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Los Alamos NATIONAL LABORATORY EST.1943

Environment & Remediation Support Services

Standard Operating Procedure

for SAMPLE CONTAINERS AND PRESERVATION

APPROVAL SIGNATURES:

Subject Matter Expert: [redacted]	**Organization** ERSS-GS	**Signature**	**Date** 5/22/07
Quality Assurance Specialist: [redacted]	**Organization** QA-IQ	**Signature**	**Date** 5/22/07
Responsible Line Manager: P. Dwain Farley	**Organization** ERSS-RS	**Signature**	**Date** 5/23/07

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1.0 PURPOSE AND SCOPE

The purpose of this procedure is to delineate the responsibilities, specific requirements, and process for sample containers, preservation techniques, and holding times as specified by field regulations and guidance documents within the Los Alamos National Laboratory (LANL or Laboratory) Environment & Remediation Support Services (ERSS) group. This procedure is applicable to ERSS activities involving the collection and chemical and physical preservation of samples that will be taken to the ERSS Sample Management Office (SMO) for subsequent chemical or physical testing. Subcontractors performing work under any ERSS program will follow this SOP for Sample Containers and Preservation.

2.0 BACKGROUND AND PRECAUTIONS

2.1 Background

The use of specific types of sample containers and preservation techniques is mandatory for hazardous site investigations because the integrity of any sample is diminished over time. Physical factors (light, pressure, temperature, etc.), chemical factors (changes in pH, volatilization, etc.), and biological factors may alter the original quality of the sample. Because the various target parameters are uniquely altered at varying rates, distinct sample container, preservation techniques, and holding time have been established to maintain sample integrity for a reasonable and acceptable period of time.

2.2 Precautions

The volume of sample collected should be sufficient to perform all the required analyses, plus an additional amount to provide for any quality control needs, split samples, or repeat examinations. The volumes, preservatives, and holding times are listed in Attachment 1.

If the samples were collected in an area controlled by a Radiological Work Permit, they must be released by HSR-1 prior to transfer to the SMO. The samples shall be preserved and secured at the site until the shipping requirements are met and the samples are removed from the site. Consult procedure EP-ERSS-SOP-5057, *ERSS Handling, Packaging, and Transporting Field Samples*, for handling and transporting samples.

Never clean and re-use bottles. Keep bottles in clean, dry place until the sample has been collected and is ready to be transferred to the appropriate container.

3.0 EQUIPMENT AND TOOLS

• Certified 300 series sample containers; available from vendors such as I-CHEM (J-CHEM Certified ™ 300 Series), Environmental Sampling Supply (ESS), etc. [NOTE: A Certificate of analysis with a bar-coded production number is typically in every case supplies by the vendor. Each bottle in the 300 series has a bar-code label for absolute traceability and is for use with the automated sample tracking system. The certificate of analysis should be retained for records.]	• Chemical preservatives as shown in Attachment 1.

4.0 STEP-BY-STEP PROCESS DESCRIPTION

4.1 Obtaining Proper Sample Containers and Preservatives

Field Team Member	1.	See Attachment 1 for proper sample containers and preservatives.
	2.	Verify all materials are ready and available prior to going into the field, including all QC samples, such as trip blanks, field blanks, etc., that are required by the applicable Sample and Analysis plan (SAP).
	3.	Obtain sample collection logs (SCL), and print chain of custody forms (COCs) and individual bottle identification stickers prior to going in the field.
	4.	For sample collection, use only Certified 300 series sample containers that have been processed and meet or exceed "US EPA Specifications and Guidance for Contaminant-Free Sample Container" (Publication 9240.05A, EPA/540/R-93/051, December 1992). [NOTE: Certified 300 series sample containers are available from vendors such as I-CHEM (J-CHEM Certified ™ 300 Series), Environmental Sampling Supply (ESS), etc.] [NOTE: A Certificate of analysis with a bar-coded production number is typically in every case supplies by the vendor. Each bottle in the 300 series has a bar-code label for absolute traceability and is for use with the automated sample tracking system. The certificate of analysis should be retained for your records.]
	5.	Verify all water samples for organics contain extra aliquots for the potential of laboratory quality control problems and/or breakage during shipment.
	6.	Refer to the ERSS procedure EP-ERSS-SOP-5057, *Handling, Packaging, and Transporting Field Samples*, and follow all applicable transportation requirements.
	7.	Document all pertinent comments and any deviations on the sample collection log/chain of custody or Field logbook.

