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[c]Sample preservation should be performed immediately upon sample collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[d]When any sample is to be shipped by common carrier or sent through the U.S. Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements in this table, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation, has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.3 or less).

[e]Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid.

Some samples may not be stable for the maximum time period given in the table. A laboratory is obligated to hold the sample for a shorter time if knowledge exists to show this is necessary to maintain sample integrity.

[f]If samples cannot be filtered within 48 hours, add 1 ml of a 2.71% solution of mercuric chloride to inhibit bacterial growth.

[g]Should only be used in the presence of residual chlorine.

[h]Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustment in order to determine if sulfide is present. If sulfide is present, it can be removed by addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[i]For dissolved metals, filter immediately on site before adding preservative.

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[j]Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[k]The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within three days of sampling.

[l]When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times must be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and 40 days after extraction. Exceptions to this optimal preservation and holding time procedure are noted in footnotes g, m, and n.

[m]If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0 ± 0.2 to prevent rearrangement to benzidine.

[n]Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[o]For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.

[p]The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[q]Sample receiving no pH adjustment must be analyzed within 7 days of sampling.

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APPENDIX C

Relevant ESE Analytical Services SOPs

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C.3 Sample Storage Organization

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SOP 4101-05

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C.1

Table of Contents of SOP Manual

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ASG3242-002	Operation and Maintenance of the ABC Laboratories, Inc. Gel Permeation Chromatograph (GPC) Model 1002A
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AS3243-003	Procedure for the Operation of the Shimadzu LC-6A High Performance Liquid Chromatography System
AS3243-004	Procedure for the Operation of the PE Nelson 2700 Turbochrom Chromatography System
ASG3243-005	Procedure for the Operation of the Hewlett Packard Model 5880A Gas Chromatograph
ASG3243-006	Standard Operating Procedure for the Ramona 5 LS Radiochemical Detector

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C.2

Sample Receipt and Distribution
SOP 4122-04

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GUIDE TO ESE STANDARD OPERATING PROCEDURES

These Standard Operating Procedures (SOPs) describe how ESE's Gainesville Chemistry Laboratory organizes and executes administrative, quality control (QC), housekeeping, safety, sample handling, and data management tasks which constitute the routine daily operation of the laboratory. Specific client requests or contractual obligations may supercede these procedures; These SOPs apply in all cases where there are no such specific requirements.

The SOPs are authorized by signature of the Laboratory Manager. The Laboratory Manager or his designate keeps the master copy, a copy of each SOP is distributed to each laboratory supervisor or department manager, and copies of selected SOPs are given to each employee performing analytical procedures.

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SAMPLE RECEIPT AND DISTRIBUTION
STANDARD OPERATING PROCEDURE
SOP 4122-04

The Standard Operating Procedure (SOP) describes how ESE receives and distributes the sample and associated information to analytical departments.

DATE: 11/6/89 AUTHORIZED BY: John G. Morris

DISTRIBUTION:

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SAMPLE RECEIPT AND DISTRIBUTION
SOP 4122-04

1.0 ABSTRACT

This Standard Operating Procedure (SOP) describes how ESE receives and distributes samples and associated information to analytical departments.

2.0 SUMMARY

This SOP describes chain-of-custody and sample documentation requirements. An overview of ESE's sample receipt and distribution system is presented schematically in Exhibit A. Sample storage is described in SOP 4122-05, sample login on ESE's computer network is described in SOP 4151-03, and sample container labelling is described in SOP 4151-02.

3.0 PROCEDURES

3.1 SAMPLE SHIPMENT RECEIPT

3.1.1 COMMERCIAL DELIVERY

Receipt responsibility lies with the ESE General Receiving personnel and the ESE Chemistry Division Sample Custodian. During normal working hours, samples are received by ESE General Receiving, who then deliver the sample packages unopened to the laboratory. On weekends, sample packages are received at the ESE Mail Room. The ESE Sample Custodian or appropriate Lab Coordinator transfers them (unopened) to the laboratory.

3.1.2 DELIVERY BY ESE FIELD PERSONNEL

Receipt responsibility lies with the Field Personnel who must also transport the sample packages unopened to the laboratory.

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3.2 SAMPLE PACKAGE REVIEW AND LOGIN

The sample package deliverer enters the date and time of delivery to the laboratory, the number of physical packages, the project coordinator or addressee, and any comments including visible or suspected physical condition of the packages into the Sample Chest Custody Logbook (Exhibit B). Comments requiring more space than the logbook format allows may be continued on the page's reverse side. This entry is signed by the deliverer. During normal working hours, the deliverer places the packages on counters in the sample receiving area and leaves them there for further processing by the sample custodian. At other times, the deliverer places the packages in the coldroom after logbook entry. Specific sign-in procedures are posted at the coldroom.

3.3 SAMPLE CHEST UNPACKING, SAMPLE INTEGRITY, AND DOCUMENTATION REVIEW

3.3.1 CARTON RECEIPT DOCUMENTATION

Sample chests (packages) that have been transported to the sample receiving area in the laboratory are logged into the Sample Chest Custody book and then reviewed for integrity by the Sample Custodian. The chests are then recorded as being received by the laboratory in the Sample Chest Custody Logbook (see Exhibit B).

3.3.2 SAMPLE CHEST OPENING AND UNPACKING

Any sample chest known to contain or suspected of containing hazardous or toxic samples will be transported to a properly vented hood to be opened and unpacked employing protective measures.

Samples not expected to contain radioactive materials are handled as per SOP 4122-04, 3.3.4. Samples expected to contain radioactive materials are handled as per SOP 4122-04, 3.3.5.

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All other sample chests are opened and unpacked in well ventilated, unencumbered areas provided in the sample receiving area, employing techniques described in the Chemistry Laboratory Safety Manual.

When sample fractions are unpacked, they are arranged in a logical order corresponding to field logsheet/chain-of-custody documentation that was received with the sample chest (see Exhibit C).

3.3.3 SAMPLE INTEGRITY AND DOCUMENTATION REVIEW

The sample custodian reviews the integrity of all sample fraction containers, as well as, the accuracy and clarity of all documentation received. All insufficiencies and/or discrepancies are recorded on the logsheet and reported to the appropriate laboratory project coordinator immediately. The Sample Custodian audits a portion of all fractions requiring field preservation to assure that they have been properly preserved. The Custodian will preserve unpreserved fractions upon receipt. All deficiencies in sample preservation are made known to the Laboratory Project Coordinator; all inadequacies are recorded on the logsheet and reported to the Laboratory Project Coordinator. After it has been determined that a sample has been received intact, in proper condition, properly preserved, and with necessary and accurate documentation, the Sample Custodian signs the logsheet. Any marks or notes made on the chain of custody document by the Custodian should be clearly distinguished from original field notations.

3.3.4 HANDLING PROCEDURES WHEN SAMPLES ARE NOT SUSPECTED TO CONTAIN MATERIAL HAVING RADIOACTIVE EMISSIONS IN EXCESS OF BACKGROUND EMISSIONS

All sample shipments delivered to the Sample Receipt Area are checked for radioactivity prior to acceptance. A survey shall be made for alpha, beta and gamma activity with a G-M Survey Meter and recorded in the package survey log.

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If the shipment is found to contain material having radioactive emissions in excess of background emissions, the Radiation Safety Office (RSO) is immediately notified and the package is transferred to the radiochemistry laboratory sample receipt and log-in area. The "SOP for Sample Screening when samples are suspected to contain material having radioactive emissions in excess of background emissions" is now followed for this shipment.

If the shipment does not contain material having radioactive emissions in excess of background emissions, the package is opened and each sample surveyed for alpha, beta and gamma activity with a G-M Survey Meter and the results of the survey recorded in the sample survey log. Samples below 0.5 mRem/hr will follow standard log-in and storage procedures. If, however, samples are found to have levels in excess of 0.5 mRem/hour, the RSO is notified before any further action is taken. The RSO assesses the situation to determine if the samples may be analyzed or if the samples must be repackaged and returned to their point of origin. If the RSO determines that the samples may be analyzed, they are first transferred to the Radiochemistry Laboratory sample receipt and log-in area.

