, the same amount of potassium permanganate shall be added to the blank as is added to the sample that required the most permanganate.

If a lab control sample other than a NIST SRM® is used, a copy of the certification shall be provided with the narrative.

The laboratory shall perform a percent moisture analysis on 5 to 10 g of prepared sample, as per the SOW, <u>except</u> the sample shall be heated to 50°C, not 103 to 105°C. Do not correct results for percent moisture; report percent moisture on the Form I and note in the case narrative that results are not corrected for percent moisture.

Each analytical determination must have the resulting absorbance clearly recorded and documented in their order of determination.

The calibration range of the GFAA analyses cannot be exceeded. Dilute any sample that does exceed the calibration range.

Any sample remaining after digestion should be stored at 4°C until the validation and the acceptance of the sample result.

The IDL must be shown to have been met prior to the analysis of any samples. The lab can accomplish this by submitting their most recent Form XI with each case.

The sample solutions analyzed must have their matrix concentration fully documented in the raw data.

Spike levels should correspond to the following concentrations in the original fish tissue:

Element	Concentration (µg/g)
Al	50
Sb	50
As	2
Ba	5
Be	1
Ca	100
Cd	1
Cr	1
Co	5
Cu	10
Fe	100
Pb	2
Mn	5
Mg	100
Hg	0.5
Ni	10
K	100
Na	100
Se	1
Ag	2
Tl	1
V	5
Zn	100

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, chain-of-custody documentation, etc.). If not completed, format of results will be left to program discretion.**

The required deliverables shall be equivalent to those provided with the SOW ILM01.0.

Current quarterly Form XI, XII, and XIII for each element.

Form VIII must be modified to include the slope of each addition, as well as the correlation coefficient.

10. **Other (use additional sheets or attach supplementary information, as needed):**

None.

11. **Name of sampling/shipping contact:**

David Shekoski Phone: (414) 272-2426

I. Data Requirements

As required by the appropriate Statements of Work.

II. Quality Control Requirements

Audits Required	Frequency of Audits	Limits* (± % or conc)
Preparation Blank	At least 1 per group of 10 or fewer samples	≤IDL
Lab Duplicate	At least 1 per group of 10 or fewer samples	±25% or RPD is ≤IDL
Calibration Blank	At least 1 per group of 10 or fewer samples	≤IDL
ICs and CCVs	As per SOW/ILM01.0	As per SOW/ILM01.0
Matrix Spike	At least 1 per group of 10 or fewer samples	75 to 125% for fish samples
Lab Control Spikes	1 per group of 10 or fewer samples	75 to 125% for fish samples
*See Section III.		

III. Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contact Pat Churilla at (312) 353-5210 for problems that might result in the delay of reporting sample results.

Attachment I
Procedure for Preparing Fish for Metals Analysis

NOTE: This procedure must be carried out in a clean environment, as free from dust as possible. Use of a laminar-flow hood for the sample grinding and homogenization is recommended, but not required. If one is used, it should be documented in the case narrative.

A. The fish will be collected and placed in contaminant-free polyethylene bags. Each bag will immediately be placed in a cooler of dry ice. The samples will be packed with sufficient dry ice to keep the samples frozen until receipt by the laboratory.

B. Proceed with the sample preparation as follows:

1. Remove the frozen fish from the bag. Place the frozen fish on a clean, polyethylene or other inert, nonmetallic surface. Chop the fish into 2 to 3-in. cubes using a plastic, titanium, or Teflon-coated cleaver. Using a blender or meat grinder with either plastic, titanium, or Teflon-coated components, grind the fish cubes together with enough dry ice to keep them frozen. If only a stainless-steel cleaver and/or grinding components are available, the stainless-steel cleaver and/or components shall be acid-rinsed. The use of stainless steel equipment shall be reported to the SMO with the bid and documented in the case narrative.

2. Regrind the ground fish enough times to achieve a homogeneous paste.

3. Mix the ground fish thoroughly with a clean, Teflon-coated spatula and store in two aliquots as follows. Place a portion, about 10 to 20 g, to be used for mercury analysis, in a new certified-clean jar and freeze immediately. Store the remaining portion of the sample in the original jar. Take care not to fill the jars completely, thus avoiding breakage during storage in the freezer. Do not thaw the sample or open the container until ready to digest the sample. Store the samples in the freezer until needed.

Attachment II

A choice of methods for the fish sample digestion is offered that may be used for the analyses of the metals of interest. The laboratory may choose a digestion method for a given metal from the following table:

Digestion Procedures for Fish Samples			
Method	**Description**	**TAL Metals (minus Hg)**	**Mercury**
Attachment III	Hg in fish, digestion using sulfuric and nitric acid at 58°C. Followed by potassium permanganate oxidation.		X
Attachment IV	Estuarine and marine tissue digestion using a wet oxidation technique.	X	X
Attachment V	Microwave digestion of fish tissues.	X	
Attachment VI	Aluminum block digestion of fish tissue.	X	

Attachment III

Analysis of Fish for Mercury

Scope and Application

This method is used for determination of total mercury (organic and inorganic) in fish. Digest a weighed portion of the sample with sulfuric and nitric acid at 58°C. Follow by overnight oxidation with potassium permanganate at room temperature. Mercury is subsequently measured by the conventional cold vapor technique.

The range of the method is 0.2 to 5 $\mu g/g$, but may be extended above or below the normal instrument and recorder control.

Sample Preparation

The sample may be prepared as described under "Sample Handling," or the special metal procedure may be used. A 0.2- to 0.3-g portion should be taken for each analysis. The sample should not be allowed to thaw before weighing.

Preparation of Calibration Curve

Conduct according to SOW/ILM02.1.

Sample Procedure

Weigh 0.2- to 0.3-g portions of the sample and place in the bottom of a dry BOD bottle. Care must be taken that none of the sample adheres to the side of the bottle. Add 4 mL of concentrated H_2SO_4 and 1 mL of concentrated HNO_3 to each bottle and place in a water bath maintained at 58°C until the tissue is completely dissolved (30 to 60 min).

Cool to 4°C in an ice bath and cautiously add 5 mL of potassium permanganate solution in 1-mL increments. Add an additional 10 mL or more of permanganate as necessary to maintain oxidizing conditions. Allow to stand overnight at room temperature. Add enough distilled water to bring the total volume to approximately 125 mL. Add 6 mL of sodium chloride-hydroxylamine sulfate solution to reduce the excess permanganate.

As an alternative to the overnight digestion, the solubilization of the tissue may be carried out in a water bath at 80°C for 30 min. Cool the sample and cautiously add 15 mL of potassium permanganate solution. Return the sample to the water bath and digest for an additional 90 min at 30°C. If this method is followed, the calibration standards must also be treated in this manner.

Calculation

Measure the peak height of the unknown from the chart and read the mercury value from the standard curve.

Calculate the mercury concentration in the sample by using the following formula:

$$\mu g\ Hg/g = \frac{\mu g\ Hg\ (aliquot)}{weight\ of\ aliquot\ (grams)}$$

Report mercury concentrations as follows:

Below 0.1 μg/g, <0.1 μg; between 0.1 and 1 μg/g, to nearest 0.01 μg; between 1 and 10 μg/g, to nearest 0.1 μg; above 10 μg/g, to nearest μg.

Quality Assurance

Standard quality assurance protocols should be employed, including blanks, duplicates, and spiked samples, as described in the *Analytical Quality Control Handbook*. Spikes and duplicates will be at the frequency/levels specified in the CLP Inorganic SOW 785. Control spikes for daily analysis will be the same level as matrix spikes.

Report all quality control data when reporting results of sample analyses.

Mercury spiking solution will be an organo-mercury compound (e.g., methyl mercuric chloride) rather than inorganic mercury.

Precision and Accuracy

The following standard deviations on replicate fish samples were recorded at the indicated levels: 0.19 μg/g $\pm$ 0.02, 0.74 μg/g $\pm$ 0.05, and 2.1 μg/g $\pm$ 0.06. The coefficients of variation at these levels were 11.9%, 7.0%, and 3.6%, respectively. Recovery of mercury at these levels, added as methyl mercuric chloride, was 112%, 93%, and 86%, respectively.

Attachment IV

ANALYSIS OF METALS AND METALLOIDS IN ESTUARINE AND MARINE TISSUES

1.0 SCOPE AND APPLICATION

1.1 This method is designed to determine antimony, arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, thallium, and zinc in biological samples. The method may be adapted for the analysis of varying tissue types such as edible muscle and livers of estuarine and marine organisms.

1.2 A universal wet ashing procedure (acid digestion) is proposed that is capable of providing a clean extract suitable for analysis by atomic absorption spectrophotometry (AAS). This digestion has proven effective when determining most of the priority pollutant metals listed above. Due to a lack of reference materials certified for beryllium and thallium, little is known regarding method suitability for these elements. Additional development work is therefore recommended for these two elements.

1.3 Assuming that sample size is not restricted, limits of quantitation (LOQ) are typically in the range of 0.01 micrograms element per wet gram of tissue (Table 1). These may vary depending on element, method of detection, and instrument sensitivity.

2.0 SUMMARY OF METHOD

2.1 A representative sample of tissue is homogenized wet, subsampled and digested using a wet oxidation method. The resulting extract is analyzed for the metals of interest using various atomic absorption (AA) techniques such as:

- direct aspiration (DFAA) = for higher concentration metals
- graphite furnace (GFAA) = for lower concentration metals
- hydride generation (HYDAA) = for hydride forming elements (antimony, arsenic, selenium)
- cold vapor (CVAA) = for mercury.

2.2 Alternative methods of detection may be used providing their performance and limitations have been established.

3.0 DEFINITIONS

Certified Reference Materials (CRM): A homogeneous sample that has been analyzed a sufficient number of times by numerous qualified laboratories. The data are compiled and certified values are determined through statistical analysis. A number of CRMs are commercially available in a wide range of matricies for metals anlyses.

Control Standard: A solution, independent of the calibration standards whose analyte concentration is known. These are often analyzed as an external check after calibration.

Limit of Detection (LOD): The LOD is the lowest concentration level that can be determined to be statistically different from a blank.

Limit of Quantitation (LOQ): The LOQ is the level above which quantitative results may be obtained with a specified degree of confidence.

Matrix Modifier: A reagent added to a sample that alters some form of its composition.

4.0 INTERFERENCES

4.1 Interferences should be considered to be any chemical or physical phenomenon that can influence the accuracy of the data during an analytical

operation. These can have either a positive or a negative effect on the result depending on their nature.

4.2 Contamination of the sample can occur during any stage of the collection, handling, storage, or analysis procedures. Potential contaminant sources must be known and steps taken to minimize or eliminate these. Some of the most common sources of contamination include: prolonged exposure of the tissue to metal containing fumes and dust, insufficiently clean sample containers, storage facilities and testing apparatus as well as the use of contaminated reagents during analysis.

In general, clean laboratory procedures are extremely important when performing trace metal analysis.

4.3 Most instrumental methods are prone to matrix interferences, which can either suppress or enhance the analyte signal. If a matrix interference is suspected, its effect should be determined and corrective action taken. Some common matrix interferences are listed below along with suggested corrective measures.

4.3.1 High sample viscosity - usually due to dissolved solids and high acid content - match the matrix of the calibration standards with the samples where possible.

4.3.2 Non-specific absorption (light scatter) - usually due to dissolved solids or suspended particulates, which absorb analyte radiation. Background correction (see instrument manufacturer's instructions) should be used whenever this occurs.

4.4 Many chemical interferences, some of which are poorly understood, can occur during instrumental analysis of the sample extracts. A great many of these interferences have been addressed in the literature and in most cases a sample pretreatment or instrumental modification has been proposed as a remedy.

5.0 SAFETY

Laboratory personnel should be well versed in standard laboratory safety practices. It is the responsibility of all staff and management to ensure that safety training is mandatory and that the laboratory operates in a manner consistent with current OSHA regulations.

5.1 Chemicals and reagents should be properly labelled and stored in an area appropriate to their properties. Any reagents whose composition or properties may change with time must be dated and properly disposed of on or before the expiration date.

5.2 Areas where strong oxidizing agents and flammable or explosive materials are used should be well labelled and the necessary restrictions imposed.

5.3 Where laboratory apparatus and instrumentation are used, the manufacturer's safety precautions should be strictly followed.

6.0 APPARATUS AND EQUIPMENT

6.1 Sample containers - wide-mouth screw cap jars made of either glass or non-contaminating plastic (linear or high density polyethylene, or equivalent). All containers should be pre-rinsed with dilute acid and distilled deionized water (DDW) as described in Section 10.6.

6.2 Dissection tools - scalpels should be made of high-quality, corrosion-resistant stainless steel, while tweezers and cutting surfaces should be plastic or teflon. All tools should be thoroughly rinsed with DDW prior to use and between samples.

6.3 Tissue grinder/homogenizer - a standard tissue homogenizer can be used with minor modifications. If the apparatus contains stainless steel parts, they should be replaced with tantalum. Stainless steel blades used during homogenization have been found to be a source of nickel and chromium contamination.

6.4 Digestion vessels - 125 mL borosilicate glass Erlenmeyer flasks equipped with all glass reflux caps (Tuttle covers). Tuttle covers or equivalent reflux caps are essential for preventing evaporative loss of volatile compounds or elements during high temperature digestion. They are commercially available (Fisher Scientific) or are easily produced from borosilicate test tubes.

6.5 Hot plate - a thermostatically controlled plate with a range of 75 to 400° C.

6.6 Fumehood - a properly constructed hood capable of withstanding acid fumes. It must be equipped with an exhaust fan having sufficient capacity to remove all fumes.

6.7 Atomic absorption spectrophotometer (AAS).

6.7.1 The AAS must have sufficient sensitivity and stability to perform within the specifications required by the method (Section 11). The instrument should have automatic background correction, direct aspiration flame, as well as flameless capabilities. The instrument must have a routine maintenance program to ensure proper performance and trouble-free operation. All source lamps should be handled with care and the exit windows kept free of dust and fingerprints. Periodic intensity and stability checks of the lamps should be made. Replace any lamps showing signs of deterioration.

6.7.2 A graphite furnace (also called carbon rod) attachment for the AAS is recommended when determining most elements in the low concentration ranges. Most, if not all, AAS manufacturers offer this equipment as an accessory. The stability and sensitivity afforded by the furnace is typically one to two orders of magnitude better than direct aspiration.

6.7.3 In addition to the graphite furnace, another flameless attachment can be used in conjunction with the AAS to determine the hydride-forming elements (arsenic, antimony, and selenium). Most such attachments may also be used to analyze for mercury using the cold vapor technique. These

methods are preferable to the graphite furnace since they vaporize the analyte from the sample matrix prior to detection.

7.0 REAGENTS AND CONSUMABLE MATERIALS

The purity of all reagents used for trace metal determinations is extremely important. Reagents should be checked for purity prior to use to confirm the absence of contamination.

7.1 Distilled Deionized Water (DDW) - a water purified by distillation (or equivalent) followed by conditioning with a mixed bed ion exchanger. Such units are commercially available and yield a water with a typical resistivity of 18 megohms/cm.

7.2 Hydrochloric Acid - concentrated (35%).

7.3 Hydroxylamine hydrochloride [20% (w/v)]: - dissolve 20 g of ACS grade $NH_2OH \cdot HCl$ in 100 mL of DDW. Store in a precleaned glass or plastic bottle - prepare weekly.

7.4 Nitric Acid - concentrated (70%).

7.5 Perchloric Acid - concentrated (70%).

7.6 Sodium borohydride, ACS grade granular or powder.

7.7 Sodium hydroxide, ACS grade - pellets or flakes.

7.8 Stannous chloride [20% (w/v)] - dissolve 20 g of ACS grade $SnCl_2$ in 20 mL of concentrated hydrochloric acid. Warm gently until solution clears, cool and bulk to 100 mL with DDW. Store in a precleaned glass or plastic bottle - prepare fresh daily.

7.9 Stock standard solutions - These standards (typically 1000 ppm) can be purchased as certified solutions or prepared from ACS grade metal salts

and pure compounds. Suitable procedures for preparing stock solutions are well documented and include the steps below.

7.9.1 Accurately weigh 1,000 mg of pure metal or metal equivalent of the salt and dissolve in a minimum amount (usually about 20 mL) of an appropriate acid. Once the reagent is dissolved, dilute the solution to 1,000 mL with DDW and store in a precleaned plastic bottle. The solution is usually stable for at least a year but must be checked periodically against an in-house control standard (Section 10).

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 The major difficulty in trace metal analyses of tissue samples is controlling contamination of the sample. In the field, sources of contamination include sampling gear, engine exhaust, dust, or ice used for cooling.

8.2 Sample dissection and any subsampling of the organisms should be carried out in a dust-free room. In most cases, this requires that organisms be transported on ice to a laboratory, rather than being dissected aboard the sampling vessel. To avoid contamination from ice, the samples should be wrapped in aluminum foil and placed in watertight plastic bags. Organisms should not be frozen prior to dissection as freezing will cause organs to rupture and contaminate muscle tissue.

8.3 Molluscs should be depurated in clean seawater for 24 h before dissection, since sediment in the digestive tract could yield false high tissue values.

8.4 After dissection, samples should be stored in suitable containers (Section 6.1) and frozen at -20° C until analysis. Although specific holding times have not been recommended by U.S. EPA, a holding time of 6 months (except for mercury samples, which should be held no longer than 28 days) would be consistent with that for water samples.

9.0 CALIBRATION AND STANDARDIZATION

9.1 Calibration standards are prepared by serial dilutions of the stock solutions. Mixed standards of more than one element may be prepared only after their compatibility has been determined. Some common mixed standards are as follows:

- Cd, Cu, Pb, Ni, and Zn
- As, Se, and Sb

9.1.1 Do not add an incompatible anion to a mixed or single element standard. For example, adding chloride to a silver standard could form a precipitate of silver chloride (AgCl).

9.1.2 Do not mix metals that are incompatible in solution. For example, lead and chromium may form a precipitate of lead chromate ($PbCrO_4$).

9.2 Concentration ranges of the standards should bracket those for the samples to be analyzed. At least four standards (one blank and three standards of increasing concentration) should be used to calibrate the instrument. The acid matrix of the standards should be as closely matched to the samples as possible.

9.3 Stability of a calibration standard varies with element, acid matrix, concentration, and presence of other elements. As a general rule, standards should be continuously monitored and replaced when necessary. As a matter of protocol, the following can be used as a guideline:

less than 0.1 ppm	-	prepare daily
0.1 to 1 ppm	-	prepare weekly
1.0 to 10 ppm	-	prepare monthly
10 to 100 ppm	-	prepare quarterly
100+ ppm	-	prepare yearly (at a minimum)

9.4 Initial standardization - follow manufacturer's suggestions for standardizing instrument and check sensitivity performance with specifications. If performance is acceptable, proceed with analysis; if not, refer to manufacturer's troubleshooting guide.

9.5 After standardizing the instrument, analyze an independent control standard as a check. If the result is acceptable, proceed; otherwise, troubleshoot calibration standards, control standard, or instrument.

9.6 Once the standardization is acceptable, samples may be analyzed, however, periodic calibration checks must be performed. Analyze a standard solution every tenth sample or every two hours during an analysis run, whichever is more frequent, and make the necessary corrections for sensitivity and baseline changes. A calibration check should also be run after the last sample.

9.7 In the event that a sample is outside of the linear response of the instrument, it must be diluted to within range or reanalyzed using a less sensitive setup. This is commonly accomplished by calibrating the instrument with higher concentration standards using a secondary or tertiary wavelength having less sensitivity.

10.0 QUALITY CONTROL

A good quality control (QC) program is mandatory in that it is the only process through which a judgement can be made concerning the reliability of the data. The minimum requirements of the QC program include analyses of blanks, assessments of recovery, and assessments of precision and accuracy with duplicates and certified reference materials.

10.1 Method blanks -- these include container blanks, transportation blanks, dissection blanks, and reagent blanks.

10.1.1 Sources of sample contamination are numerous and can only be estimated or controlled through the use of method blanks. All containers and apparatus

should be checked for contamination prior to use. This can be performed using an appropriate wash or dilute acid leach that can then be analyzed in the same manner as the samples. Transportation blanks are derived from empty containers that have been stored and carried with the samples. A small amount of dilute acid [5% (v/v) HNO_3] is used to rinse the inside of the container. The acid rinse is then reserved for analysis.

10.1.2 Dissection blanks are prepared by rinsing utensils that have been used for dissection with a known volume of metal-free water. A dissection blank should be analyzed with each batch of samples.

10.1.3 Reagent blanks are by far the most common form of method blanks. For tissues, a reagent blank contains the same acid volumes and is treated identically to the samples. A minimum of three reagent blanks should be analyzed with every digestion set.

10.2 The results obtained from the blanks should be used to calculate the limit of quantitation (LOQ) for the method. This is the assigned value above which reliable data can be reported. A common method for calculating the LOQ is as follows:

$$LOQ = \frac{2S(B)}{M}$$

where $S(B)$ = standard deviation of the blanks (in instrument response units)

M = slope of the calibration curve

10.3 Sample duplicates - these can include duplicate samples (two fish) from the same area, duplicate subsamples from the same fish, duplicate digestions from the same subsample, or duplicate analyses of the same sample extract. The choice of duplicate type and the frequency of duplicate analysis is a function of the analysis requirements.

10.3.1 Duplicates are analyzed in order to determine the degree of repeatability or precision of an analysis. Duplicates are not approopriate criteria for determining analytical accuracy.

10.3.2 The minimum number of duplicates required per sample batch is difficult to estimate as it depends on a number of factors. As a general rule, one should duplicate at least every tenth sample. Precision can be determined from the standard deviation of a number of replicate analyses.

10.4 Certified reference materials (CRM) are invaluable as a means for determining the suitability of a particular method. They can be purchased from a number of agencies and are available in a range of matrices for inorganic substances (e.g., U.S. EPA Trace Metals in Fish Tissue or the NBS Oyster Tissue).

10.4.1 Unlike an analyte spike, a CRM tests the dissolution technique as well as instrument calibration and matrix interferences. An analyte spike must be used when CRMs are not available. A spike should be added prior to digestion to duplicate aliquots of a sample being analyzed. The concentration of the spike should be approximately 0.5 to 2.0 times what is already present and at a level that is readily detected by the method.