4.2 Collecting Samples

Field Team Member	1.	For all matrices, fill bottles in the following order: • Volatile organics; • Semi-volatile organics; • Metals; • Other inorganic parameters; and • Radiochemistry.
	2.	Take special consideration when sampling volatile organic constituents

Field Team Member (Continued)	3.	Follow the following vial filling techniques for volatiles: • Add the preservative before the sample is taken. • Pour liquid samples into the vials without introducing any air bubbles. • If bubbling occurs as a result of vigorous pouring, discard the sample and refill the vial. • Completely fill the vial at the time of sampling so that when the septum cap is fitted and sealed, and the vial is inverted, no headspace is visible. • Do not open appropriately filled vials again prior to analysis. [NOTE: Pea-sized bubbles may accumulate in the vials during transportation and storage due to solubility differences affected by temperature change. This should not adversely affect the sample integrity. This will happen during storage but should not be present at the time of sampling.]
	4.	Collect solid samples in the following manner: • Collect the solid sample in EnCore ™ samplers, or fill the specific jar as completely as possible; • Tap the sides of the jar slightly during filling to try and eliminate as much air space as possible; • If samples are shipped to the laboratory in EnCore ™ samplers, extrude the samples and place them in sample containers within 48 hours of sample collection.
	5.	Collect sludge samples in the following manner: • Take into consideration the consistency of the material since the analytical laboratory will extract or analyze the sample with respect to the relative pecent of liquid solid components; • If the sludge is mosly water with relatively low solid content (<40% solids), use the appropriate water sample containers; • If the specific analysis to be performed is only applicable to a certain fraction of the sludge, note this on the analytical request form.

4.3 Preserving Samples

Field Team Member	1.	Determine the type of preservation required for the specific analyses requested for all samples in accordance with EPA SW-846 and established industry practices for use by accredited analytical laboratories by using Attachment 1. [NOTE: Acid, base, or buffer preservative quantities to be added to samples shall be in accordance with Attachment 2.]

Field Team Member (Continued)	2.	Preserve samples immediately following sample collection (except in the case of samples for organics analyses as described above). [NOTE: The SMO does not provide or perform preservation capabilities.] [NOTE: The proper reagent for pH adjustment should be in an easily usable form that can be added at the time of sampling.]
	3.	Store samples in a cooler with ice, or other appropriate cooling material, until they are delivered to the SMO by using the following process: • Place the samples in an insulated container (cooler) and maintain on ice (ice in bags or chemical "blue" ice) at 4^{o} Centigrade within 8 hours of sample collection (where applicable); and • Avoid freezing the sample, particularly when using a small, < 40 ml glass container, by wrapping it in bubble pack to isolate it from the "blue" ice.
	4.	Check the pH with pH paper if using an acid or base preservative; however, never insert the pH paper directly into the sample vial.

4.4 Implementing Holding Times

Field Team Member	1.	Consider holding times and shipment schedules when collecting samples in order to minimize potential effects to samples due to holding time concerns. [NOTE: Holding times are shown in Attachment 1.]
	2.	Use the sample collection date and time for the beginning of the holding time: [NOTE: Both the sampler and the subcontract analytical laboratory must use this date/time. If the holding times are expressed in days, the sample must be extracted/analyzed before the time frames specified in Attachment 1 are exceeded. If the holding times are expressed in hours then the sample must be extracted/analyzed before the time frames expresses in Attachment 1 are exceeded. Remember to take into account time zone differences when collecting samples.]
	3.	When parameters are required to be analyzed in the field, use the allowable holding times listed in Attachment 1, which are the maximum times that samples are considered valid.
	4.	If the site has suspected radiation contamination, obtain radiation screening results for the SMO or BUS-4 to ship the samples. (See procedure EP-ERSS-SOP-5057, *Handling, Packaging, and Transporting Field Samples*, for handling and transporting the samples. [NOTE: These results may be from historical knowledge or may be derived from field screening measurements of gross alpha/beta and gross gamma.]
	5.	If the samples are collected in an area controlled by a Radiological Work Permit, obtain a release by HSR-1 prior to transfer to the SMO.

Field Team Member (Continued)	6.	Preserve and secure the samples at the site until the shipping requirements are met and the samples are removed from the site.

4.5 Records

Field Team Member	1.	Submit the following records generated by this procedure to the applicable Field Operations Task Leader • Daily Activity Log forms or field notebooks that include deviations (if applicable), calibration information, record of daily activities, and any other pertinent information, at a minimum; • Completed Chain-of-Custody Form; and • Sample Collection Log.