If it is determined by the RSO that a sample contains radioactivity above acceptable levels (1 mRem/hr), the appropriate Laboratory Coordinator and Section Manager are notified to determine with the RSO the disposition of the samples. Samples will be stored within the Radiochemistry Sample Storage Area during this period.

3.3.5 HANDLING PROCEDURES WHEN SAMPLES ARE SUSPECTED TO CONTAIN MATERIAL HAVING RADIOACTIVE EMISSIONS IN EXCESS OF BACKGROUND EMISSIONS
These shipments are delivered to the Radiochemistry Laboratory sample receipt and log-in area. A survey is then performed for alpha, beta and gamma activity with a G-M Survey Meter and recorded in the package

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survey log. This survey determines whether any sample containers have been ruptured and/or the packing materials contaminated. If the levels exceed 0.5 mRem/hr, the RSO shall be notified immediately to assess the situation. Upon initial determination of safe external activities (less than 0.5 mRem/hr), the package is opened in a hood and each sample surveyed for alpha, beta and gamma activity with a G-M Survey Meter and the results of the survey recorded in the sample survey log. This log is maintained by the Radiochemistry Department. The unused portion of the samples will be stored within the Radiochemistry Sample Storage Area during sample preparation and analysis.

After analyses have been completed, any remaining sample material shall be disposed by either dilution of low level materials into the Low Level Radiochemistry Waste Barrel or by internment in the Hazardous Materials Shed for samples with activities above 2 mRem/hr for later disposal by a commercial waste handling corporation.

3.4 SAMPLE LOG-IN AND SAMPLE FRACTION DISTRIBUTION AND STORAGE

The Sample Custodian submits the original signed logsheet to Data Management and a photocopy to the Laboratory Project Coordinator. Freight-slips are attached to the original logsheet for filing.

The sample identification, analytical requirements, and all project information are entered into the computer system by Data Management personnel in accordance with the Computer Sample Log-in/Sample Arrival Notice SOP 4151-03. This process initiates the sample holding time tracking systems, available number systems, and sample arrival notice system.

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An example of computer-generated sample arrival notices is presented in Exhibit D-1. Manually generated sample arrival notices are required for all short-holding-time sample fractions; an example is presented in Exhibit D-2.

Sample Fraction Distribution and Storage

The Sample Custodian distributes sample fractions as presented below:

Sample Fraction	Delivered to	Documented On
R	Radiochem Laboratory	DFRS
V, VP (GC)	Volatiles-GC Laboratory	DFRS
V, VP (GCMS)	GCMS Laboratory	DFRS
Fast turnaround/SHT	Appropriate Laboratory	MGSAN
All Other Fractions	Cold Room	CRSAL

Note: DFRS - Departmental Fraction Receipt Sheet
MGSAN - Manually Generated Sample Arrival Notice
CRSAL - Cold Room Sample Arrival Logbook
SHT - Short Holding Time

All fractions delivered to the specific laboratories must be logged into the Departmental Fraction Receipt Sheets by the Sample Custodian (see Exhibit E). All fractions placed in the coldroom must be logged on the coldroom sample arrival logsheet (see Exhibit F). All fractions removed and returned by analysts are to be documented on check-in/check-out logs (Exhibits G and H).

Delivery and Storage in the Radiochemistry Department

At the Radiochemistry Department, samples are delivered with arrival notices and chain of custody sheets to the Department Manager or the analyst on duty. Samples are then logged into the radiochemistry sample log book and are stored inside the padlocked storage room located in the radiochemistry wet lab trailer.

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When samples are to be analyzed they are checked in and out of the storage room and logged on the Radiochemistry Storage and Custody log book located on the storage room door (see Exhibit I.).

After all analyses have been performed on the samples and the data has been delivered to the client, the samples are retained in the storage room for an interim period of one month. After this period has passed, the samples are either returned to the client (if requested) or turned over to the Hazardous Waste department for proper disposal. The samples transferred to Hazardous Waste are classified as Low Level or High Level radioactive waste by the radiochemistry department, prior to their transfer. Based upon the level of radioactivity present (Low Level Waste or High Level Waste), the samples are disposed of in proper NRC/Florida DHRS regulated manner.

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MS/SOP ESE 4120-3

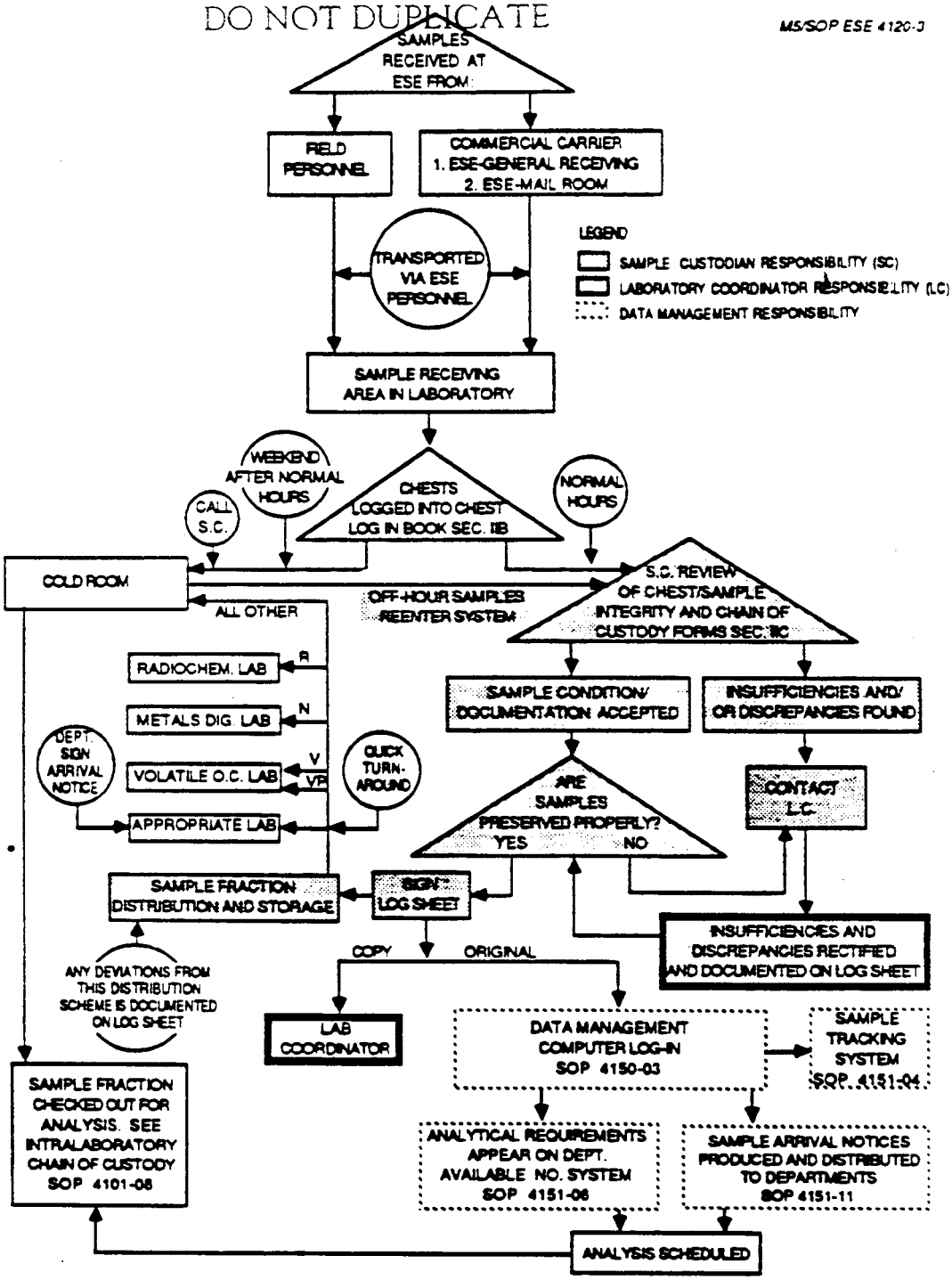

Exhibit A
SAMPLE RECEIPT, LOG-IN, AND DISTRIBUTION FLOW CHART (SOP #4122-04)

SOURCE: ESE, 1987.

ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.

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SAMPLE CHEST CUSTODY FORM

****DO NOT REMOVE FROM CHECK-IN AREA****

Relinquished By:					Received By:						
Time	Date	Signature	No. of Chests	Coordinator	Date	Time	Project Name	Signature	Comments *	Rads>Background	
										Chest?	Sample?

* No comment means that the container was relinquished/received in good condition, properly sealed and that there was no evidence of tampering.

CHEST(12/11/89)

EXHIBIT B

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ENVIRONMENTAL SCIENCE & ENGINEERING 01-20-87 *** FIELD LOGSHEET *** FIELD GROUP: FTA2W
PROJECT NUMBER 87424 0000 PROJECT NAME: CAPE CANAVERAL LAB COORD. DAVE KNOTHE

FTA2W

ESE #	SITE/STA	HAZ?	FRACTIONS(CIRCLE)	DATE	TIME	PARAMETER LIST	H2O TEMP C	FIELD PH STD UNITS	SP COND UMHOS/CM
*1	CCFTA2-1	N VP	O W V V V VP VP VP	25 Feb 87	1125	CAPEW8	23.4	6.9	1232
*2	CCFTA2-2	N VP	O W V V V VP VP VP		1248	CAPEW8	23.4	6.9	1429
*3	CCFTA2-3	N VP	O W V V V VP VP VP		1405	CAPEW8	25.3	7.7	2640
*4	CCFTA2-4	N VP	O W V V V VP VP VP		1430	CAPEW8	24.4	7.3	1170
*5	CCFTA2-5	N VP	O W V V V VP VP VP		1325	CAPEW8	24.5	7.0	2100
*6	CCFTA2-6	N VP	O W V V V VP VP VP		1215	CAPEW8	25.2	6.7	1281
*7	CCFTA2-DUP	N VP	O W V V V VP VP VP		—	CAPEW8	—	—	—
*8	BLANK	N VP	O W V V V VP VP VP			CAPEW8			

NOTE -CHANGE OR ENTER SITE ID AS NECESSARY; UP TO 9 ALPHANUMERIC CHARACTERS MAY BE USED
-CIRCLE FRACTIONS COLLECTED. ENTER DATE, TIME, FIELD DATA (IF REQUIRED), HAZARD CODE AND NOTES
-HAZARD CODES: I-IGNITABLE C-CORROSIVE R-REACTIVE T-TOXIC WASTE O-OTHER ACUTE HAZARD; IDENTIFY SPECIFICS IF KNOWN
-PLEASE RETURN LOGSHEETS WITH SAMPLES TO ESE

	RELINQUISHED BY: (NAME/ORGANIZATION/DATE/TIME)	RECEIVED BY (NAME/ORGANIZATION/DATE/TIME)
1	Robert Bush / ESE / 25 Feb 87 / 1800	[illegible] ESE 8/26/87 10[illegible]
2		
3		

OTHER FIELD NOTES FOR FIELD GROUP FTA2W:

FTA2-6 — Hydrocarbons - free product - caution!!!

EXHIBIT C.

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ENVIRONMENTAL SCIENCE & ENGINEERING 08/26/87 STATUS: PAGE# 1

*** SAMPLE ARRIVAL NOTICE FOR DEPARTMENT 4133 ***

PROJECT NUMBER 87846 0100 PROJECT NAME CHEM SUPP 87830
FIELD GROUP NW CARBIDE PROJECT MANAGER JFG
LAB COORDINATOR JOHN GEMOULES
COLLECTION DATES: 08/20/87, 08/19/87
DUE DATES : DATE NOT SPECIFIED
TURNAROUND MATRIX : NONE SPECIFIED LIMIT SPEC'D : NONE SPECIFIED
QC : SAMPLE FRACTIONS
COMMENTS/QC FR:

SAMPLE SEQ #

DET.LMT. 11111111 3
CRITERIA 678901234567123455

ARSENIC,DISS	.UG/L	1000*GFAA	NNNNNNNNNNOONNNNNO
BARIUM,DISS	.UG/L	1005*ICAP	NNNNNNNNNNOONNNNNO
CADMIUM,DISS	.UG/L	1025*ICAP	NNNNNNNNNNOONNNNNO
CHROMIUM,DISS	.UG/L	1030*ICAP	NNNNNNNNNNOONNNNNO
LEAD,DISS	.UG/L	1049*ICAP	NNNNNNNNNNOONNNNNO
MERCURY,DISS.	.UG/L	71890*CVAA	NNNNNNNNNNOONNNNNO
SELENIUM,DISS	.UG/L	1145*GFAA	NNNNNNNNNNOONNNNNO
SILVER,DISS	.UG/L	1075*ICAP	NNNNNNNNNNOONNNNNO

O=OPEN L=IN LAB E=EXTRACTED N=NOT REQUESTED D=DONE S=SCHEDULED

EXHIBIT D-1.

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ESE VOA GC/MS SAMPLE THRU - LOG WEEK ENDING_____

Project Coordinator	Samples & Description	Arrival Date	Checked in by	Screen Date	Analysis Date	Complaints	Comments VOA Screen Requested

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ESE VOA GC SAMPLE THRU - LOG WEEK ENDING_____

Project Coordinator	Samples & Description	Arrival Date	Checked in by	Screen Date	Analysis Date	Complaints	Comments VOA Screen Requested

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EXHIBIT D-2.

ESE. ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.

SAMPLE ARRIVAL NOTICE

TO: _____ DATE: _____

FROM: _____

PROJECT NAME: _____

PROJECT NO.: _____

PLEASE NOTE THAT THE FOLLOWING SAMPLES HAVE BEEN CHECKED IN:

FIELD GROUP	SAMPLE SEQUENCE NO.	MATRIX	COLLECTION DATE/TIME	ARRIVAL DATE	PARAMETER REQUESTED

NOTES: _____

WHITE COPY: DEPARTMENT MANAGER YELLOW COPY: COORDINATOR

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HUNTER/ESE DEPT. SAMPLE RECEIPT-LOG

PROJECT COORDINATOR	PROJECT NAME	FIELD GROUP	SAMPLE SEQUENCE	ARRIVAL DATE	DELIVERED BY:	RECEIVED BY:	PRE-LOG ARR. NOTICE:

JDS2(11-28-88)

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EXHIBIT F1

COLD ROOM SAMPLE ARRIVAL LOGBOOK

DO NOT REMOVE FROM COLD ROOM

Date	Time	Project Name or Field Group	No. Containers or Sample Nos.	Coordinator or Fractions	Signature	Comments

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SAMPLE CHECK IN/OUT LOG

SAMPLE	FRACTION	DATE/INIT OUT	DATE/INIT IN	SAMPLE	FRACTION	DATE/INIT OUT	DATE/INIT IN

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ENVIRONMENTAL SCIENCE AND ENGINEERING, INC.
P.O. BOX ESE
GAINESVILLE, FL. 32602
(904) 332-3318

ORGANICS

CONTRACT LABORATORY PROGRAM
SAMPLE CONTROL RECORD
CASE # _____

ORGANICS

EPA SAMPLE NUMBER	DATE & TIME REMOVED	REMOVED BY	FRACTION	REASON AND DESTINATION	DATE & TIME RETURNED

DO NOT DUPLICATE

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ESE
P.O. Box 1703
Gainesville, Fl. 32602

Exhibit I

Radiochemistry
Sample Storage and Custody
Log Sheet

Field Group	Sample Number	Delivery Date / Time	Check Out	Check In	Purpose of Removal
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	
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			Time / Date Analyst	Time / Date Analyst	
			Time / Date Analyst	Time / Date Analyst	

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SAMPLE STORAGE ORGANIZATION
STANDARD OPERATING PROCEDURE
SOP 4123-01

This Standard Operating Procedure (SOP) describes how ESE stores samples in coldrooms.