10.4.2 Each analysis batch should include at least one CRM or spike digested in triplicate. If a second CRM of similar matrix is available, each should be digested in duplicate. The data obtained on each standard analyzed must be used to troubleshoot the method if the results are outside the acceptable range. At no time should the data be used to determine a scale up or scale down factor to compensate for recovery. The recovery of a method can be calculated using the data obtained from the CRM as follows:

$$\% \text{ Rec} = \frac{\bar{x}}{c} \times 100$$

Where $\bar{x}$ = the mean result obtained

c = the mean certified value or level of the spike

10.5 Maintenance of records - The data obtained from any QC work should be recorded in an organized manner to allow for easy retrieval and reviewing. If sufficient data has been collected, it is recommended that these be plotted on a control chart for a quick visual assessment. A typical control chart is presented in Figure 1.

10.5.1 The quality control chart can be used to determine if the following recommended guidelines are met.

10.5.1.1 Not more than 1 in 20 results lie outside two standard deviations (warning limit). A result outside three standard deviations requires action.

10.5.1.2 Not more than seven consecutive results are on the same side of the mean.

10.5.1.3 There are no regular periodic variations.

10.6 Cleaning and preparation of labware is an integral part of a quality assurance/quality control (QA/QC) program. Many cleaning procedures have been proposed in the literature that are suitable for decontaminating apparatus. The main concerns with cleaning are removing elements of interest from labware while maintaining an inactive surface. Some cleaning procedures tend to be too harsh, producing a surface with an ion exchange capacity. In this case a solution could partially or completely "lose" an analyte to the container walls.

10.6.1 If at all possible, labware should not be used for work where analyte concentrations vary by more than ten times. For example, never use glassware for tissue analysis that has also been used for sediments. If one can use dedicated glassware, the cleaning requirements are greatly simplified.

10.6.2 A good universal cleaning procedure for glass and plasticware is outlined below.

10.6.2.1 If required, first use a metal-free detergent and warm water.

10.6.2.2 Rinse with tap water followed by distilled deionized water (DDW).

10.6.2.3 Soak apparatus in a dilute acid (25% HNO_3) bath for 24 hours. If possible, the bath should be maintained at an elevated temperature (50 to 60° C).

10.6.2.4 Rinse apparatus with large volumes of DDW and use immediately. If a time lapse must exist, the apparatus should be stored under dust-free conditions and rinsed further with DDW prior to use.

NOTES:
- Change the acid bath periodically such that no significant buildup of metals occurs.
- At no time should a metal containing reagent such as chromic acid be used.

10.7 Round robin or interlaboratory check programs - In addition to the quality control measures discussed above, all laboratories should participate in interlaboratory check programs.

11.0 PROCEDURE

11.1 Homogenize samples prior to analysis to ensure that a representative aliquot is taken. Any grinder or homogenizer that has been found to be free of contamination may be used. Samples should be ground wet to avoid losses of volatile elements (Hg, Se, etc.) during drying.

11.2 Transfer the sample paste to a container suitable for storage. If not immediately analyzed, the samples should be frozen (-20° C) until required.

11.3 Accurately weigh representative aliquots of homogenized tissue to the nearest 0.1 mg. If sample size permits, approximately 5g is required to maintain optimum detection limits. Transfer the weighed tissue to a precleaned 125-mL Erlenmeyer flask equipped with an all-glass reflux cap.

Analyze a sufficient number of reagent blanks, sample duplicates, analyte spikes, and certified reference materials concurrently (Section 10).

11.4 Add 10.0 mL of concentrated nitric acid (ACS grade or better), replace cap and swirl. Allow flask to stand at room temperature for about 15 hours in a dust-free ventilated environment. Periodically swirl the contents to help solubilize the tissue.

11.5 After 15 hours, gently heat the flask to approximately 100° C - hold at this temperature for one hour. Gradually increase the temperature in 50° C increments to a maximum of 250° C. Continue digesting until all tissue has been solubilized. This usually takes about four hours. Do not rush the initial digestion as losses of volatile elements will likely occur. Once digestion is complete, cool flasks to room temperature and add 4.0 mL of perchloric acid.

CAUTION: Perchloric acid is a strong oxidizing agent. The analyst must be fully aware of the precautions associated with its use. The technique, as described, does not require the use of a perchloric hood.

11.6 Return flasks to the hotplate which has been cooled to about 200° C. Continue heating for 1 h, then increase plate temperature to 300° C. Hold at this temperature until all traces of nitric acid fumes have disappeared and the solutions have become clear. Do not overheat flasks or allow perchloric fumes (dense white) to appear. Remove the extracts and cool to room temperature.

11.7 When the digestion is complete, rinse the caps into the flasks and transfer the extract to a precleaned 100-mL volumetric flask. Rinse the flasks 3 times with DDW and combine with the extract. Adjust the volume with DDW and transfer to a precleaned plastic bottle.

NOTE: Some elements are not as stable as others in solution and therefore should be analyzed first. Stability can be determined by daily analysis

of the extracts, however, the following can be used as a guideline:

Sb, Pb, Hg, Se and Ag - analyze within one day
As and Cd - analyze within two days
Cr, Cu, Ni and Zn - analyze within one week
Be and Tl - to be determined.

11.8 Instrumental analysis - The extracts will be analyzed using various techniques of atomic absorption spectrophotometry (AAS). The method of choice (i.e., GFAA vs HYDAA) depends on instrument availability, analyte concentration and sample matrix. In some instances it may be useful to use more than one AAS method to confirm a result.

11.8.1 Follow the manufacturer's instructions for initial setup and calibrate as outlined in Section 9 of this method. As every instrument responds uniquely to a given set of conditions, it is the analyst's responsibility to develop the optimum set of parameters. Use calibration standards and CRMs to ensure that optimum conditions exist.

11.8.2 Table 1 lists some general information for each of the priority pollutant metals.

11.8.3 It is possible to use alternate methods of detection providing they have been validated using a sufficient number of previously analyzed samples or CRMs.

11.9 All data generated must be clearly recorded on a strip chart, printer or manually logged in prepared tables. The order in which the extracts are analyzed should be the same as they appear in the records. The data, when assembled, should be reported in consistent units (i.e., mg/L) to avoid errors when calculating the final results (ug/g). The final report should contain all necessary methodology, results, quality control data (e.g., blank values) and limits of quantitation for each element. The report must clearly state if any data were blank-corrected.

12.0 CALCULATIONS

12.1 All results are reported as micrograms of element per wet gram of tissue:

$$\text{ug/g ELEMENT (wet weight basis)} = \frac{C \times V}{W}$$

where:

C = concentration (may be blank corrected) of element in final extract (ug/mL)

V = volume of final extract (mL)

W = weight of wet tissue (g)

Reagent blank corrections may be made and blank values must always be reported.

13.0 PRECISION AND ACCURACY

In order to estimate precision and accuracy (single lab, multi-operator), a number of CRMs and analyte spikes were analyzed using this method. Table 2 summarizes typical data obtained. No data are currently available for either beryllium or thallium.

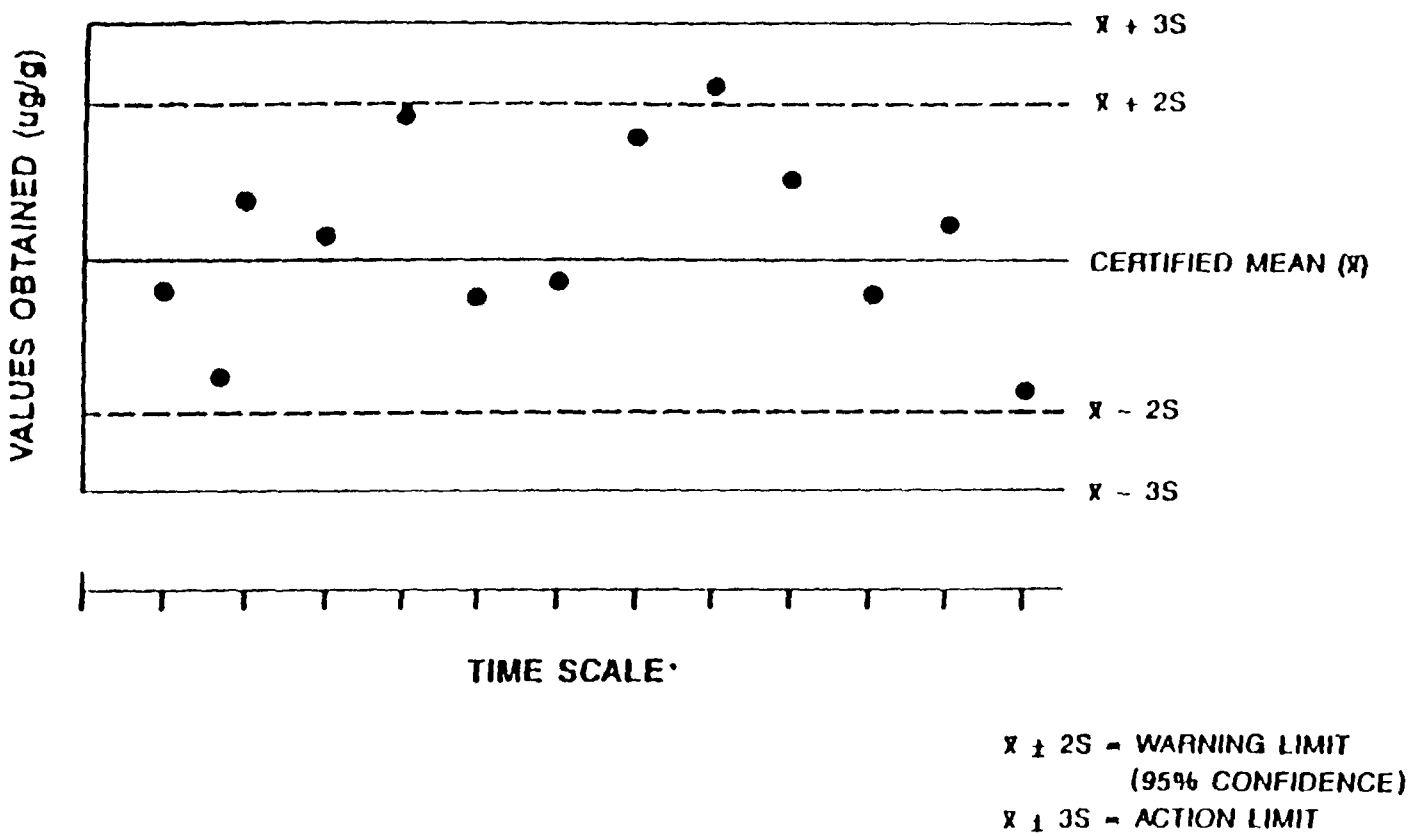


Figure 1. Quality control chart.

TABLE 1 - GENERAL INFORMATION FOR EACH PRIORITY POLLUTANT METAL

Element	Method[1]	Wavelength (nm)	L.O.Q.[2]	Signal	Notes
Antimony	HYDAA	217.6	0.002	Peak Area	
	GFAA	217.6	0.02	Peak Height	
Arsenic	HYDAA	193.7	0.002	Peak Area	
	GFAA	193.7	0.02	Peak Height	Requires a Matrix Mod
Beryllium	GFAA	234.9	0.002	Peak Height	
Cadmium	DFAA	228.8	0.1	Direct	
	GFAA	228.8	0.01	Peak Height	
Chromium	DFAA	357.9	0.2	Direct	
	GFAA	357.9	0.02	Peak Height	
Copper	DFAA	324.7	0.1	Direct	
	GFAA	324.7	0.01	Peak Height	
Lead	DFAA	383.3	1.0	Direct	
	GFAA	383.3	0.02	Peak Height	Requires a Matrix Mo
Mercury	CVAA	253.6	0.01	Peak Height	
Nickel	DFAA	232.0	0.5	Direct	
	GFAA	232.0	0.02	Peak Height	
Selenium	HYDAA	197.3	0.01	Peak Area	
	GFAA	197.3	0.02	Peak Height	Requires a Matrix M
Silver	DFAA	328.1	0.1	Direct	
	GFAA	328.1	0.01	Peak Height	
Thallium	GFAA	276.8	0.02	Peak Height	
Zinc	DFAA	213.9	0.1	Direct	

[1] HYDAA = Hydride generation atomic absorption.
GFAA = Graphite furnace atomic absorption.
DFAA = Direct flame atomic absorption.

[2] L.O.Q. = limit of quantitation - micrograms of element per wet gram of tissue based on 5g (wet) to 100 mL.

TABLE 2 - TYPICAL DATA OBTAINED ON A CERTIFIED REFERENCE MATERIAL (NATIONAL BUREAU OF STANDARDS OYSTER TISSUE)

Element		Certified/Spiked ($\bar{x} \pm$ S.D.)	n	Found ($\bar{x} \pm$ S.D.)
Antimony	Sb	5.0 (spike)	5	4.6 ± 0.8
Arsenic	As	13.4 ± 1.9	18	12.5 ± 0.8
Beryllim	Be	- - -	-	No Data
Cadmium	Cd	3.5 ± 0.4	18	3.84 ± 0.22
Chromium	Cr	0.69 ± 0.27	10	0.65 ± 0.08
Copper	Cu	63.0 ± 3.5	18	62.1 ± 1.2
Lead	Pb	0.48 ± 0.04	18	0.48 ± 0.08
Mercury	Hg	0.057 ± 0.015	10	0.060 ± 0.019
Nickel	Ni	1.03 ± 0.19	10	0.90 ± 0.12
Selenium	Se	2.1 ± 0.5	5	2.35 ± 0.33
Silver	Ag	0.89 ± 0.09	10	0.83 ± 0.06
Thallium	Tl	- - -	-	No Data
Zinc	Zn	852. ± 14.	18	855. ± 19.

All results expressed as micrograms of element per gram of tissue.

Lipid Determination

Sample Extraction

The fish tissue will be received from the field ready for extraction. No grinding, blending, or homogenizing will be needed.

The extraction will be carried out using SW-846 (3rd Edition) Method 3540, steps 7.3 - 7.10. Ten grams of sample will be used for the extraction. It will not be necessary to add the surrogate spiking solution or the matrix spiking solution since no organic analysis will be conducted on the extracts. The final volume of the extract should be 10 ml.

Solvent Evaporation

Pipette 1 ml of the extract into a weighed aluminum drying pan. Place the pan in a fume hood (fan off, door closed) for 24 hours to remove the solvent by evaporation. The percent extractable lipid is calculated as follows:

$$\% \text{ Lipids} = \frac{\text{final weight - total weight}}{1 \text{ g}} \times 100$$

Attachment V

Microwave Application Note for Acid Digestion

Sample Type: Fish Tissue

Summary:

This method provides for the acid digestion of whole-ground swordfish in a closed Teflon® PFA vessel using microwave heating for analysis by spectroscopic or wet chemical methods.

Required Equipment:

1. MDS-81D Microwave Digestion Oven, CEM
2. Teflon® PFA Vessels (120-mL size) with pressure relief valve
3. Digestion Turntable
4. Capping Station
5. Volumetric Glassware

Reagents:

1. Nitric Acid (70%)

Method:

1. Grind the sample so that it is of uniform consistency and no solid pieces larger than 1 mm in diameter remain.

2. Transfer .5 g of sample into a vessel and add 6 mL of HNO_3. Place a safety valve on the vessel and then tighten the cap using the Capping Station. Place the vessel in the turntable and attach a venting tube.

3. Repeat Step 2 until the turntable contains three evenly spaced vessels.

4. Turn the MDS-81D exhaust on to the maximum fan speed. Activate the turntable so that it is rotating.

5. Program the instrument for 3 min time and 45 percent power in Program 1 and 20 min time and 30 percent power in Program 2. Depress the START key and allow the sample mixtures to heat.

6. Allow the solutions to cool for 5 min and manually vent each vessel. Open the vessels and transfer to 100 mL volumetric flasks. Dilute to volume.

CAUTION: Manual venting of CEM closed vessel should only be performed when the vessel contents are at or below room temperature to avoid the potential for chemical burns. When venting vessels, hand, eye, and body protection are recommended.

Attachment VI

Single Acid Digestion of Fish Tissue for Metals Using an Aluminum Block Digester

Summary

An aluminum block digester has been successfully employed to simultaneously prepare up to 40 tissue samples using only nitric acid as the oxidizing reagent. Recovery was acceptable for all the following metals in a water matrix: Ag, Al, As, Ba, Be, Cd, Cr, Cu, Fe, Mn, Ni, Sb, Pb, Se, Tl, Zn, Ca, and Mg. Recovery of spiked tissue was acceptable for all these metals, except selenium.

Apparatus

1. Block Digester
2. Digester tubes with constricted neck
3. Glass weighing boats
4. Teflon® rod
5. Volumetric glassware

Reagents

1. Nitric acid (70%), Ultrex® or equivalent.

Procedure

1. Place 5 g of the homogenized tissue into 10-in. digester tubes. The process of placing a wet tissue sample through the constriction on the digester tube may be facilitated by using glass weighing boats with an elongated neck and a Teflon® rod.

2. Prepare quality control blanks, standards, duplicates, and spikes along with the samples.

3. Add 10 mL of concentrated nitric acid to each tube. Although not imperative, allowing samples to remain in contact with the acid at room temperature for an hour is desirable to reduce foaming when heat is applied.

4. Place the samples on an aluminum block digester at room temperature. Because of the large amount of acid evolved, it is recommended that an acid scrubber be used in conjunction with a conventional hood.

5. Set the controller for 60 min at 150°C and 90 min at 250°C. The gradual increase from room temperature to 150°C allows the samples to dissolve with minimal foaming. The 250°C temperature is required to overcome the reflux action at the constriction on the digester tube. The samples must be taken to dryness to completely decompose the lipids. Most 5-g samples will be black when dryness is reached.

6. Remove samples from digester, cool, and add an additional 10 mL of concentrated nitric acid. Return the samples to a cool block set for 250°C for 90 min.

7. Placing cold samples on a hot block may cause bumping, so the temperature of the block should be reduced to near 100°C. The 150°C step is necessary only for preliminary digestion. About

60 min is required to heat the block to 250°C, and about 20 min at temperature is required to remove 10 mL of acid. A white residue indicates that the digestion is complete. Usually over half of the samples will turn white after addition of 20 mL of acid, and over 90% are digested after 30 mL. Attempts to add larger amounts of acid initially proved unsuccessful because of foaming and bumping.

8. Redissolve the white residue by adding 10 mL of 10 percent nitric acid. Heat to about 90°C and mix with a vortex-type mixer.

9. Transfer to a 50-mL volumetric flask and dilute to volume (final matrix 2 percent HNO_3).

U.S. Environmental Protection Agency
CLP Sample Management Office
P. O. Box 818
Alexandria, Virginia 22313
Phone: (703) 557-2490

SAS Number
[ ]

Special Analytical Services
Regional Request

[X] Regional Transmittal [ ] Telephone Request

A. **EPA Region and Site Name:** Region V/Kerr-McGee, Kress Creek Site

B. **Regional Representative:** Pat Churilla

C. **Telephone Number:** (312) 353-5210

D. **Date of Request:** January 29, 1993

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. **General description of analytical service requested:**

   Metals leachability analysis of sediment samples by a procedure analogous with the Toxicity Characteristic Leaching Procedure (TCLP). Instead of the samples being extracted with the buffered extraction fluid called for in the TCLP, the samples will be extracted with Kress Creek surface water. The extracts will then be analyzed, for the TAL elements (Attachment I), per the procedures described in the SOW/ILM01.0.

   Due to the possibility of the samples being radiologically contaminated, a laboratory that is set up to deal with wastes of this nature will need to be procured.

2. **Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentrations):**

   Three sediment samples will be collected during the sampling event. These sediment samples will contain low to medium concentrations of the target elements.

3. **Purpose of analysis [specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.]:**

   Superfund Remedial

4. **Estimated date(s) of collection:**

   January 1993 and again May through July 1993

5. **Estimated date(s) and method of shipment:**

Method of shipment will be daily shipments by overnight carrier.

6. **Approximate number of days results required after lab receipt of samples:**

Samples must be extracted within 7 working days from the day of collection.

The leachate extracts must be analyzed within 28 days of extraction for Hg, and the other metals must be analyzed within 180 days of extraction.

The laboratory will be required to provide results within 35 days of receipt of the samples.

7. **Analytical protocol required (attach copy if other than a protocol currently used in this program):**

The sample extraction will be done with a modified version of Method 1311, as described in the Federal Register—June 29, 1990. The modification calls for the samples to be extracted with Kress Creek surface water, instead of TCLP buffered extraction fluid. The modified extraction procedure can be found in Attachment II. The extracts will be analyzed for the inorganics listed in Attachment I, according to the procedures described in the SOW/ILM01.0.

After extraction, the extracts must be kept at 4°C and preserved, acidifying to a pH <2 with HNO_3.

8. **Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):**

See Attachment II for the extraction modifications to the TCLP.

Sediments will be collected in 1-L widemouth glass jars. Samplers are instructed to only add sediment to the sample jars. If interstitial water is present in the sample at the time of sample initiation, remix the water with sediment and proceed.

If dilutions are necessary due to saturation peaks, they must be done in increments of 10. The results of all dilutions must be reported.

A TCLP blank must be carried through the extraction procedure.

Follow the method of standard additions for the metals analyses.

Detection limits shall not exceed one-tenth of the concentration that would define the sample as a hazardous waste for each parameter. If the concentration of the analyte after correction for the matrix spike recovery is $\geq 10\%$ of the regulatory level, the sediment extract must be reextracted using a smaller aliquot and spiked at the regulatory level such that the native analyte is at approximately the regulatory level.

A minimum of a four point calibration curve must be used for all analyses. If sample concentrations exceed the calibration range, sample must be diluted to fall within the calibration range.

Matrix spike and matrix spike duplicate (MS/MSD) recoveries must be determined. MS/MSD recoveries must be performed for each of the analytes.

The case narrative must discuss any sample problems.

9. **Analytical results required (if known, specify format for data sheets, QA/QC reports, chain-of-custody documentation, etc.). If not completed, format of results will be left to program discretion.**

Deliverables equivalent to those required in the TCLP shall be provided.

Report as per referenced SOW. Provide all QA/QC data associated with analyses. Provide data summary and chain of custody information. All raw data must be delivered with the data results.

The specific deliverables must also include:

A physical description of the samples.

The calculations used to compute percent dry solids and the weight of the liquid phase.

Sample extraction dates must be provided.