5.0 PROCESS FLOW CHART

None

6.0 ATTACHMENTS

Attachment 1 5056-1 Sample Preservation Techniques and Holding Times (6 pages)

Attachment 2 5056-2 Preservative Checklist (1 page)

7.0 REVISION HISTORY

Author: Keith Greene

Revision No. *[Enter current revision number, beginning with Rev.0]*	Effective Date *[DCC inserts effective date for revision]*	Description of Changes *[List specific changes made since the previous revision]*	Type of Change *[Technical (T) or Editorial (E)]*
1.0	07/29/05	New document derived from E-SOP-1.02 and WQH-SOP-020	E
0.0	10/16/07	New Document number, reformatted, minor technical changes. Supersedes ENV-DO-206	T, E

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ATTACHMENT 1: PRESERVATION TECHNIQUES AND HOLDING TIMES

5056-1

Preservation Techniques and Holding Times

Records Use only

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Method (use most recent version)	Parameters	Matrix	Volume/Container	Preservation [a]	Holding times [a]	
					Sample	Extract
305.1,310	Acidity, Alkalinity	Water	500 mL Plastic or glass	4 °C	14 Days	NA
300.0, 320.1, 325,340, 375	Bromide, Chloride, Fluoride, Sulfate	Water	1 L Plastic	4 °C	28 Days	NA
405.1	BOD	Water	1 L Plastic	4 °C	48 Hours	NA
9010B,9013, 9014, 335,1, 335.3	Total Cyanide Amenable Cyanide	Water	1 L Plastic	4 °C; NaOH; pH > 12	14 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	14 Days	NA
415, 9060	DOC, TOC	Water	250 mL Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
200.7, 200.8, 6010B, 6020	All metals except Cr(VI) and Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	180 Days	NA
		Solid/Other	250 mL Glass Jar		180 Days	NA
3060A, 7196A, 7197	Cr(VI)	Water	500 mL Plastic	4 °C	24 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	30 Days	24Hrs
245.1, 7470A, 7471A	Hg	Water	500 mL Plastic	HNO_3 ; pH < 2	28 Days	NA
		Solid/Other	250 mL Glass Jar	4 °C	28 Days	NA
130.1	Hardness	Water		HNO_3 ; pH < 2; 4 °C	180 Days	NA
345.1	Iodide	Water	500 mL Plastic or Glass	4 °C	24 Hours	NA
353, 351, 365.4, 350	Ammonium, Nitrate + Nitrite, Total Phosphorus, TKN	Water	1 L Plastic	4 °C; H_2SO_4; pH < 2	28 Days	NA
300.0 354.1	Nitrate, Nitrite, Ortho Phosphorus	Water	500 mL Plastic	4 °C	48 Hours	NA
365	Ortho Phosphorus	Water	500 mL Plastic	4 °C; H_2SO_4; pH < 2	48 Hours	NA

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9210/9211	Nitrate	Water	1 L Plastic	4 °C; 1M Boric Acid	48 Hours	NA
		Solid/Other	250 mL Glass Jar	4 °C	48 Hours	NA
314.0, 9058	Perchlorate	Water	250 mL Plastic or Glass	4 °C	28 Days	NA
8321A (modified)	Perchlorate by LC/MS/MS	Water	250 mL Plastic or Glass	4 °C	28 Days	60 days
		Solid	4 oz. Wide-mouth jar	4 °C	28 Days	60 days
410.x	Chemical Oxygen Demand (COD)	Water	250 mL Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
1664	Total Recoverable Oil and Grease	Water	1 L Glass	4 °C; H_2SO_4 or HCl; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9070/9071A	Total Recoverable Oil and Grease	Water	1 L Glass	4 °C; HCl; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
ASTM D-854	Specific Gravity	Water	500 mL Plastic or Glass	None	None	NA
376/9030B/ 9031	Sulfide	Water	1 L Glass	4 °C; NaOH; Zinc acetate; pH > 9	7 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	7 Days	NA
160.x	TDS, TSS, TS	Water	1 L Plastic	4 °C	7 Days	NA
160.4	volatile solids (volatile residue)	Water	Plastic or glass	4 °C	7 Day	NA

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9020B	TOX	Water	1 L Amber Glass	4 °C; H_2SO_4; pH < 2	28 Days	NA
		Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9060	TOC	Water	Glass	4 °C; H_2SO_4 or HCL; pH <2 if analyzed >2 hours after collection	2 hours, unless acidified	NA
418.1	TRPH	Water	1 L Amber Glass	4 °C; HCl; pH < 2	28 Days	NA
1664	TPH	Water	1 L Amber Glass	4 °C; H_2SO_4 or HCl; pH < 2	28 Days	NA
8440	TPH	Solid/Other	125 mL Glass Jar	4 °C	28 Days	NA
9065, 9066 , 420	Total Recoverable Phenols	Water	1 L Glass	4 °C; H_2SO_4; pH < 4	28 Days	NA
		Solid	125 mL Glass Jar	4 °C	28 Days	NA
150.1, 9040B	pH	Water	125 mL Plastic	4 °C	24 Hours	NA
110, 180.1	Color, Turbidity	Water	500 mL Plastic	4 °C	48 Hours	NA
120.1, 9050	Specific Conductance	Water	125 mL Plastic	4 °C	28 Days	NA
	All radiochemical parameters except Rn-222 and tritium	Water	1 L Plastic (2 x 2 L Preferred)	HNO_3 ; pH < 2	180 Days	NA
		Solid/Other	250 mL Glass Jar		180 Days	NA
913.0	Radon 222	Water	125 mL Glass	None	72 Hours	NA
906.0	Tritium	Water	1 L Glass	None	180 Days	NA
		Solid/Other	Sample size will vary with moisture content	None	180 Days	NA