DATE: 9/16/87 AUTHORIZED BY: John J Mauer

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C.3

Sample Storage Organization
SOP 4123-01

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SAMPLE STORAGE ORGANIZATION

SOP 4123-01

1.0 ABSTRACT

This Standard Operating Procedure (SOP) describes how ESE stores samples in coldrooms.

2.0 PROCEDURE

Assign shelf space for each fraction and place the samples on the assigned shelves. Enter the shelf assignments into the CLASS computer network by logging into CONTROL (see SOP for CONTROL) and choosing coldroom management from the miscellaneous menu (#9). Produce a sample location report from the coldroom menu and post a copy on the coldroom door. When samples are thrown away, the coldroom technician will delete them from the location report.

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COLDROOM STORAGE QUALITY CONTROL
STANDARD OPERATING PROCEDURE
SOP 4123-02

This Standard Operating Procedure (SOP) describes ESE's coldroom quality control.

DATE: 11/6/89 AUTHORIZED BY: John J. Moore

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C.4

Coldroom Storage Quality Control
SOP 4123-02

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COLDROOM STORAGE QUALITY CONTROL
STANDARD OPERATING PROCEDURE
SOP 4123-02

1.0 ABSTRACT

This Standard Operating Procedure (SOP) describes ESE's coldroom quality control.

2.0 PROCEDURE

The coldroom will be locked with a combination lock at all times unless someone is inside. The combination will be known by chemistry personnel only. A sign-out sheet will be posted outside the coldroom, and anyone removing samples will list the samples removed from the coldroom (or returned) along with the person's name and date. The coldroom's interior temperature will be read each morning. The temperature will be recorded in the coldroom temperature book. If the temperature deviates from 4 degrees Celsius (°C) by more than ± 2°C, the maintenance department will be immediately notified to repair the cooling system.

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EXHIBIT E

DEPARTMENT FRACTION-RECEIPT LOG SHEETS

(See SOP 4101-08)

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C.5

Preventative Maintenance
SOP 4101-05

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PREVENTIVE MAINTENANCE
STANDARD OPERATING PROCEDURE
SOP 4101-05

This Standard Operating Procedure (SOP) describes how the ESE Chemistry Division regularly services its analytical instrumentation to help prevent data error or instrument failure.

DATE: 9/16/87 AUTHORIZED BY: John J. Meuse

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PREVENTIVE MAINTENANCE

SOP 4101-05

1.0 ABSTRACT

This Standard Operating Procedure (SOP) describes how the ESE Chemistry Division regularly services its analytical instrumentation to help prevent data error or instrument failure.

2.0 PROCEDURES

To minimize the occurrence of instrument failure and other system malfunction, the ESE Chemistry Division conducts a preventive maintenance program for field and laboratory instruments. Preventive maintenance performed for major pieces of field and analytical equipment is listed below.

2.1 YSI-SCT METER AND PROBE

Preventive maintenance protocol involves red-lining the meter to check the condition of the batteries. Probe preventive maintenance involves verifying temperature readings using a mercury thermometer and verifying that the probe does not need cleaning. A fouled probe is discovered by measuring a standard on the X100 and X10 ranges, then depressing the CELL TEST button. If the meter reading falls more than 2 percent, the probe is fouled and will be cleaned. Replacement membranes will be kept onsite.

2.2 pH METERS AND COMBINATION pH ELECTRODES

Preventive maintenance for the pH meter and electrodes primarily involves the proper care of the electrode. Electrodes are stored in a 1:1 solution of pH 7 buffer and deionized water. The hole used to add internal filling solution must be plugged at all times to prevent evaporation of the solution when the electrode is not in use. When the internal filling solution has dried out, the chamber will be rinsed with

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D.3

Comparison of USATHAMA/CLP Data Packages

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Page_____ of_____

METALS SUBSAMPLE CHAIN-OF-CUSTODY
(Continued)

Project Name:_____
Project Number:_____
Analytical Method Number:_____

Lot Number:_____

Sample Number	Spiking Information/Additional Comments

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CONTRACT LAB (SOW 788) DOCUMENT INVENTORY

1. Chain File Document Inventory Sheet
2. Chain-of-Custody Records
3. Shipping Manifests
4. Sample Tags
5. SMO Inorganics/Organics Traffic Reports
6. Inorganics Analysis Data Summary Sheets
7. Analysts' Notebook Pages
8. Instrument Logbook Pages

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D.3.a Comparison of Document Inventories

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USATHAMA

TABLE OF CONTENTS - METALS

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(to be supplied by Information Services)

_____1. Army Data Review Form

_____2. IRDMS Transfer File

_____3. Results of IRDMS Record and Group Check

B. ANALYST DOCUMENTS

_____1. Method Summary (supplied by Info. Services)

_____2. ESE Data Batch Report, with:
a. Analyst Signature
b. Computer Checklist
c. Manual Checklist
d. Example Calculation

_____3. Completed Control Chart and Comment/Corrective Action Form

_____4. Copies of Instrument Logbook Pages (with analytical conditions)

_____5. Raw Data, with:
a. Responses (emission/area counts, peak heights, etc.)
b. Calibration Curve
c. Changes made properly and initialed.

_____6. Copies of Sample Extract/Prep Logbook Pages

_____7. Chain-of-Custody and Possession Records
a. Field Chain-of-Custody Sheets (supplied by Info. Services)
b. Laboratory Subsample Chain-of-Custody

C. APPENDIX

_____1. Preliminary Data, Unused Data (if applicable)

_____2. Laboratory Coordinator and QA Review Files

_____3. Validation File in USATHAMA Format

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A.1

ARMY DATA REVIEW FORM

ARMY LOT _____ INSTALLATION _____

DATA BATCH _____ METHOD _____

REQUIRED LEVEL TWO DATE _____

	DUE DATE	INITIALS	DATE	COMMENTS
DATA COORDINATOR				
GROUP LEADER				
TASK MANAGER				
DATA COORDINATOR				
QA COORDINATOR DATA (VALIDATION COMPLETE)				
(PACKET COMPLETE)				
DATA COORDINATOR (FILE TRANSMITTED)				

OMMENTS :

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DOCUMENT INVENTORY FOR:

_____

ESE (contract lab.)

ENCLOSED ARE THE FOLLOWING ITEMS IN ORDER OF APPEARANCE:

		NO. OF PAGES
1)	EXPLANATION PACKET OF THE ITEMS BELOW.	_____
2)	DELIVERY ORDER/S:	
	A) _____	_____
	B) _____	_____
	C) _____	_____
	D) _____	_____
	E) _____	_____
3)	SAMPLE ARRIVAL/ CUSTODY FORMS;	_____
4)	ANALYSIS REPORT (ESE FORMAT);	_____
5)	BATCH REPORT;	_____
6)	LOT REPORT;	_____
7)	LOT SUMMARY (LOT, TYPE OF ANALYSIS, SAMPLES IN LOT, ETC.);	_____
8)	CHEMICAL SUMMARY REPORT;	_____
9)	COMMUNICATION (DISPOSITION LETTERS, ETC.);	_____
10)	LOTS IN REPORT; NO. OF LOT FOLDERS	_____

COMMENTS:

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D.3.b Organic Data Packages comparison

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USATHAMA

TABLE OF CONTENTS - GC/MS METHODS

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_____3. Results of IRDMS Record and Group Check

B. ANALYST DOCUMENTS

_____1. Method Summary (supplied by Info. Services)

_____2. ESE Data Batch Report, with:
- a. Analyst Signature
- b. Computer Checklist
- c. Manual Checklist

_____3. Completed Control Chart and Comment/Corrective Action Form

_____4. Laboratory Chronicle:
- a. ESE GC/MS Instrument Logsheet
- b. Copies of CLP/Pesticides Calibration Standards Notebook Pages
- c. Extraction Data Sheet