Sample preparation data including (but not limited to):

a. Weight(s) of extracted samples (100 g minimum aliquot size is required for 100% solids content) and volume of any filtrate (Sections 7.2.2 and 7.2.5).

b. Preliminary evaluations of percent solids.

c. pH data for the extraction fluid.

d. Dates of each preparation step, with associated weights and measured volumes.

e. pH value of final extract.

f. Holding times (Section 7.4) are to be met and are to be counted from the date of collection.

g. The final volumes of the extract and the volume of the extract analyzed.

ITEMS "a" - "e" AND ITEM "g" MUST BE A PART OF FORM I REPORT.

10. **Other (use additional sheets or attach supplementary information, as needed):**

None.

11. **Name of sampling/shipping contact:**

David Shekoski Phone: (414) 272-2426

I. Data Requirements

Precision and detection limits as required by the appropriate SOWs.

II. Quality Control Requirements

Audits Required	Frequency of Audits	Limits* (± % or conc.)
TCLP Extraction		
Preparation Blank for Extract Fluid (see Section 8.1 of Method 1311)	Each set of solid samples	<5% of regulatory levels of Table 1. Discuss in case narrative if larger than CRDLs of SOW.
Analysis of TCLP Extracts		
Preparation Blank for TCLP Extract Determinations	Per appropriate SOW and set-up with each TCLP extract batch	CRDL of appropriate SOW for Attachment I constituents.
MS/MSD	See Attachment II	Advisory – Used to correct TCLP values recovery.
	One for each set sample extracts	RPD ≤20% (MS/MSD)
All other QC audits per ILM01	Per ILM01	Per ILM01
Note: These are minimum requirements. Report the actual detection limits used based on allowable methodology options.		

III. Action Required if Limits are Exceeded:

Take corrective action and retest samples. Contact Dennis Wesolowski at (312) 886-1970 for discussion of problems that might result in the delay of reporting sample results.

Attachment I

Inorganic Target Analyte List (TAL)	
Analyte	**Contract Required Detection Limit (μg/L)**
Aluminum	300
Antimony	100
Arsenic	2
Barium	200
Beryllium	20
Cadmium	10
Calcium	1,000
Chromium	40
Cobalt	60
Copper	20
Iron	30
Lead	60
Magnesium	100
Manganese	20
Mercury	0.5
Nickel	70
Potassium	100
Selenium	3
Silver	30
Sodium	100
Thallium	100
Vanadium	100
Zinc	10

Attachment II
TCLP Extraction Procedure Modifications

Samples will be wet; therefore, the filtration procedure (Section 7.1.1.7) will produce interstitial water. Also, any water collecting on top of sediment or soil is not to be discarded, but mixed with sample prior to filtration or percent solid determination (Section 7.1.1). Particle size reduction will not be necessary.

Instead of the extraction fluids described in Method 1311, as noted in the Federal Register of June 29, 1990, Kress Creek surface water will be substituted. The extraction will be performed as described in the method with this simple substitution of solutions. Four liters of Kress Creek surface water will be collected at each sampling location prior to sediment sampling. The surface water will be collected in an amber 4-L glass sample bottle, to which no preservative has been added.

All constituents of Attachment I are required to be determined and reported for TCLP extracts. A matrix spike/matrix spike duplicate (MS/MSD) for all constituents in Attachment I will be prepared and determined.

MS/MSD results are advisory and used for calculation purposes. The sediment results will be corrected for MS recoveries.

The average MS/MSD recovery developed for one of the soil extracts will be applied to all of the soil extracts.

Appendix D
Data Validation Procedures

CONTENTS

Radiological Data Review and Validation Guidelines, Ra-226 by Radon Emanation

January 1993

Radiological Data Review and Validation Guidelines, Ra-226 by Radon Emanation

1. Scope and Applicability

This document provides guidance for the review of laboratory data packages and the validation of results for Ra-226 by radon emanation analyses of environmental samples. It is applicable to results obtained as part of the Kress Creek site Remedial Investigation/Feasibility Study (RI/FS).

2. Purpose

The purpose of review and validation is to determine the quality of each data point and to flag each data point with a qualifier. In addition, data validation provides a review of laboratory quality control (QC) measures so that corrections to laboratory procedures can be implemented, if necessary. It is assumed that field samplers and analytical laboratories have followed approved methods and adhere to good laboratory practices. This procedure provides guidelines for review and validation of radioanalytical data packages and establishes criteria for applying appropriate data qualifiers to individual data points.

3. Criteria

This document provides criteria for evaluating Ra-226 by radon emanation data under the general categories of radiological data package completeness, holding times, calibration, blanks, lab and field replicates, matrix spike/matrix spike duplicate (MS/MSD) samples, cell constants, result verification, minimum detectable activities (MDAs), and overall data assessment. The criteria for each of these categories are discussed in detail in the following sections.

3.1 Radiological Data Package Completeness

Each data package should be checked for completeness prior to initiating data validation. The data validator should request the laboratory to submit any missing information. A complete data package consists of a case narrative, a QC data package, and a sample results data package. The contents of these packages are described below.

3.1.1 Case Narrative

The case narrative should include the following items:

- Cross reference of sample and laboratory numbers
- Problems encountered (reanalyses, broken sample containers, insufficient sample, excessive holding times, matrix problems, instrument problems, etc.)
- Descriptions of each out-of-control situation, corrective actions taken, and resolution
- Signature of the laboratory manager or designee

3.1.2 QC Package

The QC summary package should contain the following items:

- **Calibrations Data Summary.** This summary should include the identification number of each scintillation detector calibrated, calibration date, identification, activity, certification, and expiration date of standard material. The midpoint voltage of the plateau curve for the photomultiplier tube in each detector, and raw count rate data for calibrations of each detector should also be included.

  Continuing calibration verification data should include identification of each detector system checked, date of the check, identification and activity level of standard material used for the checks, and raw count rate data from the detectors checked.

  Instrument background data should also be included in the calibrations data summary, including detector identification, count duration, QC charts, background counts, and any statistical evaluation results for background counts.

- **Reagent Blanks Data Summary.** The reagent blanks data summary should include blank identification numbers, identification of samples analyzed with the blank, type of method blank used, MDA calculated for each blank, and raw data associated with the blank analysis, including detector identification, aliquot size, date of analysis, and analyst's initials. An upper limit of counts for reagent blanks should be established that, if exceeded, would be cause for data rejection and reanalysis of samples.

- **Duplicate/Replicate Data Summary.** This summary includes data on precision including identification of detector used, analyst's initials, date of analysis for sample and duplicate/replicate, sample identification, activity results for sample and duplicate/replicate, count durations, and calculated uncertainties and MDAs for sample and duplicate/replicate.

- **Cell Constants Data Summary.** This summary includes the identification of each cell/instrument combination, concentration in pCi/l of standards in solutions used to prepare standard "bubblers," time interval between initial and final deemanations, counts obtained for each cell/instrument combination, and efficiency or calibration factor for each cell/instrument combination. Background data, including counts and count durations, should also be shown for each cell.

- **MS/MSD Data Summary.** The MS/MSD summary should include date of MS/MSD analyses; detector identifications; MS/MSD identification; activity of spike added; sample activity; MS activity; MSD activity; 2σ counting error for sample, MS, and MSD activities; percent accuracy; relative percent difference; and raw count data for each result.

3.1.3 Sample Results Package

The sample results data package should contain the following items:

- Summary page (Form 1 or certificate of analysis) showing the results for each sample (including blanks, duplicates/replicates, MS/MSDs, and reruns), including counting error and detection level for results reported as less than the MDA.

- Raw data backup for sample results including chain of custody (COC), sample identification, date of analysis (counting), detector identification, raw sample count data,

background counts, count duration, aliquot used, detector efficiency, sample activity, error, and MDA results. The raw data should include any count data (background counts or spectra) necessary to support calculated MDA values.

3.2 Holding Times

Sample holding time refers to the period from the time of sample collection to the time it is analyzed (counted). Sample collection dates appear on the COC record in the data package. This date should be compared with the analysis date on the raw data count sheet and also with the data summary form to make sure dates are consistent and that no sample mixup has occurred. Dates of receipt and signatures should be checked for continuity on the COC record. The sample holding time for water samples is 6 months (180 days). There is no standard holding time for soil samples.

Flag water samples with holding times greater than 180 days as "estimated" (J). Flag water samples with holding times greater than 270 days as "rejected" (R).

3.3 Calibration and Calibration Verification

Calibration of alpha scintillation counters should be conducted at least quarterly using a standard traceable to the National Institute of Standards and Technology (NIST).

Flag results "estimated" (J) if the calibration was conducted using an expired NIST traceable standard. Flag results "rejected" (R) if the calibration was conducted using a standard that is not traceable to NIST. Flag results "rejected" (R) if the plateau curve and the "midpoint" of the plateau were not determined during calibration.

Calibration verification of alpha scintillation counters should be conducted at least weekly using a check source that approximates typical sample activity. The source check count rate should be within 3 standard deviations (3σ) of the source check count rate determined at the time of calibration.

Results from a counter that has a calibration verification result outside of the 3σ range should be flagged "estimated" (J).

Background should be measured at least weekly for each photomultiplier tube. Weekly background checks should be within 3σ of the background determined at the time of calibration. Flag results from counters with background count rates outside of the 3σ control limits as "estimated" (J).

3.4 Reagent Blanks

Reagent blanks are used to determine background counts that result from environmental and reagent radiation sources and to monitor instrument background contributions. In addition, reagent blanks are used to determine statistical errors.

Reagent blanks should have been prepared and analyzed at a frequency of 5 percent of the samples. Flag results as "estimated" (J) if blanks were not run at a frequency of 5 percent or greater.

The results for blanks should be reported and summarized on a QC chart. Control limits should be set at 3σ from the mean. Flag sample results as "estimated" (J) if the blank for that group of samples falls outside of the 3σ control limit.

If radionuclides are detected in blanks, then sample results for the same radionuclides should be considered positive only if they exceed 5 times the blank concentration. Samples that show positive results less than 5 times the blank values (for radionuclides detected in blanks) should be flagged "estimated" (J).

Compare the blank activities to the MDA. If blank results are greater than the MDA, laboratory contamination may be indicated. If blank MDAs consistently exceed the detection limit stated in the Quality Assurance Project Plan (QAPjP), unacceptable counting times may be indicated. Use professional judgment to determine if the samples should be flagged "estimated" (J) for these conditions.

If reagent blank results are greater than 3σ below the mean background, then all nondetected results should be flagged "rejected" (R), and all positive results flagged "estimated" (J).

3.5 Duplicates/Replicates

Replicate analyses are defined as identifiable aliquots of the same sample taken through the entire procedure. They provide information on reproducibility of field sampling techniques and laboratory preparation. One replicate should be analyzed for each batch of 10 samples.

Check the raw data and calculate the relative percent difference (RPD) for the sample and replicate using the following equation:

$$RPD = \frac{|S - R|}{(S + R)/2} \times 100$$

where:

S = sample result
R = replicate result

A control limit of 20 percent for water and 35 percent for soil samples for the RPD shall be used for sample results greater than 5 times the MDA.

A control limit of MDA for water samples and 2x MDA for soil samples shall be used for sample results less than 5 times the MDA, including a situation when only one of the sample/replicate pair results is less than 5 times the MDA.

If replicate results for a particular radionuclide fall outside the appropriate control windows, qualify the results for that radionuclide in all associated samples of the same matrix as "estimated" (J).

3.6 Matrix Spike/Matrix Spike Duplicate Samples

MS/MSD samples are derived from a matrix that has been fortified with a known concentration of a specific isotope. These samples should be analyzed at a frequency of 1 (MS/MSD pair) in 20 samples or 1 set per batch, whichever is most frequent. The results of the analysis of these samples provide an indication of both laboratory accuracy and precision.

Accuracy for the set of samples will be evaluated by comparing the measured concentration with the known (spike) concentration in the MS/MSD sample. The comparisons will be made using the counting error (sigma) term associated with each measured concentration as the evaluation criteria. If the known value falls within three sigma (3σ) of the measured value, the accuracy associated with the MS/MSD is acceptable.

If the results of the MS/MSD fall outside of the 3σ range, the data reviewer will use professional judgment in conjunction with other QC criteria to determine if the data should be qualified as estimated or rejected.

Precision is a measurement of the reproducibility of analytical results under a standard set of conditions. It can be viewed as a measure of the magnitude of errors occurring in an analytical procedure. RPD is the method most commonly used to estimate precision when using MS/MSD samples in a set of analyses. RPD is defined in Section 3.5, Duplicates/Replicates.

No action is taken based on MS/MSD data alone to qualify a batch of samples. Since precision cannot be measured for a pair of samples that is below the quantitation limits of the method, duplicate samples are fortified and then evaluated.

The data reviewer should use informed professional judgment in conjunction with other QC criteria and determine the necessity for further data qualification. However, when MS/MSD data are interpreted, the following will be used as guidelines:

- A control limit of $\pm 3\sigma$ will be used in conjunction with the calculated RPD when the known value of the spikes is less than 20 times the MDA. The data reviewer may flag results as "estimated" (J) if professional judgment dictates.

- A control limit of $\pm 2\sigma$ will be used when known spike values are greater than 20 times the MDA. The data reviewer may flag the results as "estimated" (J) if justified by other QC parameters and the reviewer's professional judgment.

3.7 Cell Constants

The efficiency of each cell/instrument combination that is used in calculating activities, uncertainties, and MDAs must be reviewed by the data validator. In addition, background count rates for each cell should be verified.

The cell/instrument efficiency can be determined using the formula shown below:

$$E = \frac{C_N}{(A_S)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})}$$

where:

C_N = net count rate of standard [counts per minute (CPM)]
A_S = activity of Ra-226 in standard [disintegrations per minute (DPM)]
λ = decay constant of Rn-222
t_1 = ingrowth time of Rn-222 (time interval between initial and final deemanation)
t_2 = decay time of Rn-222 (time interval between deemanation and beginning of count)

The control limit for cell/instrument efficiencies is 3σ of the mean of the last 10 efficiencies. The efficiency should be close to 5.3 CPM/pCi. Flag results as "estimated" (J) if the efficiency falls outside of the 3σ control limit.

The background for each cell/instrument should be determined immediately after a sample has been counted to determine if the background count rate has changed. The control limit for background count rate is 3σ of the average of the last 10 background measurements. Background typically should be less than 0.2 CPM for environmental samples. Flag results as "estimated" (J) if the background count rate falls outside of the 3σ control limits.

3.8 Verification of Sample Result and MDA Calculations

Manual calculations should be performed to verify sample result and MDA calculations performed by the laboratory. Errors or discrepancies should be addressed in the comments section of the data validation report. The following formulas should be used for calculating sample concentration, counting error, and MDA.

3.8.1 Sample Result

$$A = \frac{CPM_G - Bkg}{(2.22)(E)(Vol)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})} \left[\frac{\lambda t_3}{(1 - e^{-\lambda t_3})}\right]$$

where:

A =	Ra-226 sample concentration (pCi/l or pCi/g)
CPM_G =	gross sample count rate
Bkg =	background count rate
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
Vol =	sample mass or volume (grams for solids, liters for water)
λ =	decay constant for Rn-222
t_1 =	time interval between initial and final deemanation
t_2 =	time interval between final deemanation and beginning of count
t_3 =	sample count time

3.8.2 Counting Error

$$ER = \frac{1.96 \left[\frac{CPM_G}{t_3} + \frac{Bkg}{t_{Bkg}}\right]^{0.5} (\lambda t_3)}{(2.22)(E)(Vol)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})(1 - e^{-\lambda t_3})}$$

where:

ER =	2σ counting error (pCi/l or pCi/g)
CPM_G =	gross sample count rate (CPM)
t_1 =	time interval between initial and final deemanation
t_2 =	time interval between final deemanation and beginning of count
t_3 =	count time for sample (min)
Bkg =	background count rate (CPM)
t_{Bkg} =	count time for background (min)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	cell/instrument efficiency factor
Vol =	sample mass or volume (grams for solids, liters for water)
λ =	decay constant for Rn-222

3.8.3 Minimum Detectable Activity

$$MDA = \frac{4.66\ (Bkg/t_3)^{0.5}\ (\lambda t_3)}{(2.22)(E)(Vol)(1 - e^{-\lambda t_1})(e^{-\lambda t_2})(1 - e^{-\lambda t_3})}$$

where:

MDA =	minimum detectable activity or Lower Limit of Detection (LLD)
Bkg =	background count rate (CPM)
t_1 =	time interval between initial and final deemanation
t_2 =	time interval between final deemanation and beginning of count
t_3 =	count time (min)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
λ =	decay constant for Rn-222
E =	cell/instrument efficiency factor

Verify that the calculations for activity, uncertainty, and MDA for each sample are correct. Address any errors or discrepancies in the comment section of the report.

3.9 Overall Data Assessment

As part of the overall data assessment, the results of the data validation process will be documented on the appropriate summary forms. The first step in the validation process is to review the case narrative, QC data package, and sample results data package using the checklist shown in Attachment 1. If the data packages are not complete, the data validator must contact the laboratory for the appropriate data.

After the data package review is completed, detailed review of sample results should begin. The data quality flags for each sample should be listed on the radiochemical analysis analytical results form (Form 1 equivalent or certificate of analysis). One form is required for each sample.

The data quality flags used for radiological sample results are shown below.

J	=	Indicates the analyte is present, but the reported value may not be accurate or precise because the associated quality assurance (QA)/QC was unacceptable. The result is considered "estimated."
R	=	Indicates the data is unusable. This flag is used when the result should not be used to support project decisions. The result is considered "rejected."
U	=	Indicates that the sample was analyzed, but the analyte was not detected above the stated concentration. The result is considered "undetected."

The following subqualifiers give further detail of the type and amount of qualification a given result has received.

D	=	Qualified because laboratory duplicate control limits were exceeded
C	=	Qualified because of instrument calibration problems
B	=	Qualified because of blank contamination problems

Q = Qualified for reasons not stated above—refer to the text of the report

4. References

Sample Preparation Standard Operating Procedures (SOPs) from Controls for Environmental Pollution.

QA—Standard Operating Procedure for Accepting Spike and Duplicate Results (CEP-QA-102, revised January 20, 1989).

Attachment 1

Radiochemical Data Completeness Checklist for Ra-226 Analysis by Radon Emanation of Soil and Water

1. ___ Case Narrative
   - ___ Abnormalities explained
   - ___ Matrix problems explained
   - ___ Instrument problems explained
   - ___ Improper collection, storage, preservation, container explained
   - ___ Hold times were met, explained if not met
   - ___ Signature of lab representative

2. ___ Quality Control (QC) Package

   A. ___ Calibrations Data Summary
   - ___ ID of each detector
   - ___ Dates of last efficiency factor check certificates and DPMs of check sources; counts obtained; count durations
   - ___ Midpoint voltage of plateau curve for photomultiplier tube in each detector
   - ___ Background counts obtained for each alpha scintillation detector with count times

   B. ___ Reagent Blanks Data Summary
   - ___ ID of each cell/instrument combination used
   - ___ Analyst initials
   - ___ Date reagent blanks were analyzed
   - ___ ID of samples analyzed with the reagent blanks
   - ___ Type of method blank used, minimum detectable activity (MDA) of method
   - ___ Volume of aliquot for reagent blanks

   C. ___ Replicate Sample Data Summary
   - ___ ID of each cell/instrument combination used
   - ___ Analyst initials
   - ___ Date sample and replicates were analyzed
   - ___ Sample IDs, values obtained for sample and replicates
   - ___ Count durations of sample and replicates
   - ___ Volume of aliquot for sample and replicates
   - ___ Calculated uncertainties and MDAs

   D. ___ Matrix Spike/Matrix Spike Duplicate (MS/MSD) Data Summary
   - ___ ID of each detector used
   - ___ Analyst initials
   - ___ Date MS/MSDs were analyzed
   - ___ ID of MS/MSDs
   - ___ Values obtained for MS/MSDs with uncertainty and MDA
   - ___ True value of MS/MSDs with uncertainty
   - ___ ID of samples analyzed with the MS/MSDs

E. ___ Cell Constants Data Package

___ ID of each cell instrument combination

___ Concentration in pCi/l of solutions used in "standard bubblers"

___ Time interval between initial and final deemanations

___ Count obtained and count durations for each cell/instrument combination

___ Background count rate and count duration for each cell

3. ___ Sample Results Package

A. ___ Sample Summary Data

___ Printed report of results and counting errors for samples and reruns

___ MDA calculated for each isotopic analysis for samples with activity less than MDA

B. ___ Sample/MDA Raw Data

___ Date of analysis

___ Background CPM

___ ID of each cell/instrument combination used

___ Calculated MDA

___ Calculation sheets including, sample ID, cell/instrument identification sample counts, background counts, count durations, sample aliquots used, cell constant values, time interval between initial and final deemanations, time interval between final deemanation and counting, calculated sample activity uncertainty, and MDA

4. Comments

_____

_____

_____

_____

Radiological Data Review and Validation Guidelines, Isotopic Analyses by Alpha Spectroscopy

February 1993

Radiological Data Review and Validation Guidelines, Isotopic Analyses by Alpha Spectroscopy

1. Scope and Applicability

This document provides guidance for the review of laboratory data packages and the validation of results from alpha spectroscopy analyses of environmental samples. It is applicable to the validation of U-234, U-235, U-238, Th-227, Th-228, Th-230, and Th-232 results from soil, biota, and water samples taken as part of the Kress Creek site Remedial Investigation/Feasibility Study (RI/FS).

2. Purpose

The purpose of review and validation is to determine the quality of each data point and to flag each data point with a qualifier. In addition, data validation provides a review of laboratory quality control (QC) measures so that corrections to laboratory procedures can be implemented, if necessary. It is assumed that field samplers and analytical laboratories have followed approved methods and adhere to good laboratory practices. This procedure provides guidelines for review and validation of radioanalytical data packages and establishes criteria for applying appropriate data qualifiers to individual data points.

3. Criteria

This document provides criteria for evaluating alpha spectroscopy data under the general categories of radiological data package completeness, holding times, calibration, blanks, lab and field replicates, matrix spike/matrix spike duplicate (MS/MSD) samples, resolution, chemical recoveries, result verification, minimum detectable activities (MDAs), and overall data assessment. The criteria for each of these categories are discussed in detail in the following sections.