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Preservation Techniques and Holding Times

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8015 (Modified)	Petroleum Hydrocarbons (Diesel Range Organics)	Water	2 x 1 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
	Petroleum Hydrocarbons (Gasoline Range Organics)	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA
8015 (Modified)	Petroleum Hydrocarbons (Gasoline Range Organics)	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	[b]14 days	NA
8021B	Halogenated Volatile Organics	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA
5035/8021B	Halogenated Volatile Organics	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	[b]14 days	NA
8081A, 8082	Organochlorine Pesticides, PCBs	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
8141A	Organophosphorous Compounds	Water	4 L Amber Glass Bottle	4 °C; NaOH or H_2SO_4; pH 5-8	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
8151A	Chlorinated Herbicides	Water	4 L Amber Glass Bottle	4 °C;	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
8260B	Volatile Organics by GC-MS	Water	3 x 40 mL Glass Vial	4 °C; HCl; pH < 2	14 Days	NA
		Soil/Other	125 mL Glass Jar	4 °C	14 Days	NA

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Preservation Techniques and Holding Times

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8260B	Volatile Organics by GC-MS	Soil	4 x 40 mL Glass Vial	4 °C, 2 Vials $NaHSO_4$ 1 Vial CH_3OH, 1 Vial No Preservative	[b]14 days	NA
8270C	Semi-volatile Organics by GC-MS	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
8280A	Polychlorinated Dioxins and Furans by GC/MS	Water	4 L Amber Glass Bottle	4 °C	30 Days	45 Days
		Soil/Other	250 mL Glass Jar	4 °C	30 Days	45 Days
8318	N-Methylcarbamate Pesticides by HPLC	Water	4 L Amber Glass Bottle	4 °C; 0.1 N $ClCH_2CO_2H$, pH 4 - 5	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	7 Days	40 Days
8330	Nitroaromatics and Nitramines by HPLC	Water	4 L Amber Glass Bottle	4 °C	7 Days	40 Days
		Soil/Other	250 mL Glass Jar	4 °C	14 Days	40 Days
TO-15	VOC in Air	SUMMA® Canister			28 Days (by consensus)	
8321A (modified)	High Explosives by LC/MS/MS	Water	Amber Glass/Teflon lined cap	4 °C	7 Days	40 Days
		Solid	Amber Glass/Teflon lined cap	4 °C	14 Days	40 Days
SW-8332	Nitroglycerine & PETN	Water	4 L Amber Glass	4 °C	7 Days	40 Days
		Solid/Other	250 mL Glass	4 °C	14 Days	40 Days

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Preservation Techniques and Holding Times

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350.1, 350.2, or 350.3	NH3 - Nitrogen (Ammonia)	Water	1L Plastic	4 °C; H2SO4 to pH<2	28 Days	N/A
370.1	Silica, dissolved (SiO2)	Water	125 mL Plastic	Filter on site; 4 °C	28 Days	N/A
376.1 or 376.2	Sulfide (S2)	Water	500 mL Plastic	4 °C; 2 mL zinc acetate plus NaOH to pH>9	7 Days	N/A

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Other regulatory or project requirements may apply. If so, the analytical Subcontract Laboratory will be advised.

ATTACHMENT 2: PRESERVATIVE CHECKLIST

5056-2

Preservative Checklist

Records Use only

		Container Volume				
		40 mL	**125 mL**	**250 mL**	**500 mL**	**1,000 mL (1L)**
Chemical Prevervative	**HNO_3**	N/A	0.193 mL	0.386 mL	0.771 mL	1.542 mL
	H_2SO_4	N/A	0.248 mL	0.495 mL	0.99 mL	1.98 mL
	HCl	0.036 mL	0.114 mL	0.227 mL	0.454 mL	0.908 mL
	NaOH	N/A	0.417 mL	0.833 mL	1.667 mL	3.333 mL
	HaOH	N/A	0.2 mL	0.4 mL	0.8 mL	1.6 mL
	NaOH	N/A	1 mL	2 mL	4 mL	8 mL
	NaOH (solid)	N/A	0.1 g	0.2 g	0.4 g	0.8 g

Reagent Purity	Specific Gravity	Allowed concentration
69%	1.41	0.15%
96%	1.841	0.35%
37%	1.191	0.04%
solid	N/A	
6N - 0.24 g/mL%	N/A	
50% - 0.5 g/mL		
2.5N - 0.1 g/mL		