_____5. Chain-of-Custody and Possession Records
- a. Field Chain-of-Custody Sheets (supplied by Information Services)
- b. Laboratory Chain-of-Custody Sheets

_____6. Initial Calibration Forms
- a. Five point Initial Calibration with RFS, RF and % RSD (Form VI)
- b. Quantitation Report, Total Ion Chromatograms and Spectral Comparison

_____7. GC/MS Performance Tuning (Form V)

_____8. Continuing Calibration Forms
- a. Beginning Run (50 ug/mL)
  - * Form VI - Daily RF, VS, Initial RF
  - * RF list used to Quantitate Targets Analyses
  - * Quantitation Report, Total Ion Chromatograms and Spectral Comparison
- b. End Run (50 ug/mL)
  - * Form VI - Daily RF, VS, Initial RF
  - * Quantitation Report, Total Ion Chromatograms and Spectral Comparison

_____9. GC/MS Chromatograms and Spectral Data (for sample, matrix spikes and blank)
- a. Quantitation Reports, Total Ion Chromatograms and Spectral Comparison

_____10. One or two Examples of Calculations Performed

_____11. Pre-screen Raw Data (if applicable)

C. Appendix

1. Preliminary Data, Unused Data (if applicable)
2. ESE Review Files (QA and Lab Coordinator)
3. USATHAMA Review Files (QA and Lab Coordinator)

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USATHAMA & EPA

ORGANIC DATA PACKAGE COMPARISON

The same basic information is contained in both data packages. The main differences are in format and presentation of various specific information.

EPA organic packages contain all volatiles, BNA and pesticide/PCB results in one package. In contrast, USATHAMA packages are grouped by method and each package contains all custody and supporting information for that method.

Presentation of quality control data is another major difference. In EPA packages, all QC data are tabulated and presented on forms in Section III and VI of the data package. Control Charts (submitted weekly) are USATHAMA's method of presenting and reporting QC data.

Attached are basic outlines of EPA and USATHAMA data packages. A list of EPA-CLP form descriptions is cross referenced to show where each item is contained in the USATHAMA package.

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CONTRACT LAB (SOW 788) FORM DESCRIPTIONS

1. CASE NARRATIVE

2. TRAFFIC REPORTS

3. VOLATILES DATA

A. QC Summary		
1) Surrogate % Recovery Summary	(FORM II)	B.2
2) MS/MSD Summary	(FORM III)	NA
3) Method Blank Summary	(FORM IV-VOA)	B.2
4) GC/MS Tuning/Mass Calibration	(FORM V)	B.7
B. Sample Data (arranged in packets in increasing EPA Sample Number)		
1) TCL Results - Analysis Data Sheet	(FORM I)	B.2
2) Tentatively Identified Compounds	(FORM I-TIC)	TIC Lot Folder
3) Reconstructed Total Ion Chromatogram		B.9
4) Raw Data		
a) Copies of raw spectra and background substance mass spectra of target compounds		
b) Copies of mass spectra of tentatively identified compounds with associated best match spectra		
C. Standard Data		
1) Initial Calibration Data	(FORM VI)	B.6
a) Standard reconstruction chromatograms and quantitation reports for initial calibration	(5-point)	
2) Continuing Calibration	(FORM VIII)	B.8
a) Reconstructed ion chromatograms and quantitation reports		
3) Internal Standard Area Summary	(FORM VIII)	B.9
D. Raw QC Data		
1) BFB or DFTPP with Bar graph Spectrum and Mass Listing		B.7
2) Blank Data		
a) Tabulated results	(FORM I)	B.2
b) Tentatively identified compounds	(FORM I-TIC)	
c) Reconstructed ion chromatograms and quantitation reports		

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d) TCL spectra with lab generated standard		
e) GC/MS library search spectra for Tentatively Identified Compounds		
f) Quatitation/Calculation of Tentatively Identified Compounds		
3) Matrix Spike Data		
a) Tabulated results	(FORM I)	NA
b) Reconstructed ion chromatograms and quantitation reports		Standard Control Spike
4) Matrix Spike Duplicate Data		B.2,B.9
a) Tabulated results	(FORM I)	NA
b) Reconstructed ion chromatograms and quantitation reports		

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1. SEMI-VOLATILES DATA

A. QC Summary		
1) Surrogate % Recovery Summary	(FORM II)	B.2
2) MS/MSD Summary	(FORM III)	NA
3) Method Blank Summary	(FORM IV-VOA)	B.2
4) GC/MS Tuning/Mass Calibration	(FORM V)	B.7
B. Sample Data (arranged in packets in increasing EPA Sample Number)		
1) TCL Results - Analysis Data Sheet	(FORM I)	B.2
2) Tentatively Identified Compounds	(FORM I-TIC)	TIC Lot Folder
3) Reconstructed Total Ion Chromatogram		B.9
4) Raw Data		
a) Copies of raw spectra and background substance mass spectra of target compounds		
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a) Reconstructed ion chromatograms and quantitation reports		
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D. Raw QC Data		
1) BFB or DFTPP with Bar graph Spectrum and Mass Listing		B.7
2) Blank Data		
a) Tabulated results	(FORM I)	B.2
b) Tentatively identified compounds	(FORM I-TIC)	
c) Reconstructed ion chromatograms and quantitation reports		
d) TCL spectra with lab generated standard		
e) GC/MS library search spectra for Tentatively Identified Compounds		
f) Quatitation/Calculation of Tentatively Identified Compounds		

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3) Matrix Spike Data		
a) Tabulated results	(FORM I)	NA
b) Reconstructed ion chromatograms and quantitation reports		Standard Control Spike
4) Matrix Spike Duplicate Data		B.2,B.9
a) Tabulated results	(FORM I)	NA
b) Reconstructed ion chromatograms and quantitation reports		

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5. PESTICIDE/PCB DATA (GC/ECD)

A. QC Summary
   1) Surrogate % Recovery Summary (FORM II)
   2) MS/MSD Summary (FORM III)
   3) Method Blank Summary (FORM IV)

B. Sample Data (arranged in packets in increasing EPA Sample Number)
   1) TCL Results - Analysis Data Sheet (FORM I)
   2) Copies of chromatograms
   3) Copies of chromatograms from 2nd column confirmation
   4) Integration Reports
   5) Manual Work Sheets
   6) GPC chromatograms
   7) GC/MS confirmation raw spectra and background subtracted mass spectra

C. Standard Data
   1) Pesticide Evaluation Standards Summary (FORM VIII)
   2) Pesticide/PCB Standards Summary (FORM IX)
   3) Pesticide/PCB Identification for positive results (FORM X)
   4) Standard chromatograms and data system printouts

D. Raw QC Data
   1) Blank Data
      a) Tabulated results (FORM I)
      b) Chromatograms and data system printouts
   2) Matrix Spike Data
      a) Tabulated results of non-spike TCL compounds (FORM I)
      b) Chromatograms and data system printouts
   3) Matrix Spike Duplicate Data
      a) Tabulated results of non-spike TCL compounds (FORM I)
      b) Chromatograms and data system printouts

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USATHAMA

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3. USATHAMA Review Files (QA and Lab Coordinator)

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D.3.c Inorganic Data Package Comparison

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USATHAMA & EPA

INORGANIC DATA PACKAGE COMPARISON

The same basic information is contained in both data packages. The main differences are in format and presentation of various specific information.

EPA-CLP inorganic packages contain all metals and inorganic analytes in one package. In contrast, USATHAMA packages are grouped by method and each package contains all custody and supporting information for that method.

Presentation of quality control data is the other major difference. In EPA packages, all QC data are tabulated and presented on forms in Section I of the data package. Control Charts (submitted weekly) are USATHAMA's method of presenting and reporting QC data.