3.1 Radiological Data Package Completeness

Each data package should be checked for completeness prior to initiating data validation. The data validator should request the laboratory to submit any missing information. A complete data package consists of a case narrative, a QC data package, and a sample results data package. The contents of these packages are described below.

3.1.1 Case Narrative

The case narrative should include the following items:

- Cross reference of sample and laboratory numbers
- Problems encountered (reanalyses, broken sample containers, insufficient sample, excessive holding times, matrix problems, instrument problems, etc.)
- Descriptions of each out-of-control situation, corrective actions taken, and resolution
- Signature of the laboratory manager or designee

3.1.2 QC Package

The QC summary package should contain the following items:

- **Calibrations Data Summary.** This summary should include efficiency, resolution, and background data used in the calibration and in the calibration verification process.

  The efficiency documentation should include detector and system identification numbers, date of calibration, standard reference isotopes and efficiencies, and certificates and activity values for standard calibration and reference sources.

  The resolution documentation should include a listing of names and activities of the isotopes used to determine system resolution, range in alpha energy of the detector system(s), memory (in channels) of the detector system(s), and results of resolution calculations for each system.

  Background documentation should include results of background checks for each system, including counts in each region of interest and count duration. Also, any QC charts available should be included.

- **Reagent Blanks Data Summary.** The reagent blanks data summary should include blank identification numbers, identification of samples analyzed with the blank, type of method blank used, MDA calculated for each blank, and raw data associated with the blank analysis, including detector identification, aliquot size, date of analysis, and analyst's initials. An upper acceptance limit should be identified for the maximum counts (or calculated concentration) that a reagent blank may exhibit before reanalysis is necessary.

- **Duplicate/Replicate Data Summary.** This summary includes data on precision including identification of detector used, analyst's initials, date of analysis for sample and duplicate/replicate, sample identification, activity results for sample and duplicate/replicate, count durations, and calculated uncertainties and MDAs for sample and duplicate/replicate.

- **MS/MSD Data Summary.** The MS/MSD summary should include date of MS/MSD analyses; detector identification; MS/MSD identification; activity of spike added; sample activity; MS activity; MSD activity; 2σ counting error for sample, MS, and MSD activities; relative percent difference; and raw count data for each result.

- **Chemical Recovery Data Summary.** This summary includes the activity of each tracer used, net counts for each isotope of interest, efficiency for each isotope of interest, count duration, and calculated chemical recovery for each sample analyzed.

3.1.3 Sample Results Package

The sample results data package should contain the following items:

- Summary page (Form 1 or certificate of analysis) showing the results for each sample (including blanks, duplicates/replicates, MS/MSDs, and reruns), including counting error and detection level for results reported as less than the MDA.

- Raw data backup for sample results including chain of custody (COC), sample identification, date of analysis (counting), detector identification, raw counts for each isotope of interest, background counts, tracer counts and tracer activity, chemical recovery, count duration, aliquot used, detector efficiency, sample activity, error, and

MDA results. The raw data should include any count data (background counts or spectra) necessary to support calculated MDA values.

3.2 Holding Times

Sample holding time refers to the period from the time of sample collection to the time it is analyzed (counted). Sample collection dates appear on the COC record in the data package. This date should be compared with the analysis date on the raw data count sheet and also with the data summary form to make sure dates are consistent and that no sample mixup has occurred. Dates of receipt and signatures should be checked for continuity on the COC record. The sample holding time for water samples is 6 months (180 days). There is no standard holding time for soil samples.

Flag water samples with holding times greater than 180 days as "estimated" (J). Flag water samples with holding times greater than 270 days as "rejected" (R).

3.3 Calibration and Calibration Verification

For alpha particle measurements, the detectors must be calibrated to obtain the counting efficiency for each of the radionuclides with a standard traceable to the National Institute of Standards and Technology (NIST). Each detector should be calibrated with an alpha standard representative of the target radionuclides within 1 year of the analysis date. The standard should be prepared in the geometry and weight ranges expected to be encountered.

Verify that the standards used to prepare efficiency and calibration verification standards are current and traceable to NIST. Flag the results "rejected" (R) if the standards are not traceable to NIST. Flag the results "estimated" (J) if they were obtained with expired NIST-traceable standards.

A calibration verification should have been performed weekly with an independently prepared verification standard. The measured efficiency value should not be more than three standard deviations (3σ) from the value determined at the time of initial calibration. Results for samples analyzed after a verification beyond control limits and before the next adjacent acceptable verification should be flagged "estimated" (J). If the calibration verification was not performed, all results should be flagged "estimated" (J).

Alpha spectrometers require a weekly energy vs channel calibration verification, with a source having at least two alpha emitters. The results from any detector where the energy calibration is more than three channels out of calibration should be flagged "estimated" (J).

Background spectra should be collected on each detector at least monthly for alpha spectrometer systems. Flag results "estimated" (J) if the current background is outside of the range of $\pm 3\sigma$ of the background established at the time of calibration.

3.4 Reagent Blanks

Reagent blanks are used to determine background counts that result from environmental and reagent radiation sources and from monitor instrument background contributions. In addition, reagent blanks are used to determine statistical errors.

Reagent blanks should have been prepared and analyzed at a frequency of 5 percent of the samples. Flag all affected results as "estimated" (J) if blanks were not run at a frequency of 5 percent or greater.

The results for blanks should be reported and summarized on a QC chart. Control limits should be set at 3σ from the mean. Flag sample results as "estimated" (J) if the blank for that group of samples falls outside of the 3σ control limit.

If radionuclides are detected in blanks, then sample results for the same radionuclides should be considered as positive only if they exceed 5 times the blank concentration. Samples that show positive results less than 5 times the blank values (for radionuclides detected in blanks) should be flagged "estimated" (J).

Compare the blank activities to the MDA. If blank results are consistently greater than the MDA, laboratory contamination may be indicated. If blank MDAs consistently exceed the detection limits stated in the Quality Assurance Project Plan (QAPjP), unacceptable counting times may be indicated. Use professional judgment to determine if the samples should be flagged "estimated" (J) for these conditions.

If reagent blank results are greater than 3σ below the mean background, then all nondetected results should be flagged "rejected" (R) and all positive results flagged "estimated" (J).

3.5 Duplicates/Replicates

Replicate analyses are defined as identifiable aliquots of the same sample taken through the entire procedure. They provide information on reproducibility of field sampling techniques and laboratory precision. One replicate should be analyzed for each batch of 10 samples.

Check the raw data and calculate the relative percent difference (RPD) for the sample and replicate using the following equation:

$$RPD = \frac{|S - R|}{(S + R)/2} \times 100$$

where:

S = sample result
R = replicate result

A control limit of 20 percent for water and 35 percent for soil samples for the RPD shall be used for sample results greater than 5 times the MDA.

A control limit of MDA for water samples and 2x MDA for soil samples shall be used for sample results less than 5 times the MDA, including an instance when only one of the sample/replicate pair results is less than 5 times the MDA.

If replicate results for a particular radionuclide fall outside the appropriate control windows, qualify the results for that radionuclide in all associated samples of the same matrix as "estimated" (J).

3.6 Matrix Spike/Matrix Spike Duplicate Samples

MS/MSD samples are derived from a matrix that has been fortified with a known concentration of a specific isotope. These samples should be analyzed at a frequency of 1 (MS/MSD pair) in 20 samples or 1 set per batch, whichever is most frequent. The results of the analysis of these samples provide an indication of both laboratory accuracy and precision.

Accuracy for the set of samples will be evaluated by comparing the measured concentration with the known (spike) concentration in the MS/MSD sample. The comparisons will be made using the counting error (sigma) term associated with each measured concentration as the evaluation criteria. If the known value falls within three sigma (3σ) of the measured value, the accuracy associated with the MS/MSD is acceptable.

If the results of the MS/MSD fall outside of the 3σ range, the data reviewer will use professional judgment in conjunction with other QC criteria to determine if the data should be qualified as estimated or rejected.

Precision is a measurement of the reproducibility of analytical results under a standard set of conditions. It can be viewed as a measure of the magnitude of errors occurring in an analytical procedure. RPD is the method most commonly used to estimate precision when using MS/MSD samples in a set of analyses. RPD is defined in Section 3.5, Duplicates/Replicates.

No action is taken based on MS/MSD data alone to qualify a batch of samples. Since precision cannot be measured for a pair of samples that is below the quantitation limits of the method, duplicate samples are fortified and then evaluated.

The data reviewer should use informed professional judgment in conjunction with other QC criteria and determine the necessity for further data qualification. However, when MS/MSD data are interpreted, the following will be used as guidelines:

- A control limit of $\pm 3\sigma$ will be used in conjunction with the calculated RPD when the known value of the spikes is less than 20 times the MDA. The data reviewer may flag results as "estimated" (J) if professional judgment dictates.

- A control limit of $\pm 2\sigma$ will be used when known spike values are greater than 20 times the MDA. The data reviewer may flag the results as "estimated" (J) if justified by other QC parameters and the reviewer's professional judgment.

3.7 Recovery Factors

An isotopic tracer solution is used to spike each sample prior to analysis by alpha spectroscopy. The tracer used should have chemical behavior similar to the target radionuclides. For most procedures, the recovery is determined using an isotope of the analyte of interest. This isotope is one that is not expected to occur in the samples to be analyzed.

The chemical recovery is calculated based on the net count rate [counts per minute (CPM)] obtained from the tracer, the actual activity or disintegration rate [disintegrations per minute (DPM)] contained in the tracer solution, and the instrument efficiency. The above terms are related in the following formula:

$$\text{CPM tracer found/DPM} = \text{eff} \times \text{chem recovery}$$

Chemical recoveries for thorium analyses should be greater than 20 percent, but less than 105 percent. Chemical recoveries for isotopic uranium analyses should be greater than 30 percent, but less than 105 percent.

Flag results "rejected" (R) if these criteria are not met and the sample activity is below the MDA.

For thorium analyses, flag results "estimated" (J) if the chemical recovery is greater than 10 percent, but less than 20 percent, and the sample activity is greater than the MDA. Flag these results "rejected" (R) if the chemical recovery is less than 10 percent and the sample activity is greater than the MDA.

For uranium analyses, flag results "estimated" (J) if the chemical recovery is greater than 20 percent, but less than 30 percent, and the sample activity is greater than the MDA. Flag uranium results "rejected" (R) if the chemical recovery is less than 20 percent and the sample activity is greater than the MDA.

3.8 Verification of Sample Result and MDA Calculations

Manual calculations should be performed to verify sample result and MDA calculations performed by the laboratory. Errors or discrepancies should be addressed in the comments section of the data validation report. The following formulas should be used for calculating sample concentration, counting error, and MDA.

3.8.1 Sample Result

$$A = \frac{CPM_G - Bkg}{(2.22)(E)(Vol)(R)}$$

where:

A =	sample concentration (pCi/l or pCi/g)
CPM_G =	gross sample count rate (CPM)
Bkg =	background count rate (CPM)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
Vol =	sample mass or volume (grams for solids, liters for water)
R =	chemical recovery

3.8.2 Counting Error

$$ER = \frac{2.0\left[\frac{CPM_G}{T_S} + \frac{Bkg}{T_{Bkg}}\right]^{0.5}}{(2.22)(E)(Vol)(R)}$$

where:

ER =	2σ counting error (pCi/l or pCi/g)
CPM_G =	gross sample count rate (CPM)
T_S =	count time for sample (min)
Bkg =	background count rate (CPM)
T_{Bkg} =	count time for background (min)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
Vol =	sample mass or volume (grams for solids, liters for water)
R =	chemical recovery

3.8.3 Minimum Detectable Activity

where:

$$MDA = \frac{4.66\ (Bkg/T)^{0.5}}{(2.22)(E)(Vol)(R)}$$

MDA =	minimum detectable activity
Bkg =	background count rate (CPM)
T =	count time (min)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
R =	chemical recovery

Verify that the calculations for activity, uncertainty, and MDA for each sample are correct. Address any errors or discrepancies in the comment section of the report.

3.9 Overall Data Assessment

As part of the overall data assessment, the results of the data validation process will be documented on the appropriate summary forms. The first step in the validation process is to review the case narrative, QC data package, and sample results data package using the checklist shown in Attachment 1. If the data packages are not complete, the data validator must contact the laboratory for the appropriate data.

After the data package review is completed, detailed review of sample results should begin. The data quality flags for each sample should be listed on the Form 1 equivalent or certificate of analysis. One form is required for each sample.

The data quality flags used for radiological sample results are shown below:

J	=	Indicates the analyte is present, but the reported value may not be accurate or precise because the associated quality assurance (QA)/QC was unacceptable. The result is considered "estimated."
R	=	Indicates the data is unusable. This flag is used when the result should not be used to support project decisions. The result is considered "rejected."
U	=	Indicates that the sample was analyzed, but the analyte was not detected above the stated concentration. The result is considered "undetected."

The following subqualifiers give further detail of the type and amount of qualification a given result has received.

D	=	Qualified because laboratory duplicate control limits were exceeded
S	=	Qualified because tracer recovery control limits were exceeded
C	=	Qualified because of instrument calibration problems
B	=	Qualified because of blank contamination problems
Q	=	Qualified for reasons not stated above—refer to the text of the report

4. References

Sample Preparation Standard Operating Procedures (SOPs) from Controls for Environmental Pollution.

QA—Standard Operating Procedure for Accepting Spike and Duplicate Results (CEP-QA-102, revised January 20, 1989).

Attachment 1

Radiochemical Data Completeness Checklist for Alpha Spectrometric Analyses of Soil and Water

1. ___ Case Narrative
   - ___ Abnormalities explained
   - ___ Matrix problems explained
   - ___ Instrument problems explained
   - ___ Improper collection, storage, preservation, container explained
   - ___ Hold times were met, explained if not met
   - ___ Signature of lab representative

2. ___ Quality Control (QC) Package

   A. ___ Calibrations Data Summary
      - ___ ID of each detector
      - ___ Dates of last efficiency check including spectra and/or channel by channel printout, certificates, and DPMs of check sources; counts obtained; count durations; and channels selected for regions of interest (ROIs)
      - ___ Proper channel numbers of isotopes of interest based on calibration of data of Pu, Am, and U standards
      - ___ Total memory (channels per detector)
      - ___ Energy range of the alpha detection system (keV)
      - ___ Gain (keV/channel) of the alpha detection system
      - ___ Dates of last background spectra including spectra and/or channel by channel printout; count durations; counts obtained; and channels selected for ROIs

   B. ___ Reagent Blanks Data Summary
      - ___ ID of each detector used
      - ___ Analyst initials
      - ___ Date reagent blanks were analyzed
      - ___ ID of samples analyzed with the reagent blanks
      - ___ Type of method blank used, minimum detectable activity (MDA) of method
      - ___ Volume of aliquot for reagent blanks

   C. ___ Replicate Sample Data Summary
      - ___ ID of each detector used
      - ___ Analyst initials
      - ___ Date sample and replicates were analyzed
      - ___ Sample IDs, values obtained for sample and replicates
      - ___ Count durations of sample and replicates
      - ___ Volume of aliquot for sample and replicates
      - ___ Calculated uncertainties and MDAs

D. ___ Matrix Spike/Matrix Spike Duplicate (MS/MSD) Data Summary

___ ID of each detector used
___ Analyst initials
___ Date MS/MSDs were analyzed
___ ID of MS/MSDs
___ Values obtained for MS/MSDs with uncertainty and MDA
___ True value of MS/MSDs with uncertainty
___ ID of samples analyzed with the MS/MSDs

E. ___ Chemical Recovery Data Summary

___ Efficiency factor provided for each detector used
___ ID of each detector used
___ Net counts obtained for each isotopic tracer used
___ Count duration
___ DPM value of each isotopic tracer
___ Calculated chemical recovery

3. ___ Sample Results Package

A. ___ Sample Summary Data

___ Printed report of results and counting errors for samples and reruns
___ MDA calculated for each isotopic analysis for samples with activity less than MDA

B. ___ Sample/MDA Raw Data

___ Background measurements including counts and count durations of samples and backgrounds taken during the same weekly time period
___ Date of analysis
___ Background CPM
___ Computer calculations sheet including sample IDs, detector IDs, isotopes of interest, counts obtained for samples, background counts obtained, isotopic tracer counts obtained, count durations, DPMs of tracer used, aliquots of sample and tracer, detector efficiency, chemical recovery, activities obtained for samples, uncertainties, and MDAs

4. Comments

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Radiological Data Review and Validation Guidelines, Radiochemical Determination of Ra-228

February 1993

Radiological Data Review and Validation Guidelines, Radiochemical Determination of Ra-228

1. Scope and Applicability

This document provides guidance for the review of laboratory data packages and the validation of results from environmental samples analyzed for Ra-228 from soil, biota, and water samples taken as part of the Kress Creek site Remedial Investigation/Feasibility Study (RI/FS).

2. Purpose

The purpose of review and validation is to determine the quality of each data point known and to flag each data point with a qualifier. In addition, data validation provides a review of laboratory quality control (QC) measures. It is assumed that field samplers and analytical laboratories have followed approved methods and adhere to good laboratory practices. This procedure provides guidelines for review and validation of radioanalytical data packages, and establishes criteria for applying appropriate data qualifiers to individual data points.

3. Criteria

This document provides criteria for evaluating Ra-228 data under the general categories of radiological data package completeness, holding times, calibration, blanks, lab and field replicates, matrix spike/matrix spike duplicate (MS/MSD) samples, chemical recovery, result verification, minimum detectable activities (MDAs), and overall data assessment. The criteria for each of these categories are discussed in detail in the following sections.

3.1 Radiological Data Package Completeness

Each data package should be checked for completeness prior to initiating data validation. The data validator should request the laboratory to submit any missing information. A complete data package consists of a case narrative, a QC data package, and a sample results data package. The contents of these packages are described below.

3.1.1 Case Narrative

The case narrative should include the following items:

- Cross reference of sample and laboratory numbers
- Problems encountered (reanalyses, broken sample containers, insufficient sample, excessive holding times, matrix problems, instrument problems, etc.)
- Descriptions of each out-of-control situation, corrective actions taken, and resolution
- Signature of the laboratory manager or designee

3.1.2 QC Package

The QC summary package should contain the following items:

- **Calibrations Data Summary.** This summary should include efficiency and background data used in the calibration and in the calibration verification process.

  The efficiency documentation should include detector and system identification numbers, date of calibration, standard reference isotopes and efficiencies (with self-absorption correction information), and certificates and activity values for standard calibration and reference sources.

  Background documentation should include results of background checks for each system, including counts in each region of interest and count duration.

- **Reagent Blanks Data Summary.** The reagent blanks data summary should include blank identification numbers, identification of samples analyzed with the blank, type of method blank used, MDA calculated for each blank, and raw data associated with the blank analysis, including detector identification, aliquot size, date of analysis, and analyst's initials.

- **Duplicate/Replicate Data Summary.** This summary includes data on precision including identification of detector used, analyst's initials, date of analysis for sample and duplicate/replicate, sample identification, activity results for sample and duplicate/replicate, count durations, and calculated uncertainties and MDAs for sample and duplicate/replicate.

- **MS/MSD Data Summary.** The MS/MSD summary should include date of MS/MSD analyses, detector identification, analyst's initials, MS/MSD identification, activity of each nuclide in the MS/MSD, and raw count data.

- **Chemical Recovery Data Summary.** This summary includes the activity of each tracer used, net counts for each isotope of interest, efficiency for each isotope of interest, count duration, and calculated chemical recovery for each sample analyzed.

3.1.3 Sample Results Package

The sample results data package should contain the following items:

- Summary page (Form 1 or certificate of analysis) showing the results for each sample (including blanks, duplicates/replicates, MS/MSDs, and reruns), including counting error and detection level for results reported as less than the MDA.

- Raw data backup for sample results including chain of custody (COC), sample identification, date of analysis (counting), detector identification, raw counts for each isotope of interest, background counts, tracer counts and tracer activity, chemical recovery, count duration, aliquot used, detector efficiency, sample activity, error, weight of sample on the planchet, self-absorption curve or table, and MDA results. The raw data should include any count data necessary to support calculated MDA values.

3.2 Holding Times

Sample holding time refers to the period from the time of sample collection to the time it is analyzed (counted). Sample collection dates appear on the COC record in the data package. This date should be compared with the analysis date on the raw data count sheet and also with the data summary form to make sure dates are consistent and that no sample mixup has occurred. Dates of receipt and signatures should be checked for continuity on the COC record. The sample holding time for water samples is 6 months (180 days). There is no standard holding time for soil samples.

Flag water samples with holding times greater than 180 days as "estimated" (J). Flag water samples with holding times greater than 270 days as "rejected" (R).

3.3 Calibration and Calibration Verification

For beta particle measurements, the counting system must be calibrated to obtain the counting efficiency for each of the target radionuclides with a standard traceable to the National Institute of Standards and Technology (NIST). Each counting system should have been calibrated with a beta standard representative of the target radionuclides within 1 year of the analysis date. The standard should have been prepared in the geometry and weight ranges expected to be encountered, and a self-absorption correction curve should have been constructed for each gas proportional counting system.

Verify that the standards used to prepare efficiency and calibration verification standards are unexpired and traceable to NIST. Flag the results "rejected" (R) if the standards are not traceable to NIST. Flag the results "estimated" (J) if they were obtained with expired NIST-traceable standards.

Verify that self-absorption corrections have been made to raw count data using the most recent self-absorption curve or table. Samples with mass greater than 300 mg (on a 2-in.-diameter planchet) should not be reported for Ra-228 analyses even with self-absorption corrections. Flag the data "rejected" (R) if self-absorption corrections were not made or if they were made incorrectly.

A calibration verification should have been performed daily with an independently prepared verification standard. The measured efficiency value should not be more than three standard deviations (3σ) from the value determined at the time of initial calibration. Results for samples analyzed after a verification beyond control limits and before the next adjacent acceptable verification should be flagged "estimated" (J). If the calibration verification was not performed, all results should be flagged "estimated" (J).

Background spectra should be collected on each detector at least monthly for beta counting systems. Flag results "estimated" (J) if the current background is outside of the range of $\pm 3\sigma$ of the background established at the time of calibration.

3.4 Reagent Blanks

Reagent blanks are used to determine background counts that result from environmental and reagent radiation sources and from monitor instrument background contributions. In addition, reagent blanks are used to determine statistical errors.

Reagent blanks should have been prepared and analyzed at a frequency of 5 percent of the samples. Flag results as "estimated" (J) if blanks were not run at a frequency of 5 percent or greater.