Attached are basic outlines of EPA-CLP and USATHAMA inorganic data packages. A list of the EPA-CLP form descriptions is cross referenced to show where each item is contained in the USATHAMA package.

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CONTRACT LAB (SOW 788) FORM DESCRIPTIONS

1.	Cover Page - Inorganic Analyses Data Package Lists all samples analyzed within a Sample Delivery Group and general comments. Signed by the Laboratory Manager.	[COVER PAGE-IN]	
2.	Inorganic Analysis Data Sheet Used to tabulate and report sample analysis results for target analytes. (One form for each sample)	[FORM 1-IN]	
3.	Initial and Continuing Calibration Verification	[FORM II(PART 1)-IN]	B.2,B.5
4.	CRDL Standard for AA and ICP Reports analyte recoveries from analyses of the CRDL standards for AA (CRA) and ICP (CRI).	[FORM II(PART 2)-IN]	NA
5.	Blanks Used to report ICB, CCB, PB.	[FORM III-IN]	B.2,B.5
6.	ICP Interference Check Sample	[FORM IV-IN]	B.2,B.5
7.	Spike Sample Recovery	[FORM V(PART 1)-IN]	B.3
8.	Post Digestion Spike Sample Recovery	[FORM V(PART 2)-IN]	NA
9.	Duplicates	[FORM VI-IN]	B.3
10.	Laboratory Control Sample Reports recoveries for solid and aqueous LCS (taken through digestion process).	[FORM VII-IN]	B.3
11.	Standard Addition Results (furnace)	[FORM VIII-IN]	NA
12.	ICP Serial Dilution	[FORM IX-IN]	NA
13.	Instrument Detection Limits (Quarterly)	[FORM X-IN]	B.1
14.	ICP Interelement Correction Factors (Annually)	[FORM XI(PART1)-IN]	
15.	" " " " "	[FORM XI(PART2)-IN]	

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16.	ICP Linear Ranges (Quarterly)	[FORM XII-IN]	B.1
17.	Preparation Log	[FORM XIII-IN]	B.6
18.	Analysis Run Log	[FORM XIV-IN]	B.4

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CONTRACT LAB (SOW 788) DOCUMENT INVENTORY

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2. Chain-of-Custody Records
3. Shipping Manifests
4. Sample Tags
5. SMO Inorganics/Organics Traffic Reports
6. Inorganics Analysis Data Summary Sheets
7. Analysts' Notebook Pages
8. Instrument Logbook Pages

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Lot No.:_____

B1

SUMMARY OF METHOD

NUMBER	TITLE	QC REFERENCE
ID19	Analysis of Arsenic in Soils by GFAA.	USATHAMA

DIGESTION PROCEDURE:

1. Transfer a 1.0 g aliquot of well-mixed sample to a 150 mL beaker.

2. Prepare 1000 ug/L spike solution: transfer 10 mL of a 10 mg/L As solution to a 100 mL volumetric flask, add 10 mL conc. HNO_3 & dilute to volume.
Give an aliquot of the spike solution to an ICAP operator for verification.

3. Prepare quality control samples:
   a. Method blank - a 0.1 g aliquot of standard sandy soil.
   b. Low spike - add 0.5 mL of the spiking solution to a 0.1 g aliquot of standard sandy soil. [target = 0.5 ug/g (5.0 ug/L)]
   c. High spikes - add 8.0 mL of the spiking solution to 2-0.1 g aliquots of standard sandy soil [target = 8.0 ug/g (80 ug/L)]

4. Add 10 mL (1+1) HNO_3 to all samples and QC. Cover with a watch glass and reflux for 10-15 minutes without boiling. Cool and add 5 mL conc. HNO_3. Cover and reflux for 30 min. Volume should not be reduced to less than 5 mL. Again add 5 mL conc. HNO_3 and reflux for 30 min. Cool. Add 2 mL type I water and 3 mL 30% H_2O_2. Cover and heat. Remove beaker from hot plate if effervescence is excessive. Heat until effervescence subsides. Cool and add 1 mL aliquots of H_2O_2 and heat until appearance is unchanged or effervescence is minimal. Do not add more than 10 mL H_2O_2. Cool, add 10 mL type I water and reflux for 15 min. without boiling. Cool, filter and adjust final volume to 100 mL with type I water.

   NOTES:

   Signed _____ Date _____

INSTRUMENT ANALYSIS:

1. Instrument: 5100

2. Calibration curve: 100, 50, 25, 5.0, 2.5, 0.0 ug/L (linear). Prepare 100 and 10 ug/l standards for instrument dilution. Certified reporting limit = 0.25 ug/g (2.5 ug/L), upper certified limit = 10.0 ug/g (100 ug/L). Note: Standards should be 10% HNO_3.

3. Modifier: palladium nitrate + magnesium nitrate.

4. QC: analyze a reference at the beginning and end of the run and reanalyze the 100 ug/l standard after every 10 samples and at the end of the run. Recoveries must be ± 10%.

   NOTES:

   Signed _____ Date _____

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JD19 Method Summary cont.

ESE DATA ENTRY

1. Enter calibration standards (even if data is being reported in final concentration). Do not enter a detection limit - it will be supplied by Information Services.

2. If blank correction is being applied to the soil samples, an appropriate explanation must be supplied.

3. Enter a DA sample with the same field group as the method blank. If blank correction is being applied the "DA Blank" response should be twice the MB response. If the MB has a negative response or the response is less than the detection limit, enter the response as 0 Final.

4. Use 10X the actual standard soil weight for the method blank, DA blank, and spike calculations.

5. Responses and targets must be entered in mg/L.

6. Generate spike recovery/control chart data for Information Services.

NOTES:

Signed _____ Date _____

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USATHAMA - Metals
Computer QC Checks

Batch No.:_____ Lot No.:_____ Analysis Date:_____ Analyst:_____

	YES	NO	Comment/Corrective Action
Extract holding time within criteria?			
Analysis holding time within criteria?			
No. of calibration standards present acceptable?			
Curve correlation coefficient >= 0.995?			
Sample responses within the highest standard response?			
Interference Check Solution (ICS) present? ICS within acceptance criteria?			
Initial Calibration Verification solution (ICV) present? ICV within acceptance criteria?			
End of Run Calibration Verification solution (ECV) present? ECV within acceptance criteria?			
Continuing Calibration Verification solution (CCV) present? CCV within acceptance criteria?			
Continuing Calibration Blank (CCB) present?			
Method blank present?			
Standard matrix spikes present? Standard matrix spike targets correct?			

Note: Any "NO" answer requires a comment.

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USATHAMA
Manual QA/QC Batch Checklist for Atomic Spectroscopy

Batch No.:____ Lot No.:_____ Analysis Date:_____ Analyst:_____

	N/A	YES	NO	Comment/Corrective Action
Are reported values consistent with the required units?				
Do the calibration standards bracket the concentration equivalent to the method detection limit and the upper certified concentration?				
Is the calibration curve entered correctly?				
If sample concentrations above the certified range are reported, were extracts diluted to within the certified range prior to analysis?				
Are dilution factors entered correctly?				
Have reported sample concentrations been adjusted for percent moisture and sample volume/weight?				
If sample concentrations have been corrected for the method blank, was an appropriate explanation supplied?				
Was the batch checked for data entry errors?				
Were continuing calibration standards (CCV) analyzed at the appropriate frequency?				
Were continuing calibration blanks (CCB) analyzed at the appropriate frequency?				
Is an example calculation provided?				
Has the lot table of contents/checklist been completed?				

Note: Any "NO" answer requires a comment.

Completed By_____
Date_____

Reviewed by_____
Date_____

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APPENDIX E

Data Management SOPs

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E.1

Laboratory Data Management

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LABORATORY DATA MANAGEMENT

ESE has developed the Chemical Laboratory Analysis and Scheduling System (CLASS), c. 1989., which is an automated in-house data management system that integrates information from sample collection, laboratory analyses, and QC requirements and calculates, checks, stores, and reports data in many different formats. The CLASS system resides on a Novell Arcnet PC network with 1600 megabytes of disk storage, and is connected to 65 PC's placed throughout the chemistry laboratories and offices. ESE has incorporated into this system, the ability to combine analytical results, method certification, QC, and site-specific data in order to produce files in a format acceptable to USATHAMA data checking routines. The way in which data will be handled for this project is shown in detail in Fig. 3-1.

USATHAMA QC procedures have been integrated into ESE CLASS. The data system manages the flow of samples through the laboratory. Prior to sampling, the laboratory coordinator provides information on the number of samples, site identifications, parameters to be analyzed, and estimated collection date. This information is entered into CLASS and is used to produce sample labels and chain-of-custody logsheets (Figs. 3-2 and 3-3). A unique ESE number is assigned to each sample, and labels with that number are placed on each container for that sample. Site identification will be on the labels and logsheets to ensure the integrity of the chain-of-custody and tracking system.

At each site, samples are collected and placed in the appropriate pre-labeled containers. The logsheet is marked, noting the collection date and time and recording the sample type, site type, sample depth, and sampling technique.

After collection, samples accompanied by the logsheet are sent to the laboratory and received by the laboratory coordinator. The laboratory coordinator checks the sample containers against the information recorded on the logsheet and notes any discrepancies. The laboratory coordinator submits the logsheet to data management personnel. Samples are logged into the CLASS system by the data management personnel. Sample collection date and time are entered so that holding times can be readily monitored.

ESE uses a combination of U.S. Environmental Protection Agency (EPA) Storage and Retrieval (STORET) numbers and USATHAMA method codes to designate parameters required for analysis. For example, arsenic in water would be denoted by 1002*SD03, since the STORET number for arsenic is 1002 and the USATHAMA certified method code is SD03. A list of all required parameters is logged into the computer with each sample. Each STORET-method

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combination has its own QC requirements specific to that analysis.

Each list that is set up on the system includes the following data entries to facilitate converting data into the IRDMS format.

ESE and EPA STORET numbers	Parameters
71999	Sample Type
99759	Site Type
72015	Sample Depth [Feet (FT)]
72005	Sampling Technique
99720	Installation Code

The information is taken from the field log sheet and entered into CLASS so that it can be incorporated into the chemical data record.

The sampling information is entered into the computer to activate the parameter list for the samples collected and received by the laboratory. A notice of sample arrival and short holding times is immediately sent to the analytical team leaders. The report of samples available for each analysis will indicate the number of days left before the holding time is exceeded. This report is distributed to each analytical department daily, and the information also can be accessed readily from CLASS by the laboratory coordinator or any analyst in the Chemistry Division. Samples are stored in a locked coldroom and can be removed only by signing the appropriate chain-of-custody form.

SAMPLE IDENTIFICATION NUMBERS

ESE uses a batch method for analyzing, checking QC, and calculating final results of samples. Prior to analyzing a batch of samples, the analyst will designate a specified group of samples in the computer and the sample-parameter status will be updated to IL (in laboratory). The basis for this grouping technique is that samples are analyzed in groups for the same method that can be extracted or analyzed (whichever is the most limiting factor) in one 24-hour period. The analytical batch is assigned a unique batch control number, which is stored with all final data, so data can be checked and original documentation can be retrieved. For USATHAMA projects, a 3-character alphabetical lot designation also is assigned to each analytical batch. Only USATHAMA samples will be included in any batches that contain USATHAMA samples. Each sample in the batch is assigned a numerical suffix, starting with 001 to the 3-character

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alphabetical prefix. Using this scheme, there will be a direct relationship between ESE's unique sample number and the USATHAMA sample number for each lot.

REQUIREMENTS FOR SAMPLE ANALYSIS AND DATA INTERPRETATION

Samples will be analyzed within the holding times for each method. The analysis of each laboratory batch usually consists of a multistage process. In the first stage, instrument calibration, the ESE data management system includes several QC checks. The linear or quadratic regression equation and correlation coefficient are calculated from the calibration curve data, and the correlation coefficient is tested to determine whether it is within an acceptable range specific to the analysis. In addition, the constancy of the calibration response is checked by comparing preanalysis and postanalysis standard response and/or response factors. Method blank and control spike information are then entered and results are calculated and checked against control limits as calculated for that method by certification and prior lots. Sample responses are entered into the batch, and final concentrations are calculated for each sample.

Software has been developed to interface output of data from some laboratory instruments directly into CLASS, thus minimizing typographical errors. These instruments include:

1) GC/MS for both volatile and semi-volatile analysis.
2) ICP and Graphite Furnace.
3) Ion chromatography and Tracs autoanalyzer.

If the data fail the QC checks, appropriate actions (such as reanalyzing the samples or correcting transcription or data reduction errors) are taken.

Analysts use the computer to reserve samples for analysis and to interactively check calibration curves and QC results. By allowing the analyst to enter data directly and check QC and sample results, the analyst is notified immediately of QC problems. Thus, any necessary corrective actions can be taken before more analyses are completed. Laboratory analysts are not permitted to update sample records. When the analyst has entered the QC and sample data, the Army lot, including worksheets and any other pertinent documentation (such as chromatograms), is placed in a file folder and submitted to the data management supervisor. The computer produces a batch summary report by incorporating chemical, field, and certification data, and a file formatted so that it can be read into and checked by the IRDMS data checking routines. These printouts, along with the data

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package from the laboratory analyst, are placed in a folder and attached to the Army data transmittal form (Fig. 3-5) for review and approval by the team leader, the laboratory coordinator, and the QA supervisor. The QA supervisor will then validate the data by checking random data points against raw data.

After review and QA validation are complete for a lot, the data management personnel will enter the formatted file into the IRDMS data acceptance routines for record and group checking.

Record check verifies that:

1. Data are correctly formatted,
2. The laboratory is certified for the method,
3. Test names are valid for the method,
4. Concentrations are within certified range or properly diluted within range.

Group check verifies that:

1. A lot contains the correct number of QC samples,
2. All analytes for the method are present, and
3. All sites in a lot have a map record in the database.

When all checks are passed, a transfer file will be created from the system and sent to the USATHAMA computer via telephone lines.

USATHAMA will run further data checks when ESE data files are received. After passing the checks, the files will be elevated to Level 2.

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DATA TRACKING

ESE understands the importance to USATHAMA of maintaining task order analysis schedules. Data generated in a timely manner are critical to decision making concerning remedial actions and also for meeting regulatory reporting deadlines on monitoring efforts. A coordinated functional mechanism for maintaining analytical data reporting schedules is critically important.

ESE uses tested mechanisms to:

* Notify analytical task managers and team leaders of the arrival of samples for analysis in a timely manner;
* Track the status of samples through the laboratory analysis and review procedures;
* Monitor compliance with sample holding times via a computerized tracking procedure; and
* Communicate delays in the sample analysis and/or review chain to project management in a timely manner to implement effective corrective actions.

AVAILABLE NUMBERS REPORT

As part of ESE CLASS, each analytical team leader receives a daily listing of all samples requiring analysis by a particular method. Samples appear on this report within 24 hours of receipt in the laboratory and after log-in into CLASS. In addition to providing notification of sample availability for analysis, the available sample numbers report also provides the team leader and analyst a list of the number of days left in order to meet the required holding times for the analysis and the number of days before the data are due.

In addition, the ESE laboratory has a person designated as the Sample Tracking Assistant (STA) whose responsibilities include the daily checking of the status of samples which are nearing holding times for extraction and/or analysis. If a sample has only 1 day (3 days if on a weekend) left before holding time will be exceeded, the STA notifies the analytical team leader and the laboratory task manager in writing. Followup and corrective action may then be instituted.