The results for blanks should be reported and summarized on a QC chart. Control limits should be set at 3σ from the mean. Flag sample results as "estimated" (J) if the blank for that group of samples falls outside of the 3σ control limit.

If radionuclides are detected in blanks, then sample results for the same radionuclides should be considered as positive only if they exceed 5 times the blank concentration. Samples that show positive results less than 5 times the blank values (for radionuclides detected in blanks) should be flagged "estimated" (J).

Compare the blank activities to the MDA. If blank results are consistently greater than the MDA, laboratory contamination may be indicated. If blank MDAs consistently exceed the detection limits stated in the Quality Assurance Project Plan (QAPjP), unacceptable counting times may be indicated. Use professional judgment to determine if the samples should be flagged "estimated" (J) for these conditions.

If reagent blank results are greater than 3σ below the mean background, then all nondetected results should be flagged "rejected" (R) and all positive results flagged "estimated" (J).

3.5 Duplicates/Replicates

Replicate analyses are defined as identifiable aliquots of the same sample taken through the entire procedure. They provide information on reproducibility of field sampling techniques and laboratory precision. One replicate should be analyzed for each batch of 10 samples.

Check the raw data and calculate the relative percent difference (RPD) for the sample and replicate using the following equation:

$$RPD = \frac{|S - R|}{(S + R)/2} \times 100$$

where:

S = sample result
R = replicate result

A control limit of 20 percent for water and 35 percent for soil samples for the RPD shall be used for sample results greater than 5 times the MDA.

A control limit of MDA for water samples and 2x MDA for soil samples shall be used for sample results less than 5 times the MDA, including an instance when only one of the sample/replicate pair results is less than 5 times the MDA.

If replicate results for a particular radionuclide fall outside the appropriate control windows, qualify the results for that radionuclide in all associated samples of the same matrix as "estimated" (J).

3.6 Matrix Spike/Matrix Spike Duplicate Samples

MS/MSD samples are derived from a matrix that has been fortified with a known concentration of a specific isotope. These samples should be analyzed at a frequency of 1 (MS/MSD pair) in 20 samples or 1 set per batch, whichever is most frequent. The results of the analysis of these samples provide an indication of both laboratory accuracy and precision.

Accuracy for the set of samples will be evaluated by comparing the measured concentration with the known (spike) concentration in the MS/MSD sample. The comparisons will be made using the counting error (sigma) term associated with each measured concentration as the evaluation criteria. If the known value falls within three sigma (3σ) of the measured value, the accuracy associated with the MS/MSD is acceptable.

If the results of the MS/MSD fall outside of the 3σ range, the data reviewer will use professional judgment in conjunction with other QC criteria to determine if the data should be qualified as estimated or rejected.

Precision is a measurement of the reproducibility of analytical results under a standard set of conditions. It can be viewed as a measure of the magnitude of errors occurring in an analytical procedure. RPD is the method most commonly used to estimate precision when using MS/MSD samples in a set of analyses. RPD is defined in Section 3.5, Duplicates/Replicates.

No action is taken based on MS/MSD data alone to qualify a batch of samples. Since precision cannot be measured for a pair of samples that is below the quantitation limits of the method, duplicate samples are fortified and then evaluated.

The data reviewer should use informed professional judgment in conjunction with other QC criteria and determine the necessity for further data qualification. However, when MS/MSD data are interpreted, the following will be used as guidelines:

- A control limit of $\pm 3\sigma$ will be used in conjunction with the calculated RPD when the known value of the spikes is less than 20 times the MDA. The data reviewer may flag results as "estimated" (J) if professional judgment dictates.

- A control limit of $\pm 2\sigma$ will be used when known spike values are greater than 20 times the MDA. The data reviewer may flag the results as "estimated" (J) if justified by other QC parameters and the reviewer's professional judgment.

3.7 Recovery Factors

A sample of acidified water spiked with a known amount of Ra-228 is used to determine chemical recovery (or yield). The chemical recovery is calculated on the basis of the net count rate [counts per minute (CPM)] obtained from the Ra-228 spike, the actual activity or disintegration rate [disintegrations per minute (DPM)] contained in the spike solution, and the instrument efficiency. The above terms are related in the following formula:

$$\text{CPM tracer found/DPM of tracer added} = \text{eff} \times \text{chem recovery}$$

Chemical recoveries for beta particle analyses should be greater than 20 percent, but less than 105 percent.

Flag results "rejected" (R) if these criteria are not met and the sample activity is below the MDA.

For Ra-228 analyses, flag results "estimated" (J) if the chemical recovery is greater than 10 percent, but less than 20 percent, and the sample activity is greater than the MDA. Flag these results "rejected" (R) if the chemical recovery is less than 10 percent and the sample activity is greater than the MDA.

3.8 Verification of Sample Result and MDA Calculations

Manual calculations should be performed to verify sample result and MDA calculations performed by the laboratory. Errors or discrepancies should be addressed in the comments section of the data validation report. The following formulas should be used for calculating sample concentration, counting error, and MDA.

3.8.1 Sample Result

$$A = \frac{CPM_G - Bkg}{(2.22)(E)(Vol)(R)(1-e^{-0.1131t_1})(e^{-0.1131t_2})}$$

where:

A =	sample concentration (pCi/l or pCi/g)
CPM_G =	gross sample count rate
Bkg =	background count rate
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
Vol =	sample mass or volume (grams for solids, liters for water)
R =	chemical recovery
t_1 =	Ac-228 ingrowth time from Ra-228 in hours
t_2 =	Ac-228 decay time in hours

3.8.2 Counting Error

$$ER = \frac{2.0\left[\frac{CPM_G}{T_S} + \frac{Bkg}{T_{Bkg}}\right]^{0.5}}{(2.22)(E)(Vol)(R)(1-e^{-0.1131t_1})(e^{-0.1131t_2})}$$

where:

ER =	2σ counting error (pCi/l or pCi/g)
CPM_G =	gross sample count rate (CPM)
T_S =	count time for sample (min)
Bkg =	background count rate (CPM)
T_{Bkg} =	count time for background (min)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
Vol =	sample mass or volume (grams for solids, liters for water)
R =	chemical recovery
t_1 =	Ac-228 ingrowth time from Ra-228 in hours
t_2 =	Ac-228 decay time in hours

3.8.3 Minimum Detectable Activity

$$MDA = \frac{4.66\ (Bkg/T)^{0.5}}{(2.22)(E)(Vol)(R)(1-e^{-0.1131t_1})(e^{-0.1131t_2})}$$

where:

MDA =	minimum detectable activity
Bkg =	background count rate (CPM)
T =	count time (min)
2.22 =	conversion factor (2.22 DPM = 1 pCi)
E =	counting efficiency
R =	chemical recovery
t_1 =	Ac-228 ingrowth time from Ra-228 in hours
t_2 =	Ac-228 decay time in hours

Verify that the calculations for activity, uncertainty, and MDA for each sample are correct. Address any errors or discrepancies in the comment section of the report.

3.9 Overall Data Assessment

As part of the overall data assessment, the results of the data validation process will be documented on the appropriate summary forms. The first step in the validation process is to review the case narrative, QC data package, and sample results data package using the checklist shown in Attachment 1. If the data packages are not complete, the data validator must contact the laboratory for the appropriate data.

After the data package review is completed, detailed review of sample results should begin. The data quality flags for each sample should be listed on the Form 1 equivalent or certificate of analysis. One form is required for each sample.

The data quality flags used for radiological sample results are shown below:

J	=	Indicates the analyte is present, but the reported value may not be accurate or precise because the associated QA/QC was unacceptable. The result is considered "estimated."
R	=	Indicates the data is unusable. This flag is used when the result should not be used to support project decisions. The result is considered "rejected."
U	=	Indicates that the sample was analyzed, but the analyte was not detected above the stated concentration. The result is considered "undetected."

The following subqualifiers give further detail of the type and amount of qualification a given result has received.

D	=	Qualified because laboratory duplicate control limits were exceeded.
S	=	Qualified because recovery control limits were exceeded.
C	=	Qualified because of instrument calibration problems.

B = Qualified because of blank contamination problems.

Q = Qualified for reasons not stated above—refer to the text of the report.

4. References

Sample Preparation Standard Operating Procedures (SOPs) from Controls for Environmental Pollution.

QA—Standard Operating Procedure for Accepting Spike and Duplicate Results (CEP-QA-102, revised January 20, 1989).

Attachment 1

Radiochemical Data Completeness Checklist for Ra-228 Analyses of Soil and Water

1. ___ Case Narrative
   - ___ Abnormalities explained
   - ___ Matrix problems explained
   - ___ Instrument problems explained
   - ___ Improper collection, storage, preservation, container explained
   - ___ Hold times were met, explained if not met
   - ___ Signature of lab representative

2. ___ Quality Control (QC) Package

   A. ___ Calibrations Data Summary
      - ___ ID of each detector
      - ___ Dates and results of last efficiency check, certificates, and DPMs of check sources; counts obtained; and count durations
      - ___ Self-absorption curve or table
      - ___ Dates and results of last background count including count durations and counts obtained

   B. ___ Reagent Blanks Data Summary
      - ___ ID of each counting system used
      - ___ Analyst initials
      - ___ Date reagent blanks were analyzed
      - ___ ID of samples analyzed with the reagent blanks
      - ___ Type of method blank used, minimum detectable activity (MDA) of method
      - ___ Volume of aliquot for reagent blanks

   C. ___ Replicate Sample Data Summary
      - ___ ID of each counting system used
      - ___ Analyst initials
      - ___ Date sample and replicates were analyzed
      - ___ Sample IDs, values obtained for sample and replicates
      - ___ Count durations of sample and replicates
      - ___ Volume of aliquot for sample and replicates
      - ___ Calculated uncertainties and MDAs

   D. ___ Matrix Spike/Matrix Spike Duplicate (MS/MSD) Data Summary
      - ___ ID of each counting system used
      - ___ Analyst initials
      - ___ Date MS/MSDs were analyzed
      - ___ ID of MS/MSDs
      - ___ Values obtained for MS/MSDs with uncertainty and MDA
      - ___ True value of MS/MSDs with uncertainty
      - ___ ID of samples analyzed with the MS/MSDs

E. ___ Chemical Recovery Data Summary

- ___ Net counts obtained for each isotopic tracer used
- ___ Count duration
- ___ DPM value of each isotopic tracer
- ___ Calculated chemical recovery

3. ___ Sample Results Package

A. ___ Sample Summary Data

- ___ Printed report of results and counting errors for samples and reruns
- ___ MDA calculated for each analysis for samples with activity less than MDA

B. ___ Sample/MDA Raw Data

- ___ Background measurements including counts and count durations of samples and backgrounds taken during the same weekly time period
- ___ Date of analysis
- ___ Background CPM
- ___ Computer calculations sheet including sample IDs, counting system IDs, counts obtained for samples, background counts obtained, tracer counts obtained, count durations, DPMs of tracer used, aliquots of sample and tracer, counting system efficiency, chemical recovery, activities obtained for samples, uncertainties, and MDAs

Appendix E

Waste Management Plan

CONTENTS

FIGURES

Appendix E
Waste Management Plan

1. Purpose

This plan outlines the methods used for the management of investigation-derived waste that is generated during Remedial Investigations (RIs) conducted at the Kress Creek site, along with the management of sample residuals returned from the laboratories. The principal concepts of this plan were derived from the source documents listed in the reference section of this report.

This plan contains definitions and identifies waste categories and classification methods, packaging requirements, and preferred management options. The approach outlined in this plan emphasizes the following objectives:

- Management of investigation-derived waste in a manner that is protective of human health and the environment.
- Minimization of investigation-derived waste generation, thereby reducing costs and use of limited permitted storage and disposal capacity.
- Compliance, to the extent practical, with federal and state requirements that are legally applicable or relevant and appropriate requirements (ARARs).

2. Definitions

Area of Concern (AOC): The area delineated by the areal extent (or boundary) of potential contamination on a project site. This boundary may contain varying concentrations and types of hazardous substances and may contain uncontaminated areas. For the purpose of this plan, the AOC will be considered represented at the site boundaries within the Kress Creek site.

Environmental Protection Agency (EPA) "Contained-In" Policy: Requires any mixture of a non-solid waste (environmental media) and a Resource Conservation and Recovery Act (RCRA)-listed hazardous waste to be managed as a hazardous waste, as long as the material contains the listed hazardous wastes above health-based standards.

Field Staging Area (FSA): An area within the project site where drums and other containers or investigation-derived waste are stored until the site investigation activities are completed or a final disposal option is selected in a Record of Decision. This area will be posted as the FSA and will be checked for leaking containers weekly during field activities. This area may be used until appropriate disposition of all containers. Additional empty drums, overpack, and absorbent materials will be kept at the FSA in the event of a leak or spill. The FSA is not considered a RCRA 90-day storage area.

Hazardous Constituent: Those substances listed in 40 CFR Part 261, Appendix VIII.

Hazardous Substances: For purposes of this plan, shall have the meaning set forth by Section 101(14) of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), 42 U.S.C. S 9601(14).

Investigation-Derived Waste: Discarded materials resulting from site investigation activities such as decontamination that, in present form, possess no inherent value or additional usefulness without treatment.

Such waste may be solid, semi-solid, liquid, or gaseous material that may or may not be hazardous as defined in 40 CFR Part 261; radioactive due to the presence of radionuclides regulated by the Atomic Energy Act (AEA) of 1954, as amended; or mixed, a waste that contains both radioactive and hazardous components. Investigation-derived waste includes materials such as used Personal Protective Equipment (PPE), decontamination fluids (wash and rinse), drilling muds and cuttings, pumped monitoring well fluids, purge water, soil, and other materials from collection of samples and spill-contaminated material.

Land Disposal: Means placement in or on the land and includes, but is not limited to, placement in a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome formation, underground mine or cave, or concrete vault or bunker intended for disposal purposes.

Land Disposal Restrictions (LDRs): Restrictions that prohibit the land disposal of certain RCRA hazardous wastes unless specified treatment standards are met. The EPA has established standards for specific hazardous wastes that are protective of human health and the environment when the wastes are land disposed. LDRs apply to waste management activities under RCRA and the Safe Drinking Water Act (SDWA), which controls underground injection of hazardous wastes in deep wells. Land disposal includes placement in a landfill, surface impoundment, waste pile, injection well, land treatment facility, salt dome or salt bed formation, underground mine or cave, or concrete vault or bunker.

Radioactive Waste: Waste that contains radioactivity above background or reference levels.

Mixed Waste: Material that has been classified as hazardous and/or polychlorinated biphenyl (PCB) waste, and is also classified as radioactive.

Movement (Non-Placement): An activity that consists of moving soil, whether excavated or surface soil, along with RCRA hazardous wastes and CERCLA hazardous constituents contained in the soil, within the site to consolidate the material within the site. Note that movement of soil with CERCLA constituents or radioactive constituents, that do not contain RCRA hazardous waste, would not trigger RCRA-LDR even if moved outside the site.

Placement: An activity that consists of moving soil contaminated with RCRA hazardous wastes offsite or outside the AOC.

Wastewater: Liquid waste consisting primarily of water without other liquid phases present that results from groundwater well installation, development, and sampling activities or from the cleaning of well installation equipment.

3. General Management Approach

The intent of this plan is to return all of the investigation-derived waste (excluding PPE, decontamination liquids, and biota residue) generated from sampling activities back to the original source, thereby reducing the volume of waste to be containerized and stored. This approach minimizes investigation-derived waste and does not add a greater threat to human health and the environment than existed prior to the investigation. Returning the investigation-derived waste to the original source will also allow the investigation-derived waste to be addressed in a manner consistent with the final remedy for the site.

Residual soils/sediments from hand-augering will be returned to the borehole from which they originated. Additional clean fill (sand) will be used to fill the remaining portions of the boreholes, as applicable.

Wastewater, PPE, and biota residue generated during decontamination operations and sampling activities will be containerized, centralized, and/or managed according to this plan.

4. Area of Concern

Prior to development of this plan, the concept of returning the residual soil and sediments back to the original borehole was evaluated regarding compliance with applicable regulations. The most significant ARAR considered included the LDRs under RCRA. For LDRs to be applicable, the action must constitute "placement" of a restricted RCRA hazardous waste in a land disposal unit. To clarify whether "placement" occurs, the concept of AOC has been adopted.

Investigation-derived waste that is generated, moved, consolidated, stored, or redeposited within the boundaries of the AOC will not constitute "placement" or trigger LDRs (EPA, 1992). However, "placement" will occur as a result of either of the two following activities: (1) investigation-derived waste is consolidated from different AOCs into a single AOC and redeposited, and (2) investigation-derived waste is moved outside of an AOC (for example, for treatment or storage) and returned to the same or a different AOC. However, for the Kress Creek site, due to the type of contaminants and the type of investigation-derived waste to be generated (mainly PPE and decontamination water), it is not anticipated that listed RCRA hazardous wastes will be found. Therefore, RCRA "contained-in" requirements, RCRA LDRs, land disposal, and placement requirements will not apply.

5. Waste Handling, Segregation, and Packaging

Investigation-derived waste generated will be containerized for characterization and classification. PPE and fish residuals will be composited into open-top, 55-gal steel 17C Department of Transportation (DOT)-approved drums lined with a minimum 25-mil plastic liner. Wastewater generated from decontamination activities will be collected in 55-gal drums as well. Such containers will be truck transportable, forklift transportable, or both.

Waste containers will be securely closed when they are filled, cleaned, and labeled. All labeling will include date, where the investigation-derived waste was generated, waste type, and any field observations. Labels will be completed with permanent markers and will be attached when the drum is full or when drilling activities are complete. An example of the type of label to be used is shown in Figure 5-1.

6. Waste Transportation and Storage

Investigation-derived waste generated during field activities will be composited into drums at the AOC. Wastewater from decontamination activities will be sampled and analyzed for Target Analyte List (TAL) and radionuclides (U-234, U-235, U-238, Th-227, Th-228, Th-230, Th-232, Ra-226, and Ra-228). It is estimated that two samples will be sufficient to characterize the decontamination water. No quality assurance/quality control (QA/QC) samples will accompany these samples. No fish residual samples will be collected.

Once the drums and containers are securely sealed and labeled, they will be moved to the FSA. Waste to be transported during sampling activities (primarily PPE and plastic) will be scanned for direct (total) radioactive contamination prior to transport to the FSA. Waste materials will be containerized at the job site after the radiological survey is conducted. If the results of the direct contamination measurements are less than the direct contamination criteria specified in Procedure RP-07 (Appendix C), the material will be considered to be non-radioactive (containing radioactivity less than 2,000 pCi/g) for purposes of compliance with DOT regulations for shipment of radioactive material. The FSA will be designated and/or provided by EPA. At the FSA, the material will be unloaded onto pallets. Each pallet will contain a maximum of four drums and will be placed in rows with at least 2 ft of aisle space. Drums will be positioned on the pallets such that the container labels are visible from the aisle.

SWMU # _____

BORING #(s) _____

_____

WASTE TYPE _____

_____

DATE GENERATED _____

FIELD OBSERVATIONS _____

_____

Figure 1
Waste Container Label

The investigation-derived waste will be temporarily stored at the FSA, pending analytical results of samples collected. Following review of environmental and investigation-derived waste sample results and comparison of this data to regulatory levels, disposal options and/or additional classification criteria will be determined by EPA.

7. Waste Classification Criteria

If needed for final disposal, EPA will classify the investigation-derived waste into two categories:

1. Non-Hazardous Wastes
2. Radiological Waste

7.1 Radiologically Contaminated Wastes

Radiologically contaminated wastes apply to waste that contains radioactivity above background levels.

7.2 Non-Hazardous Wastes

Investigation-derived waste that does not meet the classification requirements of RCRA hazardous wastes, mixed wastes, or radiologically contaminated waste will be classified as a non-hazardous waste.

8. Waste Classification

Investigation-derived waste will be classified on the basis of environmental sample results for determining disposal options for PPE and using investigation-derived waste sample results for determining disposal options for decontamination fluids. If sample results are not conclusive for disposal determination for decontamination fluids, then additional characterization may be required.

9. Sample Management

Laboratory residuals will be shipped from the laboratory to an EPA Region V shipping/receiving warehouse located in Chicago. The address for the return of the sample residuals will be included in the chain-of-custody paperwork.

Samples will be shipped in accordance with the "limited quantity" radioactive shipping regulations presented in 49 CFR 173 and as described in Appendix B (Sample Documentation and Packing and Shipping Instructions) of this document. Samples will be required to be shipped under the limited quantity rules unless the laboratory can document that levels contained in the cooler will not exceed the 2,000 pCi/g limit specified in 49 CFR 173.

The laboratories will send the sample residuals to the EPA warehouse following completion of all analysis for the site. Once samples are received at the EPA warehouse, they will be transported to the FSA for storage until disposal.

10. Disposal Options

Wastewater, PPE, and fish remains are the types of investigation-derived waste that will be generated during the site investigation activities. The approach recommended in this plan is intended to minimize investigation-derived waste generation and pursue management options consistent with the final remedy selected for the site.

10.1 Wastewater

Wastewater generated from decontamination activities will be temporarily stored at the FSA. Samples collected during the RI for decontamination fluid characterization will be evaluated regarding acceptability for disposal at the West Chicago Sewage Treatment Plant. If the investigation-derived waste wastewater is at a level that cannot be disposed of at the Sewage Treatment Plant, then the investigation-derived waste will be stored in accordance with applicable regulations until discharge limits can be achieved through treatment.

10.2 PPE

If analytical results for environmental samples indicate that contamination detected is at levels that pose no harm to human health or the environment (as determined by EPA), then the PPE would be disposed of in a landfill for non-hazardous waste. If, however, contamination is detected at levels that may pose risks to human health and the environment through transfer from the PPE, then the PPE will be stored at the FSA until final disposal is determined.

10.3 Fish

Fish remains derived from fish tissue collection will be stored at the FSA. Analytical results from the fish samples will be used to evaluate this waste. If this evaluation determines that constituents are at concentrations that would not affect human health and the environment (as determined by EPA), then the investigation-derived waste would be disposed of in a landfill for non-hazardous waste. If concentrations are such that onsite disposal would not be permitted, then this investigation-derived waste will be placed in interim storage until final disposal is determined by EPA.

Appendix F
Additional NAREL Analytical Procedures

7-28-92
Draft

DRAFT

RADIUM-226
BY THE DE-EMANATION PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine the concentration of radium-226 in samples that have been especially prepared for analysis by this procedure (see Ra-01, Radium-226 and Radium-228, Solid Samples and Ra-03, The Preparation of Water Samples for the Radiochemical Determination of Radium-226 by the De-emanation Procedures).

1.2 This method is specific for measuring the concentration of the radium-226 isotope of radium.

1.3 The method is applicable for any sample matrix containing radium-226 that can be solubilized.

1.4 Data quality objectives are identified during project scoping and development of sampling and analysis plans. DQOs are established to ensure that the data collected are sufficient and of adequate quality for their intended uses. Data collected and analyzed in conformance with the DQO process described in this document can be used in assessing the uncertainty associated with decisions related to remedial response.

2.0 SUMMARY OF METHOD

After sample preparation has been completed and individual samples sealed in disposable glass storage tubes (Ra-01 and Ra-03), radon-222 ingrowth proceeds for at least 21 days. The storage tubes are then connected to a gas manifold and the accumulated radon-222 is quantitatively transferred to evacuated Lucas alpha scintillation counting cells (see Reference 16.8). The alpha activity in the cells is measured after five hours ingrowth of radon-222 progeny.

3.0 DEFINITIONS

3.1 ALARA: Acronym for "As Low as Reasonably Achievable." A basic concept of radiation protection that specifies that radioactive discharges from nuclear plants and radiation

exposure to personnel be kept as far below regulation limits as feasible.

3.2 De-emanation: The process by which a gaseous product of radioactive disintegration (Rn-222 from Ra-226) is transferred into a counting cell by sparging with an inert gas (nitrogen).

3.3 Duplicate: Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures.

3.4 EMSL-LV: Acronym for Environmental Measurement Support Laboratory-Las Vegas.

3.5 IAEA: Acronym for International Atomic Energy Agency.

3.6 Laboratory reagent blank (LRB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if interferences are present in the laboratory environment, reagents, or apparatus.

3.7 MSDS: Acronym for Material Safety Data Sheets.

3.8 NIST: Acronym for National Institute of Standards and Technology.

3.9 R value: Ratio of observed activity divided by the actual amount of added activity.

3.10 SAP: Acronym for Sampling and Analysis Plan; rigid guidelines regarding number of samples requested, method of collection, analysis deadline, etc.

3.11 Spiked Sample: An aliquot of a sample which is spiked with a known concentration of the analyte of interest.

3.12 WHO: Acronym for World Health Organization.

4.0 INTERFERENCES

There are no expected interferences.

5.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this procedure has not been precisely defined. Each chemical compound should be treated as a potential health hazard and exposure to these compounds should be ALARA. The laboratory is responsible for maintaining a current file of regulations regarding safe handling of the chemicals specified in this procedure. A reference file of MSDS should be made available to all personnel involved in the chemical analysis. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

6.0 EQUIPMENT AND SUPPLIES

6.1 Pyrex brand glass tubing, 15-mm i.d., 45-cm lengths.

6.2 Spring Clip Stopcock, No. 0E317, Becton, Dickinson and Company, Rutherford, NJ 07070.

6.3 Male luer-lok adapter, No. 3113 (used to attach hypodermic needle to stopcock), Becton, Dickinson and Company, Rutherford, NJ 07070.

6.4 Vinyl tubing, 1.27 cm (½ in.) i.d. and 1.6 cm (5/8 in.) o.d.

6.5 18- and 26-gauge hypodermic needles.

6.6 Serum stoppers.

6.7 Magnesium Chlorate drying tubes - 10 cm long, 15 mm i.d., glass tube containing anhydrous magnesium chlorate, $Mg(ClO_4)_2$, packed between glass wool and sealed at each end with a serum stopper.

6.8 Glass-sealing torch.

6.9 Vacuum pump assembly.

6.10 Lucas alpha scintillation cells (see Figure 3).

6.11 Photomultiplier tube assembly and associated counter electronics.

7.0 REAGENTS AND STANDARDS

7.1 Acetone, reagent grade (CAS: 67-64-1).

7.2 Ethyl alcohol, 95 percent, reagent grade (CAS: 64-17-5).

7.3 Vacuum grease (CAS: 8001-79-4).

7.4 Tank of nitrogen gas (CAS: 7727-37-9).

7.5 RBS-35 cleaning solution (CAS: N/A).

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

All samples must have been collected using the appropriate SAP. Prior to the collection of an aqueous sample, consideration should be given to the type of data required, (i.e., dissolved or total recoverable), so that appropriate preservation and pretreatment steps can be followed.

9.0 QUALITY CONTROL

9.1 All quality control data should be maintained and available for easy reference or inspection.

9.2 Employ a minimum of one blank per sample batch to determine if contamination is occurring.

9.3 Analyze one duplicate sample for every 10 samples. A duplicate sample is a sample split into two identical portions, each given the same treatment throughout the sample preparation and analytical process.

9.4 One spiked sample or standard reference sample shall be analyzed for every 20 samples (5%) to ensure that correct procedures are being followed and that all equipment is operating properly.

9.5 Instrument calibration checks and backgrounds will be performed routinely on all detectors used for this procedure.

10.0 CALIBRATION AND STANDARDIZATION

The calibration constant is determined by sealing approximately 5-10 pCi/ml of radium-226, accurately known and traceable to the National Institute of Standards and

transferred to a Lucas cell and counted. The calibration constant, E, is then calculated by substituting the counting data into Equation 1 and solving for E in the denominator. Each Lucas cell is individually calibrated.

11.0 PROCEDURE

11.1 Clean 45-cm lengths of glass tubing in RBS-35 cleaning solution, rinse in alcohol, and dry.

11.2 Flame seal one end of the section of glass tubing.

11.3 Starting approximately 10 cm from the open end of a section of the tubing, soften the tubing in a flame and form a constriction approximately 0.5 cm long and 0.6 cm diameter.

11.4 Transfer the prepared sample to the de-emanation tube using distilled water as wash (see Ra-01, Radium-226 and Radium-228, Solid Samples and Ra-03, The Preparation of Water Samples for the Radiochemical Determination of Radium-226 by the De-emanation Procedures). Fill to within 3 to 5 cm of the constriction.

11.5 Flame seal the tube at constriction without overheating.

11.6 Store sample-containing sealed tubes for at least 21 days.

11.7 Soak one end of each of two 9-cm long by 1.27-cm diameter pieces of vinyl tubing in acetone until slight swelling occurs.

11.8 Place a serum stopper in the solvent treated end of each piece of tubing so that the tubing surrounds the sleeve of the stopper.

11.9 Insert 26-gauge hypodermic needles in the rubber serum stoppers.

11.10 Soak the other ends of the vinyl tubing in acetone until swelling is evident.

11.11 Slip the solvent-treated tubing over the sealed end of the sample tube. Leave the needle inserted in the stopper to relieve pressure as tubing dries.

11.12 Remove needles from stoppers after 24 hours and apply rubber adhesive to the stopper-sample-tube joints. (Rubber and Gasket adhesive, Scotch-Grip Brand, 3M Corp., Adhesive, Coatings and Sealers Division, St. Paul, MN 55144.)

11.13 Assemble cleaned valves for the cell evacuation assembly using Dow Corning vacuum grease. Coat the male luer-lok tip with Crown dry-film lubricant (a product of Crown Industrial Products Co., Hebron, IL 60034) and force the male luer-lok adapter and stopcock firmly together using pliers. Do not interchange parts from different stopcocks.

11.14 Assemble the radon transfer manifold on a bench top as shown in the cell evacuation illustration, Figure 1. (A 1000 minute background count is obtained for the scintillation cell before it is placed on the evacuation assembly.) All needle to rubber and rubber to glass joints are sealed with rubber adhesive, except for the needle/vacuum-tube joint.

11.15 Insert the needle from the vacuum line into lower stopper of the vacuum tube. Insert the needle attached to the vacuum gauge into the same stopper (not shown in Figure 1). Open the two valves and pump a vacuum on the system for ten minutes.

11.16 Remove the vacuum line needle and check the vacuum gauge over a ten minute period for possible leaks. Most leaks occur at the adapter-stopcock joint, which may require further tightening. (The stopcock is initially closed during the leak test to check the integrity of the adapter-stopcock joint. If no leak is detected, open the stopcock and check for leaks occurring in the system between the stopcock and cell.)

11.17 Carefully hang the radon transfer manifold vertically on a rack, taking particular care not to dislodge any needles.

11.18 Reapply vacuum to the tube and then close upper valve.

11.19 Insert the needle from the vacuum line into the upper stopper of the sample tube, being careful not to break the tip of the tube with the needle.

11.20 Repeat step 11.19 at the other end of the sample tube.

11.21 Allow ten minutes to elapse in order to check for air leaks at both ends of the sample tube. A leak is indicated by partial refilling of the collapsed vinyl tubing.

11.22 Remove the vacuum tube from the assembly and replace it with the sample tube as shown in the purging illustration (Figure 2). Seal the needle to the stopper with rubber adhesive.

11.23 Break both top and bottom tips of the sample tube using long-nose pliers. This should be done very carefully to prevent the broken glass from puncturing the vinyl tubing.

11.24 Record the time.

11.25 Apply rubber adhesive to the vinyl tubing in the areas of the broken glass tips as a precaution in case any punctures did occur.

11.26 Cautiously open the top valve to permit pressure equalization between sample tube and Lucas cell, being careful not to draw up sample liquid into drying tube and cell.

11.27 Close upper valve.

11.28 Start the nitrogen purge with a flow rate barely detectable through the small hole (about 3 mm) in the nitrogen delivery tube.

11.29 Flush the valve, and, with the valve closed, insert the needle on the nitrogen delivery tube into the lower stopper of the sample tube.

11.30 Cautiously open the lower valve to control the flow of nitrogen into the sample tube. The bubble rate should be between 15 and 45 per minute.

11.31 For a period of 30 minutes, open the upper valve briefly every 2 minutes in order to equalize the pressure between the Lucas cell and the sample tube.

11.32 After 30 minutes, open upper valve completely and, if necessary, open the lower valve further to increase bubble rate.

11.33 When the nitrogen flow has nearly stopped and the vinyl tubing has expanded to its original shape (about 60 to 90 minutes), simultaneously remove the needles from the stopper on the Lucas cell and from the lower stopper on the sample tube.

11.34 After the tips are broken, the Lucas cell is stored for 3 hours to allow for the ingrowth of radon-222 progeny.

11.35 Count the sample for 1000 minutes. Immediately following the count, flush the scintillation cell three times by pulling a vacuum and refilling it with nitrogen. Store the cells filled with nitrogen.

11.36 Dispose of the needles and drying tube.

11.37 Clean the upper stopcock after each de-emanation by the following steps: a) disassemble valve; b) submerge and agitate in acetone and then in hot soapy water; c) rinse with water, then alcohol followed by acetone, and d) air dry at room temperature.

12.0 DATA ANALYSIS AND CALCULATION

Calculate the concentration, Z, of radium-226 in picocuries per liter or per gram as follows:

$$Z = \frac{C_1 - C_B}{2.22 \times E \times V} \times \frac{\lambda}{1 - e^{-\lambda t_1}} \times \frac{1}{1 - e^{-\lambda t_2}} \times \frac{\lambda t_3}{1 - e^{-\lambda t_3}}$$

where

λ = decay constant for radon-222 ($t_{\frac{1}{2}}$ = 3.825 days),

t_1 = time interval allowed for ingrowth of radon from radium,

t_2 = time interval between de-emanation and counting,

t_3 = counting time,

C_1 = observed count rate of sample,

C_B = background count rate,

E = calibration constant of the scintillation cell in counts per unit time per dpm of radon plus decay products,

V = sample volume (liters) or sample mass (grams), and

2.22 = dpm/pCi.

Note - All decay corrections can be obtained directly or indirectly from Table 1.

Sample Calculation

Assume the following data:

t_1 = 13 d. 14 h. 6 m.

t_2 = 5 h. 15 m.

t_3 = 16 h. 30 m.

C_1 = 199.2 counts/hour.

C_B = 9.3 counts/hour.

E = 151.1 counts/hour/dpm. and

V = 1.0 liter.

From Table 1.

$1-e^{-\lambda t_1}$ = 1. - 0.09484 x 0.89969 x 0.99925 = 0.91474.

$e^{-\lambda t_2}$ = 0.96295 x 0.99811 = 0.96113. and

$\lambda t_3/(1-e^{-\lambda t_3})$ = 1.06358 (by linear interpolation).

From these data.

$$Ra\ (pCi/L) = \frac{(199.2 - 9.3)}{(151.1)\ (2.22)\ (1.0)} \times \frac{1}{0.91474} \times \frac{1}{0.96113} \times 1.06358$$

$$= 0.685.$$

Calculate the lower limit of detection (LLD) in picocuries per liter as follows:

$$LLD = \frac{4.66\sqrt{C_B}}{2.22 \cdot E) \cdot V \cdot T}$$

where:

C_B = background count rate,

T = counting time,

E = calibration constant of the scintillation cell in counts per unit time (hour) per disintegrations per minute (dpm) of radon plus decay products, and

V = sample volume, liters.

This LLD is valid if the background counting time is approximately equal to the sample counting time.

13.0 METHOD PERFORMANCE

13.1 Accuracy

A total of 84 sets of data pairs were used for determining the accuracy of the analytical procedure for radium-226 in water (references 11.5, 11.6, and 11.7). Comparisons were made with 78 EMSL-LV reference values, 2 WHO reference values, and 4 IAEA reference values between 1970 and 1986. The average R value (measured concentration divided by the reference value) was 1.016.

Appropriate testing of the method for measuring radium-226 in solid samples has not been accomplished.

13.2 Precision

A total of 52 sets of triplicate results were used for calculating the precision of the method for measuring radium-226 in water (references 11.6 and 11.7). Individual coefficients of variation, as defined in reference 11.7, were calculated and used as a measure of analytical precision. An average value of 9.0 percent was determined for these 52 sets of measurements.

Appropriate testing of the method for measuring radium-226 in solid samples has not been accomplished.

14.0 POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

15.0 WASTE MANAGEMENT

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15.2 Waste streams generated by this procedure include the following: needles, magnesium perchlorate, 0.1 N hydrochloric acid (25 mL), acetone, and alconol.

16.0 REFERENCES

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17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

TABLE I

A. Decay of Radon (in minutes, hours, and days)
B. Growth of Radon from Radium (in days).
C. Multiplicative Factor for Correction of Radon Activity for Decay during Counting (in hours)

(Based on 3.825 days as half-life of radon)

Time	A. $e^{-\lambda t}$			B. $1-e^{-\lambda t}$	C. $1-e^{-\lambda t}$
	Minutes	Hours	Days	Days	Hours
0	1.000.00	1.000.00	1.000.00	0.000.00	1.000.00
1	0.999,87	0.992,48	0.834,27	0.165,73	1.003,72
2	0.999,75	0.985,01	0.696.00	0.304.00	1.007,54
3	0.999,62	0.997,60	0.580,65	0.419,35	1.011,39
4	0.999,59	0.970,25	0.484,42	0.515,58	1.015,16
5	0.999,37	0.962,95	0.404,14	0.595,86	1.018,98
6	0.999,25	0.955,71	0.337,16	0.662,84	1.022,83
7	0.999,12	0.948,52	0.281,28	0.718,72	1.026,65
8	0.998,99	0.941,39	0.234,66	0.765,34	1.030,51
9	0.998,87	0.934,31	0.195,77	0.804,23	1.034,36
10	0.998,74	0.927,27	0.163,33	0.836,67	1.038,23
11	0.998,62	0.920,31	0.136,26	0.863,74	1.042,10
12	0.998,49	0.913,38	0.113,68	0.886,32	1.045,97
13	0.998,37	0.906,51	0.094,84	0.905,16	1.049,88
14	0.998,24	0.899,69	0.079,12	0.920,88	1.053,79
15	0.998,11	0.892,93	0.066,01	0.933,99	1.057,69
16	0.997,99	0.886,21	0.055,07	0.944,93	1.061,61
17	0.997,86	0.879,55	0.045,94	0.954,06	1.065,54
18	0.997,74	0.872,93	0.038,33	0.961,67	1.069,49
19	0.997,61	0.866,36	0.031,98	0.968,02	1.073,44
20	0.997,49	0.859,85	0.026,68	0.973,32	1.077,40

TABLE 1 (Continued)

Time	A. $e^{-\lambda t}$			B. $1-e^{-\lambda t}$	C. $1-e^{-\lambda t}$
	Minutes	Hours	Days	Days	Hours
21	0.997,36	0.853,38	0.022,25	0.977,75	1.081,37
22	0.997,24	0.846,96	0.018,57	0.981,43	1.085,35
23	0.997,11	0.840,59	0.015,49	0.984,51	1.089,34
24	0.996,99	0.834,27	0.012,92	0.987,08	1.093,33
25	0.996,86	0.827,99	0.010,78	0.989,22	1.097,34
26	0.996,73	0.821,77	0.008,99	0.991,01	1.101,36
27	0.996,61	0.815,58	0.007,50	0.992,50	1.105,39
28	0.996,48	0.809,45	0.006,26	0.993,74	1.109,
29	0.996,36	0.803,36	0.005,22	0.994,78	1.113,47
30	0.996,23	0.797,32	0.004,36	0.995,64	1.117,52
31	0.996,11	0.791,32	0.003,63	0.996,37	1.121,58
32	0.995,98	0.785,37	0.003,03	0.996,97	1.125,66
33	0.995,86	0.779,46	0.002,53	0.997,47	1.129,74
34	0.995,73	0.773,60	0.002,11	0.997,89	1.133,83
35	0.995,61	0.767,78	0.001,76	0.998,24	1.137,94
36	0.995,48	0.762,01	0.001,47	0.998,53	1.142,05
37	0.995,36	0.756,28	0.001,23	0.998,77	1.146,17
38	0.995,23	0.750,59	0.001,02	0.998,98	1.150,30
39	0.995,11	0.744,94	0.000,85	0.999,15	1.154,44
40	0.994,98	0.739,34	0.000,71	0.999,29	1.158,59
41	0.994,85	0.733,78	0.000,59	0.999,41	1.162,75
42	0.994,73	0.728,26	0.000,50	0.999,50	1.166,92
43	0.994,60	0.722,78	0.000,41	0.999,59	1.171,09
44	0.994,48	0.717,34	0.000,34	0.999,66	1.175,28
45	0.994,35	0.711,95	0.000,29	0.999,71	1.179,47

TABLE 1 (Continued)

Time	A. $e^{-\lambda t}$			B. $1-e^{-\lambda t}$	C. $1-e^{-\lambda t}$
	Minutes	Hours	Days	Days	Hours
46	0.994.23	0.706.59	0.000.24	0.999.76	1.183.68
47	0.994.10	0.701.23	0.000.20	0.999.80	1.187.89
48	0.993.98	0.696.00	0.000.17	0.999.83	1.192.12
49	0.993.85	0.690.77	0.000.14	0.999.86	1.196.35
50	0.993.73	0.685.57	0.000.12	0.999.88	1.200.60
51	0.993.60	0.680.42	0.000.10	0.999.90	1.204.85
52	0.993.48	0.675.30	0.000.08	0.999.92	1.209.11
53	0.993.35	0.670.22	0.000.07	0.999.93	1.213.38
54	0.993.23	0.665.18	0.000.06	0.999.94	1.217.66
55	0.993.10	0.660.18	0.000.05	0.999.95	1.221.95
56	0.992.98	0.655.21	0.000.04	0.999.96	1.226.25
57	0.992.85	0.650.28	0.000.03	0.999.97	1.230.56
58	0.992.73	0.645.39	0.000.03	0.999.97	1.234.88
59	0.992.60	0.640.54	0.000.02	0.999.98	1.239.21
60	0.992.48	0.635.72	0.000.02	0.999.98	1.243.54

Figure 1. The cell evacuation assembly consisting of (top to bottom) the scintillation cell, two 18-gauge hypodermic needles joined by fusing the two plastic collars together using a low-heat flame and then coating the joint with a rubber adhesive, drying tube, stopcock, a vacuum tube necessary to make the connection from the vacuum pump (10 cm empty glass tube closed at each end by a serum stopper),and a stopcock attached to the vacuum line.

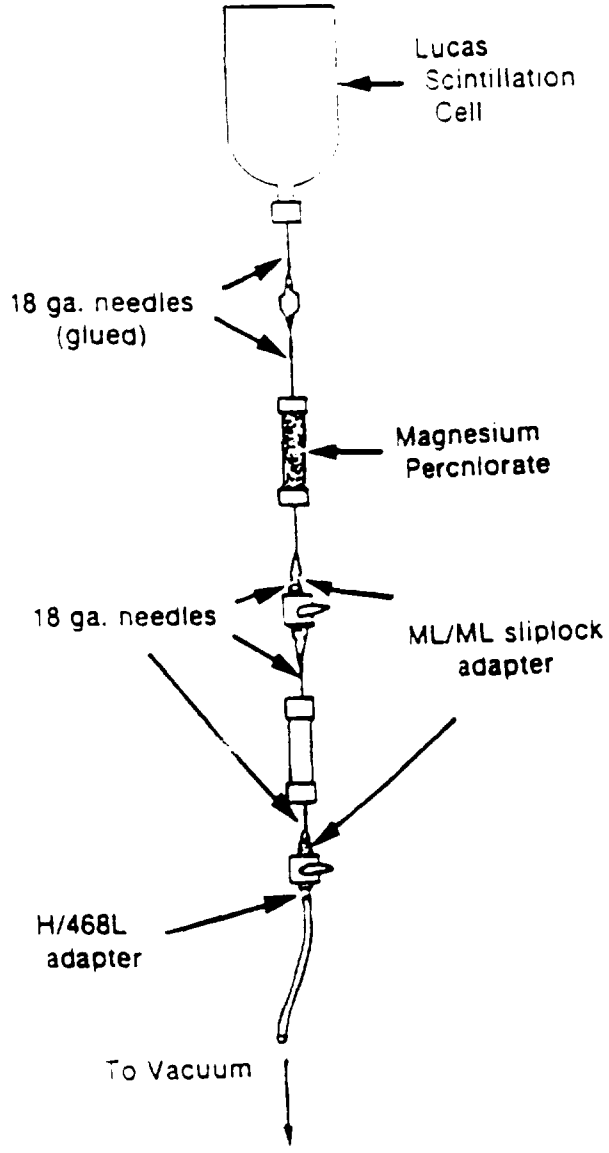


Figure 2. Cell purging assembly.