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CHEMTRAK

USATHAMA lots are tracked through the analytical laboratory and data review chain using a PC-based computer program called Chemtrak. This program was written especially for USATHAMA projects by ESE to provide a management tool for laboratory coordinators and project managers to identify problem areas causing
schedule slippage. The program functions as follows:

* As soon as lot assignments are made, the following information is entered into the computer: lot number, analytical method, installation, and date of earliest sample in the lot;

* From that information, the program calculates the date the completed analytical lot is due from the laboratory to the data management supervisor for processing and the dates due for completion of supervisory and QA review based on the required schedule (expedited, 40 or 55-day schedule) for the data to be through the IRDMS data checking routine and elevated into Level 2; and

* Weekly reports are produced by method and by installation to indicate the status of lots not completely through the system. These reports are circulated to analytical department managers, laboratory coordinators, and QA staff.

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JEFFERSON DMP BASE

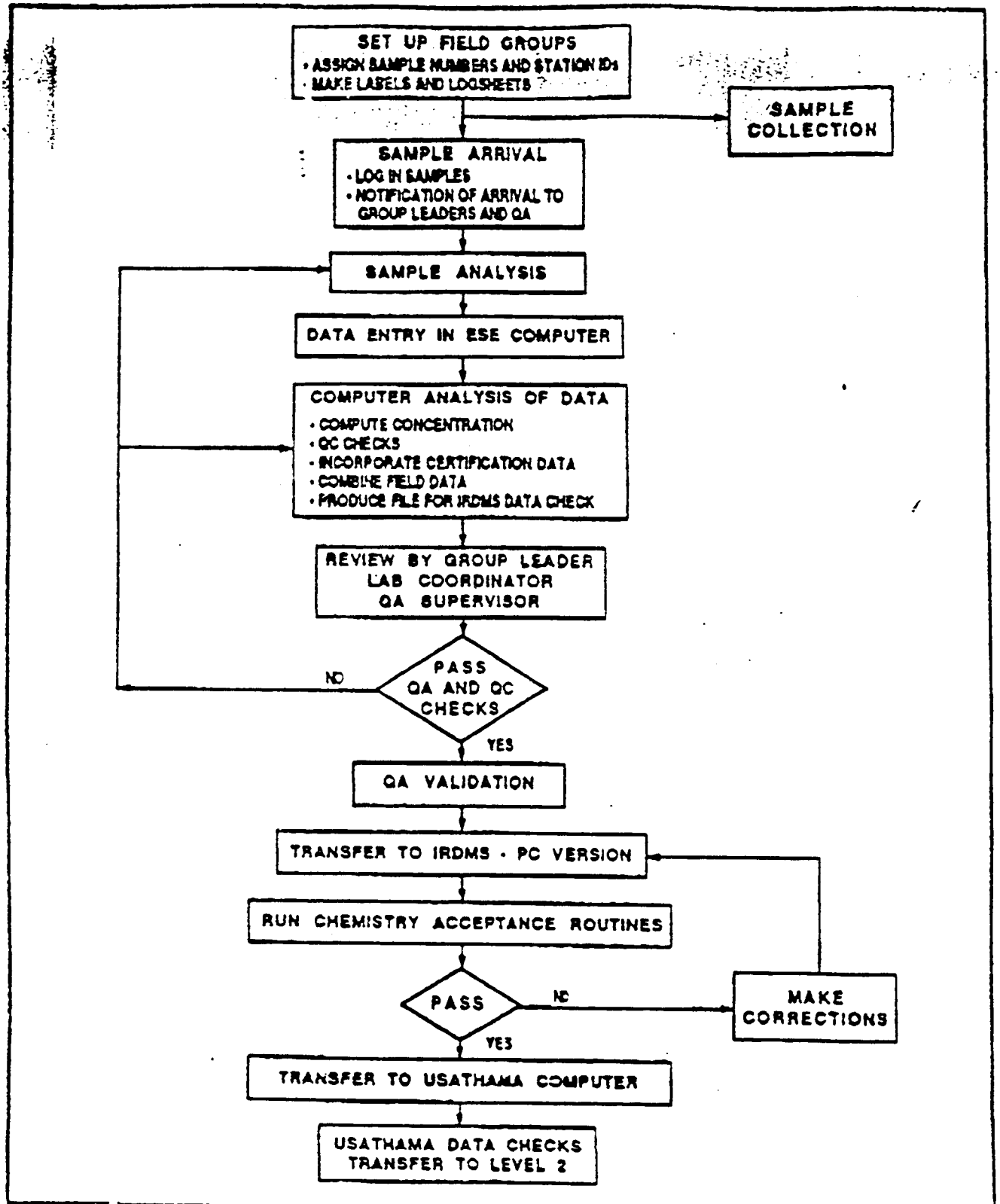

Figure 3-1
OVERVIEW OF DATA MANAGEMENT PROCEDURES FOR USATHAMA ANALYSES

SOURCE: ESE, 1988

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ESE#86536 0942 507A
SHARPE ARMY DEPOT
SHMWP1#1-VP
SAMPLER DATE TIME
PH COND

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SHARPE ARMY DEPOT
SHMWP1#1-VP
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SHARPE ARMY DEPOT
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ESE#86536 0942 507A
SHARPE ARMY DEPOT
SHMWP1#1-C
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PH COND

ESE#86536 0942 507A
SHARPE ARMY DEPOT
SHMWP1#1-HB
SAMPLER DATE TIME
PH COND

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SHARPE ARMY DEPOT
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SHARPE ARMY DEPOT
SHMWP1#2-VP
SAMPLER DATE TIME
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SHARPE ARMY DEPOT
SHMWP1#2-VP
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SHMWP1#2-C
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SHMWP1#2-HB
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ESE#86536 0942 507B
SHARPE ARMY DEPOT
SHMWP1#2-HB
SAMPLER DATE TIME
PH COND

Figure 3-2
ESE SAMPLE LABLES

SOURCE: ESE 1988

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ENVIRONMENTAL SCIENCE & ENGINEERING 05-07-87 *** FIELD LOGSHEET *** FIELD GROUP: SHMWP1
PROJECT NUMBER 86536 0942 PROJECT NAME: SHARPE ARMY DEPOT LAB COORD. JACKIE HARGROVE

SHMWP1

ESE #	SITE/BTA	HAZ?	FRACTIONS(CIRCLE)	DATE	TIME	PARAMETER LIST	SAM TYPE	SITE TYPE	DEPTH	S TECH	INSTAL SAMPLE
*1	507A		VP VP VP VP NF C HB HB			SHAD-W					
*2	507B		VP VP VP VP NF C HB HB			SHAD-W					
*3	507C		VP VP VP VP NF C HB HB			SHAD-W					
*4	511C		VP VP VP VP NF C HB HB			SHAD-W					
*5	511A		VP VP VP VP NF C HB HB			SHAD-W					
*6	511B		VP VP VP VP NF C HB HB			SHAD-W					
*7	508A		VP VP VP VP NF C HB HB			SHAD-W					
*8	508B		VP VP VP VP NF C HB HB			SHAD-W					
*9	508C		VP VP VP VP NF C HB HB			SHAD-W					
*10	520A		VP VP VP VP NF C HB HB			SHAD-W					

NOTE -CHANGE OR ENTER SITE ID AS NECESSARY, UP TO 9 ALPHANUMERIC CHARACTERS MAY BE USED
-CIRCLE FRACTIONS COLLECTED. ENTER DATE,TIME,FIELD DATA (IF REQUIRED), HAZARD CODE AND NOTES
-HAZARD CODES: I IGNITABLE C CORROSIVE R REACTIVE T TOXIC WASTE H OTHER ACUTE HAZARD; IDENTIFY SPECIFICS IF KNOWN
-PLEASE RETURN LOGSHEETS WITH SAMPLES TO ESE

	RELINQUISHED BY: (NAME/ORGANIZATION/DATE/TIME)	RECEIVED BY (NAME/ORGANIZATION/DATE/TIME)
1		
2		
3		

OTHER FIELD NOTES FOR FIELD GROUP SHMWP1:

Figure 3-3
ESE SAMPLE LOG SHEET

SOURCE: ESE, 1988.

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