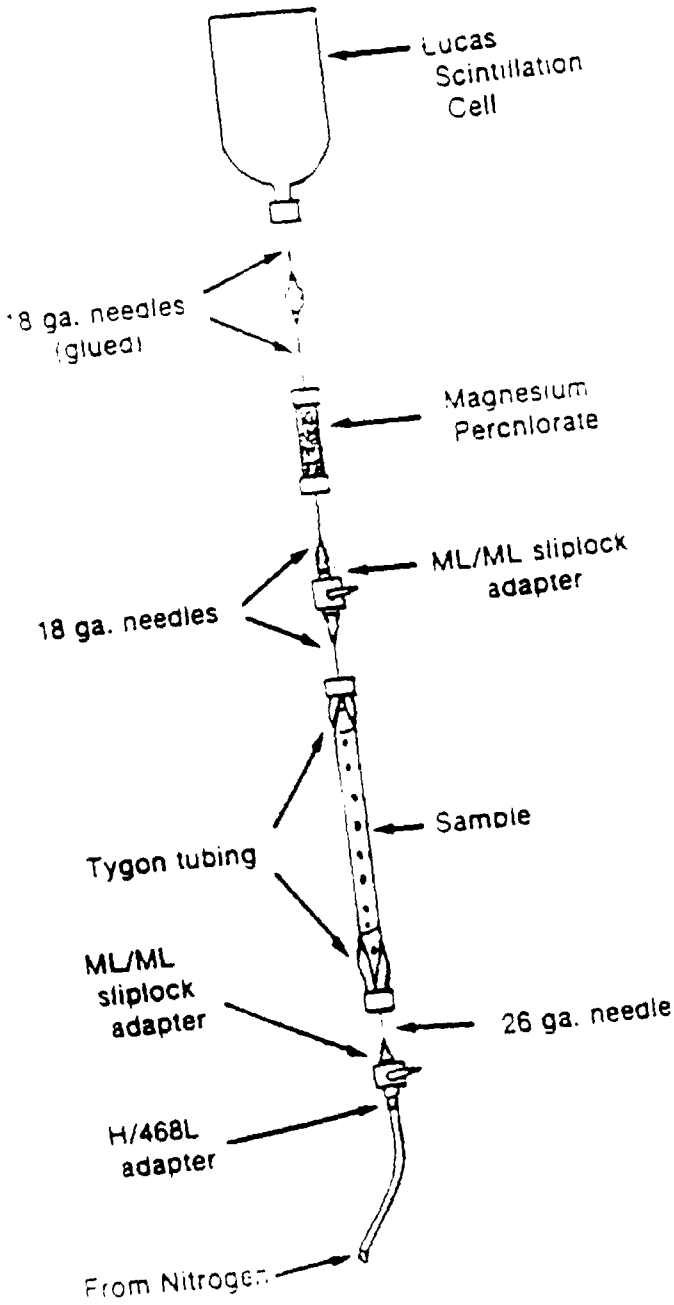
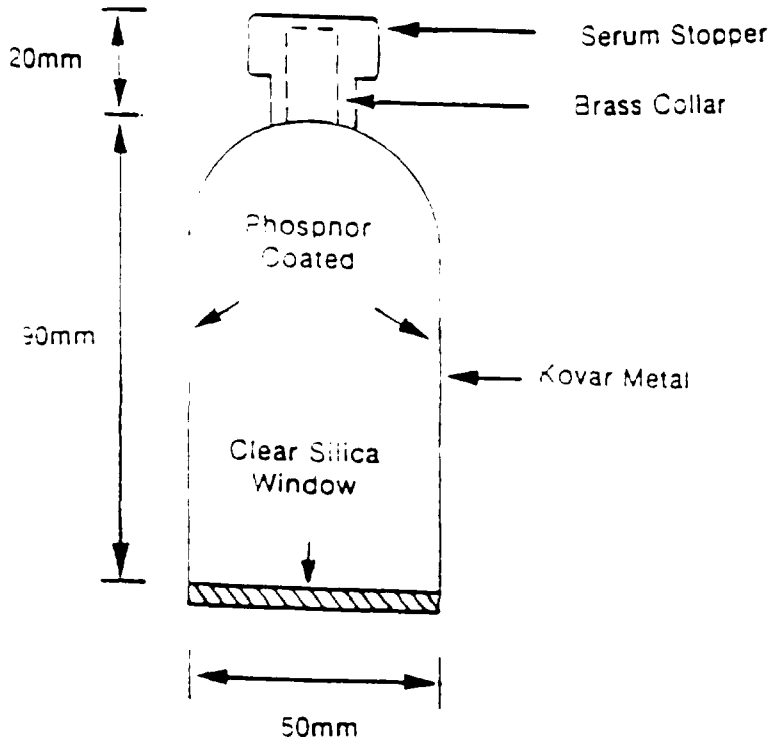
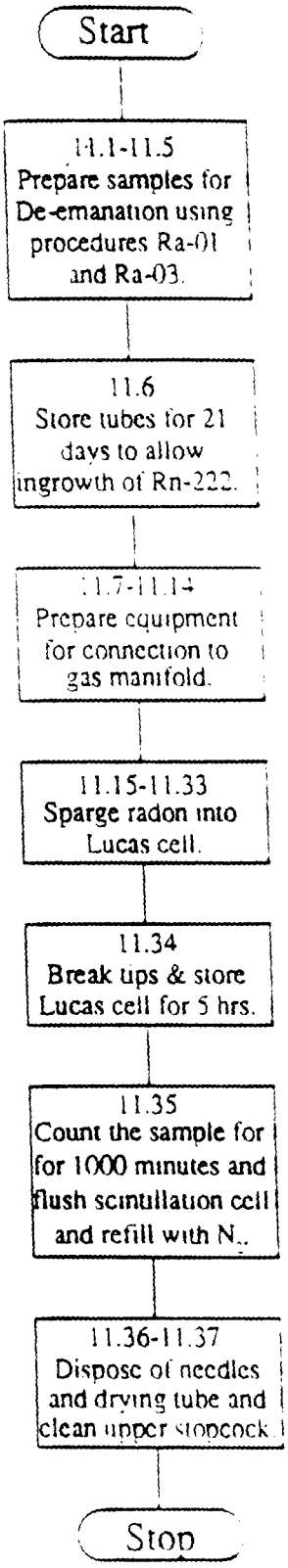


Figure 3. Lucas type scintillation cell.



Radium-226 by the De-emanation Proc



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RADIOCHEMICAL DETERMINATION OF RADIUM-228

1.0 SCOPE AND APPLICATION

1.1 This procedure describes a method for the analysis of samples for radium-228 following radium-226 de-emanation using procedure Ra-04, Radiochemical Determination Of Radium-226 By The De-emanation Procedure.

1.2 The sensitivity of this method is a function of sample size, reagent and instrument backgrounds, counting efficiency, and counting time.

1.3 Solid samples requiring analysis for radium are prepared using procedure Ra-01, The Preparation of Solid Samples For The Radiochemical Determination Of Radium-226 And Radium-228.

1.4 Urine samples requiring analysis for radium are prepared using procedure Ra-02, Radiochemical Determination Of Radium-226 In Urine.

1.5 Water samples requiring analysis for radium are prepared using procedure Ra-03, The Preparation of Water Samples For The Radiochemical Determination Of Radium-226 By The De-emanation Procedure.

1.6 Data quality objectives are identified during project scoping and development of sampling and analysis plans. DQOs are established to ensure that the data collected are sufficient and of adequate quality for their intended uses. Data collected and analyzed in conformance with the DQO process described in this document can be used in assessing the uncertainty associated with decisions related to remedial response.

2.0 SUMMARY OF METHOD

The radium is precipitated as a radium-barium sulfate. The precipitate is dissolved in a pentasodium diethylenetriamine pentaacetate (Na_5DTPA) solution. The radium-228 is a weak beta emitter and the actinium-228 daughter is allowed to grow in for 36 hours. The actinium-228 is then extracted with Di-2-ethylhexylphosphoric acid (HDEHP) and back-extracted with nitric acid. The actinium-228 is beta counted in a low-background proportional counter.

3.0 DEFINITIONS

3.1 ALARA: Acronym for "As Low as Reasonably Achievable," a basic concept of radiation protection that specifies that radioactive discharges from nuclear plants and radiation exposure to personnel be kept as far below regulation limits as feasible.

3.2 De-emanation: The process by which a gaseous product of radioactive disintegration (Rn-222 from Ra-226) is transferred into a counting cell by sparging with an inert gas (nitrogen).

3.3 Duplicate: Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures.

3.4 EMSL-LV: Acronym for Environmental Measurement Support Laboratory-Las Vegas.

3.5 IAEA: Acronym for International Atomic Energy Agency.

3.6 Laboratory reagent blank (LRB): An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if interferences are present in the laboratory environment, reagents, or apparatus.

3.7 MSDS: Acronym for Material Safety Data Sheets.

3.8 NIST: Acronym for National Institute of Standards and Technology.

3.9 R value: Ratio of observed activity divided by the actual amount of added activity.

3.10 SAP: Acronym for Sampling and Analysis Plan; rigid guidelines regarding number of samples requested, method of collection, analysis deadline, etc.

3.11 Spiked Sample: An aliquot of a sample which is spiked with a known concentration of the analyte of interest.

3.12 WHO: Acronym for World Health Organization.

4.0 INTERFERENCES

4.1 There are no known radioisotopes that interfere with this procedure.

4.2 There is no isotope of actinium available to monitor the chemical recovery of Ac-228. The chemical recovery is obtained from a sample spiked with Ra-228. The unknown sample and the spiked sample must be analyzed exactly alike for this method to work properly.

5.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this procedure has not been precisely defined. Each chemical compound should be treated as a potential health hazard and exposure to these compounds should be ALARA. The laboratory is responsible for maintaining a current file of regulations regarding safe handling of the chemicals specified in this procedure. A reference file of MSDS should be made available to all personnel involved in the chemical analysis. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

6.0 EQUIPMENT AND SUPPLIES

6.1 Centrifuge.

6.2 Planchet, stainless steel, 5-cm diameter.

6.3 Separatory funnel, 125 ml, 250 ml and 1000 ml.

6.4 Magnetic stirrer and stirring bar.

6.5 Suction filter apparatus, Kontes filter funnel, 2000 ml filter flask.

6.6 Beaker, 100 ml, 400 ml, and 1000 ml.

6.7 pH meter.

6.8 40 ml centrifuge tube.

6.9 Glass fiber filter, 2.4 cm.

7.0 REAGENTS AND STANDARDS

7.1 Acetic acid, glacial, 17.4M (CAS: 64-19-7). CH_3COOH reagent.

7.2 Acetic acid, 6M. Dilute 345 ml of the reagent grade CH_3COOH to one liter with deionized water.

7.3 Actinium wash solution. Dissolve 100 g chloroacetic acid and 2.4 ml of 41 percent Na_5DTPA in 800 ml of deionized water, adjust the pH to 3.0 with NaOH pellets using a pH meter, and dilute to one liter.

7.4 Ammonium hydroxide, 15M (CAS: 1336-21-6). Reagent grade NH_4OH.

7.5 Chloroacetic acid, 2M (CAS: 79-11-8). Add 189 g of reagent grade chloroacetic acid to a beaker. Dissolve in 800 ml of deionized water and dilute to one liter.

7.6 Diammonium citrate, 2M. Dissolve 226.2 g of dibasic ammonium citrate, $(NH_4)_2HC_6H_5O_7$ (CAS: 3012-65-5), in deionized water and dilute to 500 ml.

7.7 Di-2-ethylhexylphosphoric acid, HDEHP, 15 percent in n-heptane (CAS: 298-07-7). Dilute 150 ml HDEHP to one liter with n-heptane and transfer to a two-liter separatory funnel. Wash the HDEHP twice with 100 ml aliquots of the HDEHP wash solution. (The HDEHP wash solution should be prepared on the same day that it is used. The solution complexes after sitting form more than 24 hours, causing emulsions to form during the extractions.) Shake the funnel for one minute, venting frequently. Allow the layers to separate and discard the lower layer. Wash the HDEHP twice with 100 ml aliquots of 4M HNO_3. Shake the funnel for one minute, venting frequently. Allow the layers to separate and discard the lower layer. Store the cleaned HDEHP solution in a polyethylene bottle.

7.8 Di-2-ethylhexylphosphoric acid wash solution, HDEHP wash solution. Mix 100 ml of 2M diammonium citrate and 100 ml of 15M NH_4OH.

7.9 Diethylenetriamine pentaacetic acid, pentasodium salt, Na_5DTPA, 41 percent reagent solution (CAS: 140-01-2).

7.10 Diethylenetriamine pentaacetic acid, pentasodium salt, Na_5DTPA, 0.17M, pH 10. Add 209 ml of 41 percent Na_5DTPA to 400 ml of deionized water and filter through glass wool with suction. Adjust to pH 10 using a pH meter. (Use perchloric acid to lower the pH or sodium hydroxide to raise the pH.) Dilute to one liter with deionized water and store in a polyethylene bottle.

7.11 n-Heptane (CAS: 142-82-5). Reagent grade.

7.12 Hydrochloric acid, 12M, 37 percent HCl reagent grade (CAS: 7647-01-0).

7.13 Hydrochloric acid, 4M. Dilute 333 ml of the 37 percent reagent grade HCl to one liter with deionized water.

7.14 Hydrochloric acid, 1M. Dilute 83 ml of the 37 percent reagent grade HCl to one liter with deionized water.

7.15 Nitric acid, 16M, 70 percent HNO_3 reagent grade (CAS: 7697-37-2).

7.16 Nitric acid, 4M. Dilute 250 ml of the 70 percent reagent grade HNO_3 to one liter with deionized water.

7.17 Nitric acid, 1M. Dilute 63 ml of the 70 percent reagent grade HNO_3 to one liter with deionized water.

7.18 Perchloric acid, 12M, 70 percent $HClO_4$ reagent grade (CAS: 7601-90-3).

7.19 Sodium hydroxide (CAS: 1310-73-2). Reagent grade pellets.

7.20 Sodium sulfate, 20 percent (CAS: 7757-82-6). Dissolve 20 g anhydrous Na_2SO_4 in 70 ml deionized water and dilute to 100 ml.

7.21 Sulfuric acid, 18M, 96 percent H_2SO_4 reagent grade (CAS: 7664-93-9).

7.22 Sulfuric acid, 4 M. Dilute 222 ml of the 96 percent reagent grade H_2SO_4 to one liter with deionized water.

8.0 SAMPLE COLLECTION, PRESERVATION, AND STORAGE

All samples must have been collected using the appropriate SAP.

9.0 QUALITY CONTROL

9.1 All quality control data should be maintained and available for easy reference and inspection.

9.2 Employ a minimum of one blank per sample batch to determine if contamination is occurring.

9.3 Analyze one duplicate sample for every 10 samples. A duplicate sample is a sample split into two identical portions and given the same treatment throughout the sample preparation and analytical process.

9.4 One spiked sample or standard reference sample shall be analyzed for every 20 samples (5%) to ensure that correct procedures are being followed and that all equipment is operating properly.

9.5 Instrument calibration checks and backgrounds will be performed routinely on all detectors used for this procedure.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Efficiency Calibration

10.1.1 Prepare a solution of Antimony-124 containing 1,500-5,000 dpm/ml of activity. (Due to the short half-life of Ac-228, accurate efficiency measurements for this nuclide is impossible using this isotope. Sb-124 has a beta emission with the same energy as the Ac-228 beta particle. For this reason, the Sb-124 is used as the standard for the efficiency determination of the Ac-228).

10.1.2 Prepare at least six stainless steel planchets by washing with 4M HNO_3 and rinsing with deionized water.

10.1.3 Evaporate 1 ml of the standard on each of the planchets under a heat lamp. Continue heating the planchet until all of the acid fumes have been removed and the standard is dry.

10.1.4 Count the standards in a low-background proportional counter long enough to obtain 20,000 counts.

10.1.5 Calculate the activity in the standards at the counting time as follows:

$$A_t = A_0 e^{(-0.011510)t}$$

where

A_t = the Sb-124 activity at the counting time;

A_0 = the Sb-124 activity at the calibration time; and

t = the number of days from the calibration time to the counting time.

10.1.6 Calculate the efficiency of each standard as follows:

Efficiency = CPM/DPM,

where

Efficiency = the counting efficiency for that standard;

CPM = the counting rate of the sample; and

DPM = the disintegration rate of the sample at the counting time.

10.1.7 Calculate the average efficiency and the standard deviation for all of the standards prepared.

10.2 Carrier Standardization

This procedure is performed without the use of carriers.

11.0 PROCEDURE

11.1 Obtain the sample tube from the Ra-226 de-emanation procedure.

11.2 Holding the sample tube in the vertical position over a 400 ml beaker, carefully cut the tubing from the bottom of the tube to remove the serum stopper.

11.3 Puncture the tubing on the top of the tube to allow the liquid to flow from the tube.

11.4 Carefully invert the tube and cut the tubing and stopper away from the other end of the sample tube. Rinse the sample tube into the 400 ml beaker with 4M HCl.

11.5 Carefully decant the solution from the 400 ml beaker into a 100 ml beaker. Rinse the glass and glue remnants with 4M HCl and add the rinse to the 100 ml beaker.

11.6 Add 2 ml 18M H_2SO_4. Heat the sample on a hot plate at low heat for ten minutes. Allow the sample to settle overnight.

11.7 Carefully decant as much clear liquid as possible without losing any precipitate. Slurry the remaining liquid with the precipitate and transfer to a 40 ml centrifuge tube. Rinse the beaker with deionized water and add the rinse to the centrifuge tube. Centrifuge and discard the supernate.

11.8 Add 30 ml of 0.17M DTPA to the precipitate. Place the sample in a boiling water bath and heat with stirring to dissolve the precipitate (dissolution may require 30 minutes).

11.9 Add 1 ml of 20% Na_2SO_4, dilute to 28 ml with deionized water, and add 4 ml of 6M acetic acid. Heat for five minutes in a boiling water bath while stirring with a magnetic stir bar.

11.10 Transfer the sample to an ice bath and cool for five minutes. Remove the stir bar, centrifuge, and discard the supernate.

11.11 Repeat steps 11.8 through 11.10. Record the time of the final acetic acid addition. (Steps 11.8, 11.9, and 11.10 are performed to remove all of the Ac-228 present. The second $BaSO_4$ precipitation with acetic acid provides an actinium-free precipitate and begins the measured ingrowth of the Ac-228 from the Ra-228 present.)

11.12 Add 30 ml of 0.17M DTPA to the precipitate. Place the sample in a boiling water bath and heat with stirring to dissolve the precipitate.

11.13 Allow the solution to cool to room temperature. Cover the centrifuge tube and store for at least 36 hours to allow for Ac-228 ingrowth.

11.14 Place the sample in a boiling water bath, insert a magnetic stirring bar, and stir until any precipitate that may have formed during the ingrowth period has dissolved.

11.15 Add 1 ml of 20% Na_2SO_4, dilute to 28 ml with deionized water and add 4 ml of 6M acetic acid. Record the time of the acetic acid addition. (The precipitation of $BaSO_4$ in step 11.15 isolates the actinium in the supernate and ends the Ac-228 ingrowth period.)

11.16 Heat the sample in a boiling water bath with stirring for five minutes.

11.17 Transfer the sample to an ice bath and cool for five minutes. Remove the stirring bar, centrifuge, and decant the supernate into a clean 100 ml beaker

containing 5 ml of 2M chloroacetic acid. Rinse the walls of the tube with 2-3 ml of deionized water, being careful not to disturb the precipitate on the bottom of the tube. Add the wash to the beaker containing the sample.

11.18 Measure the pH of the solution to confirm that it is 3.0. (It is important that the pH of the solution containing the actinium is 3.0. If necessary, adjust the pH with additional 2M chloroacetic acid. Actinium extracts at a pH of approximately 3.0, so the measurment should be as close as possible.)

11.19 Add 10 ml of freshly cleaned 15% HDEHP for each sample being analyzed to a separatory funnel. Add an equal volume of deionized water and shake, with venting, for one minute. Allow the layers to separate and discard the bottom layer. Add one half volume of actinium wash solution to the funnel and shake for one minute. Allow the layers to separate and discard the bottom layer.

11.20 Transfer the sample from Step 11.18 to a 125-ml separatory funnel. Add 10 ml of the cleaned and washed 15% HDEHP. Use the last few ml of HDEHP to rinse the beaker. (It is important that the HDEHP be washed with deionized water and the HDEHP wash solution, as described in step 11.19 prior to using. This wash removes any impurities that may have formed in the solution.)

11.21 Shake vigorously for two minutes with venting. Allow the layers to separate, and discard the lower (aqueous) layer.

11.22 Add 10 ml of the actinium wash solution to the funnel. Shake for one minute, allow the layers to separate and discard the bottom layer. Repeat the wash with an additional 10 ml of actinium wash solution.

11.23 Add 10 ml of 1M HNO_3 to the funnel. Shake for one minute, allow the layers to separate, and collect the bottom layer in a 40-ml centrifuge tube.

11.24 Add an additional 5 ml of 1M HNO_3 to the funnel and repeat the back-extraction. Combine the bottom (aqueous) layers in the centrifuge tube. Discard the top (organic) layer.

11.25 Evaporate the solution on a 5-cm planchet under a heat lamp. Continue heating the planchet until all of the nitric acid fumes have been removed and the sample is dry.

11.26 Count the sample using a low-background proportional counter and calculate the Ra-228 concentration. (There is no isotope of actinium available to monitor the chemical yield of Ac-228. To determine the chemical yield, a second sample of acidified water is spiked with a known amount of Ra-228 and analyzed in the exact manner and at the same time as the unknown sample.

The chemical yield determined for the spiked sample is assumed to be equal to the yield for the unknown sample. A spiked sample must be analyzed with each batch of unknown samples.)

12.0 DATA ANALYSIS AND CALCULATION

12.1 Calculate the concentration, Z, of radium-228 in picocuries per liter as follows:

$$Z = \frac{C_1 - C_B}{(2.22)(Y)(E)(V)(1-e^{-0.1131t_1})(e^{-0.1131t_2})}$$

where

C_1 = sample count rate;

C_B = background count rate;

Y = chemical yield based on counting rate of spike added and recovered (See step 11.26);

E = beta counting efficiency(cpm/dpm);

V = sample volume (liters);

t_1 = Ac-228 ingrowth time from Ra-228 in hours (See Steps 11.15 and 11.26);

t_2 = Ac-228 decay time in hours (measured from time recorded in Step 11.15 to the midpoint of the beta count); and

2.22 = conversion from dpm to picocuries (dpm/pCi).

12.2 Calculate the counting uncertainty for the sample in picocuries per liter as follows:

$$UNC = \frac{2\left[\left(C_s/SCT\right)^2 + \left(C_B/BCT\right)^2\right]^{\frac{1}{2}}}{(2.22)(E)(V)(Y)},$$

where

UNC = the 2-standard deviation counting uncertainty;

SCT = sample counting time; and

BCT = background counting time.

12.3 Calculate the lower limit of detection (LLD) in picocuries per liter as follows:

$$LLD = \frac{4.66\sqrt{C_B T}}{(2.22)(Y)(E)(V)(T)}$$

where

C_B = background count rate;

Y = chemical yield based on counting rate of spike added and recovered (See step 11.26);

E = beta counting efficiency (cpm/dpm);

V = sample volume (liters);

T = counting time (same for sample and background); and

2.22 = conversion from dpm to picocuries (dpm/pCi).

This LLD calculation is valid if the sample counting time is the same as the background counting time.

13.0 METHOD PERFORMANCE

13.1 Accuracy

A total of 24 sets of data pairs were used for determining the accuracy of the method for measuring radium-228 in water samples (references 17.4, 17.5, and 17.6). Comparisons were made with 24 EMSL-LV reference values between 1970 and 1986. The average R value was 0.986.

Data are not presently available for determining the accuracy of this method for measuring radium-228 in soil samples.

13.2 Precision

A total of 36 sets of triplicate results were used for calculating the precision of the method for measuring radium-228 in water (references 17.5 and 17.6). Individual coefficients of variation were calculated and used as a measure of analytical precision. An average value of 21 percent was determined for these 36 sets of measurements.

Data are not presently available for determining the precision of this method for measuring radium-228 in soil samples.

14.0 POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

15.0 WASTE MANAGEMENT

15.1 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15.2 Waste streams generated by this procedure include the following: 2 mL of 18M sulfuric acid, 9 mL of 0.17M DTPA, 2 mL of 20% sodium sulfate, 8 mL of 6M acetic acid, 20 mL of actinium wash (pH 3.0), 10 mL of 15% HDEHP, and 10 mL of acetone.

16.0 REFERENCES

16.1 Johnson, J.O., "Determination of Radium-228 in Natural Waters. Radiochemical Analysis of Water", Geological Survey Water-Supply Paper 1969-G., U.S. Government Printing Office, Washington, D.C., (1971).

16.2 Percival, D.R. and Martin, D.B., "Sequential Determination of Radium-226, Radium-228, Actinium-227, and Thorium Isotopes in Environmental and Process Waste Samples", Anal. Chem. 46, 1742-1749 (1974).

16.3 Krieger, H.L. and Whittaker, E.L., "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", EPA-600/4-80-032, Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio (1980).

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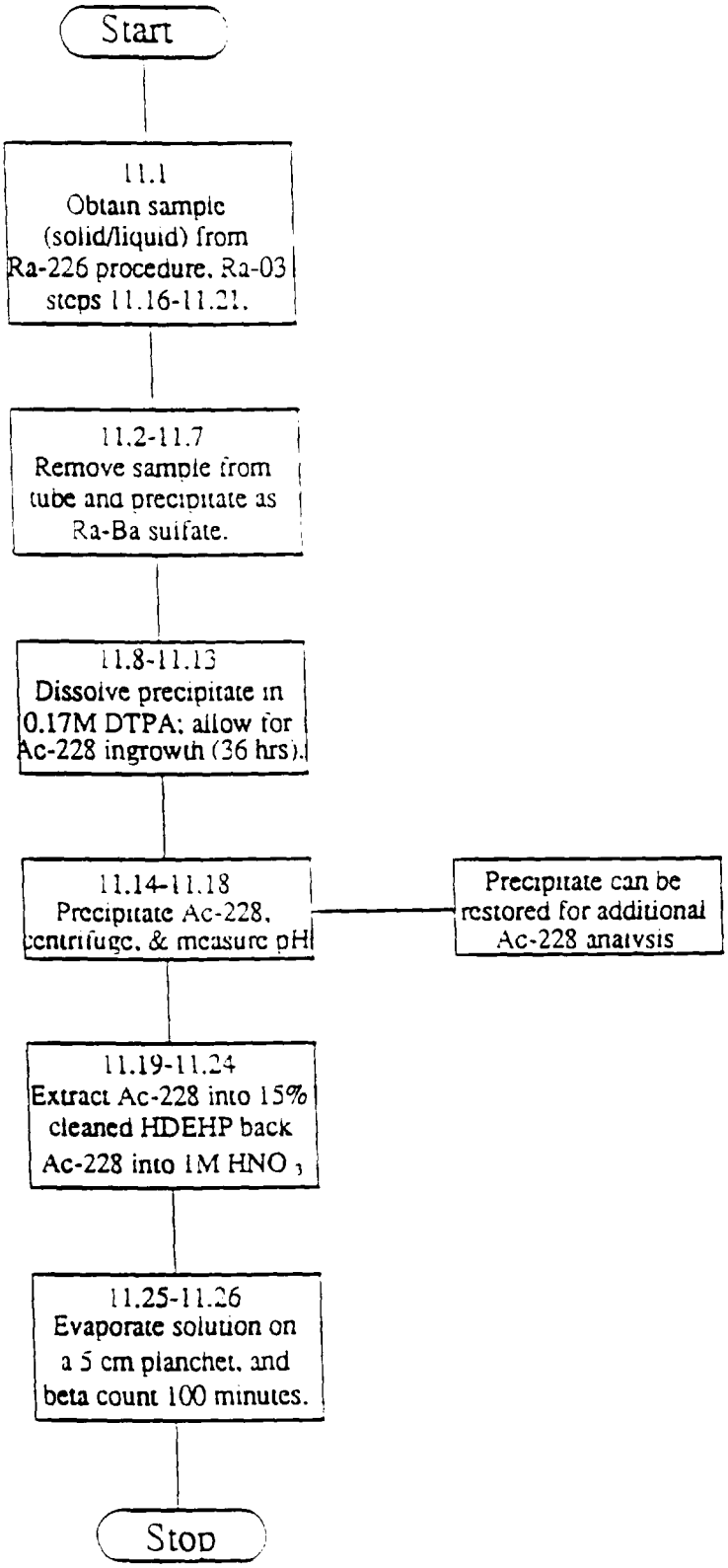
16.5 Blanchard, R.L., Broadway, J.A., and Moore, J.B., "The Eastern Environmental Radiation Facility's Participation in Interlaboratory and Intralaboratory Comparisons of Environmental Sample Analyses: 1979-1980", U.S. Environmental Protection Agency Report, EPA 520/5-82-012 (1982).

16.6 Moore, J.B., Broadway, J.A., and Blanchard, R.L., "The Eastern Environmental Radiation Facility's Participation in Interlaboratory and Intralaboratory Comparisons of Environmental Sample Analyses: 1981-1986", U.S. Environmental Protection Agency Report, EPA 520/5-89-008 (1989).

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

Radiochemical Determination of Radium-228 in Solids, Urine, and Water



DRAFT

8-11-92

THE PREPARATION OF SOLID SAMPLES FOR THE RADIOCHEMICAL DETERMINATION OF RADIUM-226 AND RADIUM-228

1.0 SCOPE AND APPLICATION

1.1 This method is used to prepare solid samples, primarily soils and sediments, for determining the concentration of radium-226 followed by radium-228.

1.2 The samples are completely solubilized by a fusion procedure which eliminates the possibility of losses due to an insoluble fraction retaining some of the radium.

1.3 Data quality objectives are identified during project scoping and development of sampling and analysis plans. DQOs are established to ensure that the data collected are sufficient and of adequate quality for their intended uses. Data collected and analyzed in conformance with the DQO process described in this document can be used in assessing the uncertainty associated with decisions related to remedial response.

2.0 SUMMARY OF METHOD

Solid samples are solubilized by fusing with a special flux at high temperature. The fused sample is dissolved and the radium separated by coprecipitation with $BaSO_4$. The procedure then continues with step 11.6 of Ra-03, The Preparation of Water Samples for the Radiochemical Determination of Radium-226 by the De-emanation Procedure.

3.0 DEFINITIONS

3.1	ALARA:	Acronym for "As Low as Reasonably Achievable," a basic concept of radiation protection that specifies that radioactive discharges from nuclear plants and radiation exposure to personnel be kept as far below regulation limits as feasible.
3.2	De-emanation:	The process by which a gaseous product of radioactive disintegration (Rn-222 from Ra-226) is transferred into a counting cell by sparging with an inert gas (nitrogen).
3.3	Duplicate:	Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures.
3.4	EMSL-LV:	Acronym for Environmental Measurement Support Laboratory-Las Vegas.

3.5	IAEA:	Acronym for International Atomic Energy Agency.
3.6	Laboratory reagent blank (LRB):	An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The LRB is used to determine if interferences are present in the laboratory environment, reagents, or apparatus.
3.7	MSDS:	Acronym for Material Safety Data Sheets.
3.8	NIST:	Acronym for National Institute of Standards and Technology.
3.9	R value:	Ratio of observed activity divided by the actual amount of added activity.
3.10	SAP:	Acronym for Sampling and Analysis Plan; rigid guidelines regarding number of samples requested, method of collection, analysis deadline, etc.
3.11	Spiked Sample:	An aliquot of a sample which is spiked with a known concentration of the analyte of interest.
3.12	WHO:	Acronym for World Health Organization.

4.0 INTERFERENCES

There are no expected interferences.

5.0 SAFETY

The toxicity or carcinogenicity of each reagent used in this procedure has not been precisely defined. Each chemical compound should be treated as a potential health hazard and exposure to these compounds should be ALARA. The laboratory is responsible for maintaining a current file of regulations regarding safe handling of the chemicals specified in this procedure. A reference file of MSDS should be made available to all personnel involved in the chemical analysis. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

6.0 EQUIPMENT AND SUPPLIES

6.1 Metricel DM-800 membrane filter, 25-mm dia., 0.8 micrometer pore size, or equivalent.

6.2 Magnetic stirrer and stirring bar.

6.3 Platinum crucibles, 20 ml and 50 ml, with lids.

6.4 Suction filter apparatus.

6.5 Tongs for platinum crucible.

6.6 Meker burner.

6.7 Tripod.

6.8 Assorted glassware.

6.9 Kontes filter funnel, Kontes Glass Co., Vineland, NJ.

6.10 Hot plate.

6.11 Hot water bath.

6.12 De-emanation storage tubes: Pyrex brand glass tubing, 15-mm i.d. and 45-cm in length.

7.0 REAGENTS AND STANDARDS

7.1 Acetone-ethanol mixture, 50 percent each by volume (CAS: 67-64-1 & CAS: 64-17-5).

7.2 Ammonium sulfate, 10 percent (CAS: 7783-20-2). Dissolve 10 g reagent grade $(NH_4)_2SO_4$ in 90 ml of distilled water.

7.3 Barium chloride, 10 mg Ba^{+2}/ml (CAS: 10361-37-2). Dissolve 17.79 g $BaCl_2 \cdot 2H_2O$ in 1 liter of distilled water.

7.4 Barium chloride, 2 mg Ba^{+2}/ml. Dilute 200 ml of the 10 mg/ml barium chloride solution to 1 liter. Filter after 24 hours.

7.5 Fusion flux. Mix thoroughly 15 mg barium sulfate ($BaSO_4$) (CAS: 7727-43-7), 32.9 g potassium carbonate (K_2CO_3) (CAS: 584-08-7), 25.3 g sodium carbonate (Na_2CO_3) (CAS: 497-19-8), 16.8 g sodium tetraborate decahydrate ($Na_2B_4O_7 \cdot 10H_2O$) (CAS: 1303-96-4). Heat to expel water, then fuse in a platinum crucible and mix thoroughly by swirling. Cool and grind in a porcelain mortar. Store in an airtight bottle.

7.6 Hydrochloric acid, 3M (CAS: 7647-01-0). Dilute 250 ml of the 37 percent HCl reagent to 1 liter with distilled water.

7.7 Hydrogen peroxide, 3 percent reagent (CAS: 7722-84-1).

7.8 Hydrofluoric acid, 29M, 48 percent HF reagent (CAS: 7664-39-3).

7.9 Phosphoric acid, 15M, 85 percent H_3PO_4 reagent (CAS: 7664-38-2).

7.10 Sulfuric acid, 18M, 96 percent H_2SO_4 reagent (CAS: 7664-93-9).

7.11 Sulfuric acid, 0.1 M. Dilute 5.6 ml of the 96 percent reagent H_2SO_4 to 1 liter with distilled water.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 All samples must have been collected using the appropriate SAP.

9.0 QUALITY CONTROL

9.1 All quality control data should be maintained and available for easy reference and inspection.

9.2 Employ a minimum of one blank per sample batch to determine if contamination is occurring.

9.3 Analyze one duplicate sample for every 10 samples. A duplicate sample is a sample split into two identical portions and given the same treatment throughout the entire sample preparation and analytical process.

9.4 One spiked sample or standard reference sample shall be analyzed for every 20 samples (5%) to ensure that correct procedures are being followed and that all equipment is operating properly.

9.5 Instrument calibration checks and backgrounds will be performed routinely on all detectors used for this procedure.

10.0 CALIBRATION AND STANDARDIZATION

N/A

11.0 PROCEDURE

11.1 Weigh 0.5 g of soil sample and place in a platinum crucible.

11.2 Add 4 g of flux and mix well. (The amount of flux should be increased proportionately with sample size; however, a minimum of 2.0 grams of flux should be used regardless of sample size.)

11.3 Put lid on crucible and place it on tripod over Meker burner. Fuse to a red heat for 30 minutes.

11.4 Swirl mixture as needed during fusion to aid in dissolution of flux.

11.5 Remove crucible from heat with tongs and swirl mixture until it begins to solidify.

11.6 Prepare the following solution in a 600 ml beaker, made up as needed: 120 ml distilled water, 10 ml 18M H_2SO_4, and 5 ml 3 percent H_2O_2.

11.7 Place platinum crucible and lid in beaker. Fused sample will dissolve away from crucible in about 30 minutes.

11.8 Remove crucible and lid from solution. Rinse crucible and lid with distilled water and pour into beaker.

11.9 Place magnetic stirring bar in beaker and begin stirring.

11.10 Add 50 ml dilute $BaCl_2$ solution to beaker.

11.11 Stir contents of beaker for 90 minutes.

11.12 Remove magnetic stirring bar, rinse with distilled water and wait overnight for the $BaSO_4$ to precipitate.

11.13 Continue at step 11.6 of Ra-03, The Preparation of Water Samples for the Radiochemical Determination of Radium-226 by the De-emanation Procedure.

12.0 DATA ANALYSIS AND CALCULATION

N/A

13.0 METHOD PERFORMANCE

N/A

14.0 POLLUTION PREVENTION

Pollution prevention encompasses any technique that reduces or eliminates the quantity and/or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

15.0 WASTE MANAGEMENT

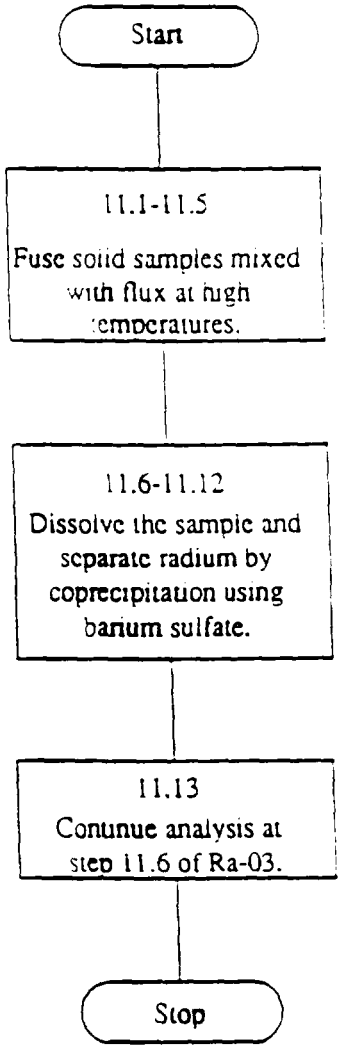
The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any sewer discharge permits and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions.

16.0 REFERENCES

Standard Methods for the Examination of Water and Waste Water, 15th Ed., American Public Health Association, Washington, D.C. (1980).

17.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

The Preparation of Solid Samples for the Radiochemical Determination of Radium-226 & Radium-228



SOIL WASHING - ORBITAL SHAKER

1.0 SCOPE AND APPLICATION

This procedure describes a method for vigorously washing a soil sample to promote particle liberation. This method can be applied to all soil samples, but works best when applied to whole soils. Its purpose for application to Kress Creek sediment samples is to approximate leaching potential of radionuclides. Therefore low mixing rates will be used.

2.0 SUMMARY OF METHOD

A sample of the soil to be tested is placed in a 1-gallon container. The sample is mixed with a known amount of ~~tap~~ Kress Creek surface water to produce a specific liquid-to-solid (L/S) ratio. The sample is placed on the orbital shaker at a predetermined speed for a specific length of time. ~~The soil particles are abraded to separate the particles for further treatment~~.

3.0 INTERFERENCES

There are no known interferences with this procedure.

4.0 APPARATUS AND MATERIALS

4.1 Lab-Line Orbit Shaker with tachometer, modified to hold 1-gal containers.

4.2 1-gal plastic containers, Nalgene or equivalent.

4.3 1000-mL graduated cylinder.

4.4 Interval timer (Note 11.1).

5.0 REAGENTS

5.1 ~~Tap water~~ Kress Creek surface water

6.0 HEALTH AND SAFETY

6.1 All personnel are required to wear protective clothing. As a minimum this will include chemical resistant gloves, a labcoat or apron, and safety glasses.

6.2 Dry samples will only be handled in a laboratory hood. Respirators approved for dusts and radionuclides will be worn when handling dry samples unless the sample has been cleared by the Environmental Compliance Officer and the Radiation Protection Officer.

6.3 Samples contaminated with chemicals and/or radioactivity will be handled with the usual precautions.

7.0 SAMPLE PRESERVATION, COLLECTION, AND HANDLING

NAREL is responsible for these activities.

8.0 PROCEDURE

8.1 Calibration

8.1.1 The tachometer was calibrated at the factory. The calibration is checked once a year to ensure the shaker is accurate to within $\pm$10%.

8.1.2 The shaker is tested at three speeds for accuracy, 50 RPM, 100 RPM, and 200 RPM.

8.1.3 Turn on the shaker and adjust the tachometer to one of the three speeds to be tested.

8.1.4 Measure the amount of time required for the table to rotate 50 times. Calculate the RPM.

8.1.5 Repeat steps 8.1.3 and 8.1.4 for the remaining speeds.

8.1.6 Determine if the tachometer readings are linear by subtracting the actual RPM from the tachometer readings. The differences should be constant for a linear correlation. If the differences are not linear return the unit to the manufacturer for repairs.

8.1.7 If the readings are linear, but not accurate, adjust the speed to the correct reading using the screw located in the center of the tachometer dial.

8.1.8 Recheck the calibration. Record the calibration date and the initials of the technician performing the

calibration on a piece of tape and attach the tape to the orbital shaker.

8.2 Method

8.2.1 Accurately weigh out a dry sample of soil of at least 250 g (Note 11.2).

8.2.2 Quantitatively transfer the sample to a clean 1-gal container.

8.2.3 Add sufficient water to obtain the desired L/S ratio. An efficient L/S ratio for this method has been determined to be 4/1. ~~An example would be a~~ 250 g sample ~~which would~~ may require 1000 mL of water for a L/S ratio of 4/1.

8.2.4 Tightly cap the container and place it securely in the holder on the rotary shaker.

8.2.5 Make sure the speed control is set to zero and turn on the power. Gradually increase the speed until the desired setting is obtained. ~~The most efficient~~ use a low speed, ~~is~~ ~~X~~50 RPM. ~~the maximum speed.~~

8.2.6 Leave the sample on the shaker for ~~the desired amount of time, usually~~ 30 min.

8.2.7 Gradually reduce the shaking speed until the shaker stops. Turn off the power and remove the sample.

8.2.8 Record the sample size, L/S ratio, L/S units, speed and time in the Experimental Notebook.

8.3 Calculation

There are no calculations for this procedure.

8.4 Analysis

8.4.1 Analyze fraction of surface water before test for radionuclides of interest

8.4.2 Analyze sediment fraction after test (after filtering with 0.45 um filter) and water fraction after test for radionuclides of interest.

9.0 QUALITY CONTROL

9.1 There are no spikes or blanks available to check this type of procedure. If sufficient sample is available repeat the test a minimum of three times to provide minimum statistics and quantify differences for the soil being tested.

10.0 METHOD PERFORMANCE

10.1 There is no performance evaluation available for this method at this time.

11.0 NOTES

11.1 A clock or watch with a second hand is accurate enough for the calibration of the orbital shaker.

11.2 250 gram samples are the smallest samples that can be analyzed with this procedure. Smaller samples will be violently thrown from the shaker at higher speeds. Some samples may require additional sample to prevent the sample from being thrown from the shaker.

11.3 This procedure was tested to determine the most efficient L/S ratio, speed and shaking time. L/S ratios from 1/1 to 10/1 were tested with the best results for Montclair soil at 4/1. The results at L/S ratios of 2/1, 3/1, and 5/1 differed very little from the results at 4/1, and are acceptable substitutes for many samples. The speed and time results showed that the faster speeds for longer times yielded the best results, with the speed influencing the results more than the time. The 350 RPM speed is the fastest speed that can be maintained for long periods of time and should be used for most tests. The 30 minute time is often increased to 60 minutes to get slightly better results, but in many cases the increased time for the experiment outweighs the minor benefits.

12.0 REFERENCES

12.1 Particle Size and Radionuclide Characterization, Chemical Wash and Extraction Studies on the Contaminated Soils From Montclair and Glen Ridge, New Jersey, W. S. Richardson, September 1989.

13.0 ALTERNATE METHODS

There are no alternative methods available at this time.