

STANDARD OPERATING PROCEDURE

SEVEN

SAMPLE PRESERVATION, STORAGE, HANDLING AND DOCUMENTATION

Modified from

U.S. Environmental Protection Agency Environmental Response Team

Response Engineering and Analytical Contract

Standard Operating Procedures

Sample Preservation, Storage, and Handling

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1.0 SCOPE AND APPLICATION

The purpose of this Standard Operating Procedure (SOP) is to provide general guidelines for the preservation, storage, and handling of air, water and soil/sediment samples. Requirements for sample volume, matrix spike/matrix spike duplicate (MS/MSD) sample volume, container type, and preservation techniques for sample preservation, storage, and handling must be established in the work plan prior to sample collection.

The methods described in this SOP are typically applicable operating procedures which may be varied or changed as required, dependent upon site conditions or equipment limitations. In all instances, the procedures employed should be documented in the site logbook and associated with the final report.

Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

2.0 METHOD SUMMARY

Proper techniques of preserving, storing, and handling air, water and soil/sediment samples are critical if the integrity of the samples are to be maintained. This SOP is applicable to all water and soil/sediment samples collected in South Dakota. This SOP will be expanded to air samples once acceptable air sampling procedures have been selected.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

3.1 Sample Preservation and Storage

Samples should be collected using equipment and procedures appropriate to the matrix, the parameters to be analyzed, and the sampling objective. The volume of the sample collected must be sufficient to perform the analysis requested, as well as the quality assurance/quality control requirements.

Table 1 contains examples of parameters which are typically of interest in environmental site investigations and indicates the required sample volume, the proper types of containers, and the preservation method for water and soil/sediment samples. All samples must be cooled to 4°C from the time of collection until analysis. This SOP will be expanded to include air samples once acceptable procedures for air sampling have been selected.

Depending on the arrangements for sample analysis and the amount of sample required for the analysis, it is possible that aliquots for several analyses may be taken from the same sample container. This should be verified with the laboratory performing the analyses prior to sample collection.

All sample containers must be clean and labeled appropriately. The exterior of the sample containers must be wiped clean and dry prior to sample packaging. All samples must be packaged according to the requirements of U.S. Department of Transportation (U.S. DOT) or International Air Transportation Association (IATA).

For more information regarding air, water and soil/sediment sample collection, refer to the Procedures section of the appropriate Standard Operating Procedure. Sample containers must not be pre-rinsed with the sample prior to sample collection. To prevent leakage of aqueous samples during shipping, sample containers should be no more than 90

percent full. If air space would affect sample integrity, such as with VOC samples, fill the sample container completely and place the container in a second container to meet the 90 percent requirement. When a preservative other than cooling is used, the preservative is generally added after the sample is collected, unless the proper amount of preservative is already present in a laboratory-supplied container. If necessary, the pH must be adjusted to the appropriate level and checked with pH paper in a manner, which will not contaminate the sample.

The laboratory performing the analysis should be contacted to confirm the requirements for sample volumes, container types, and preservation techniques. This information should be documented in the work plan.

3.2 Chain-of-Custody Procedures

In some instances, it may be necessary to prove any analytical data offered into evidence accurately represent environmental conditions existing at the time of sample collection. Due to the evidentiary nature of such samples, possession must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. It must be clearly demonstrated that none of the involved samples could have been tampered with during collection, transfer, storage, or analysis.

To maintain and document sample possession, the following chain-of-custody procedures are followed:

3.2.1 Sample custody

A sample is under custody if:

- a) It is in your possession, or
- b) It is in your view, after being in your possession, or
- c) It was in your possession and then you locked it up or placed it in a sealed container to prevent tampering, or
- d) It is in a designated secure area.

3.2.2 Field custody

- a) Advise laboratory personnel at the time a decision is made that a sample requiring a chain-of-custody record is going to be collected. Specify the data and time that it will arrive in the laboratory. In instances where it is not known in advance of field trip, the laboratory should be notified as soon as possible about the arrival of such samplers.
- b) In collecting samples for evidence, collect only that number which provides a good representation of the medium being sampled. To the extent possible, the quantity and type of samples and sample locations are determined prior to the actual field work. As few people as possible should handle the samples.
- c) The samples must be collected in accordance with required and established methods.

3.2.3 Transfer of custody and shipment

- a) To establish the documentation necessary to trace sample possession, a Chain-of-Custody Record (Figure 1) must be filled out and accompany each set of samples. The record should accompany the water quality data form and the samples to the laboratory. This record documents sample custody transfer from the sampler to the analyst at the laboratory. At a minimum, the record should contain: the station number or sample identification; the signature of the collector and witnesses when present; the date and time of collection; place and address of collection; substance sample; signature of persons involved in the chain of possession; and, inclusive dates of possession.
- b) Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis. The samples for each shipping container be placed in the large plastic bag should be sealed with a paper seal (Figure 2) to indicate for possible tampering.
- c) If samples are split with a source or government agency, it is noted in the remarks section of the Chain-of-Custody Record. The note should indicate with whom the samples are being split and signed by both the sampler and recipient.
- d) Each transfer of sample custody must be documented on the Chain-of-Custody Record; however, when the sample is to be sealed for shipment, the word "sealed" should be written after the collector's signature. Then received in the laboratory, the word "sealed" should be written after the recipient's signature if no tampering has occurred.
- e) All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and a copy will be retained by the project leader.
- f) The laboratory should have an assigned laboratory custodian and an alternate who are responsible for overseeing the reception of all controlled custody samples. Controlled custody samples will be of the highest priority and will be analyzed before all other environmental samples.
- g) In the field and in the laboratory, the number of individuals having access to these samples should be kept to a minimum to lessen the number of potential witnesses. Then the samples are not in the immediate possession of the individual having official custody, they must be kept in a locked enclosure.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

The following are interferences or potential problems associated with sample preservation, storage, and handling:

- Samples should be protected from sunlight which may initiate photodegradation of sample components.
- Delaying sample preservation may cause chemical reactions to occur, altering original sample composition.
- Improper sample preservation may adversely affect analytical results.

- Inadequate sample volume may prohibit the appropriate analyses from being performed.
- Samples can become contaminated if they come in contact with human flesh; therefore, appropriate protective gloves (i.e., rubber, latex, or plastic) should be worn at all times during sampling collection and preservation.
- Samples can also become contaminated from equipment used to collect and preserve the sample; therefore, all sample collection and preservation equipment must be kept clean.

5.0 EQUIPMENT/APPARATUS

The equipment/apparatus required to collect samples must be determined on a site-specific basis. Refer to the specific SOPs for sampling techniques, which include lists of the equipment/apparatus required for sampling.

In general, the following specific equipment/apparatus may be required for proper sample preservation:

- pipettes (various sizes)
- pipette bulb
- pH paper
- safety equipment
- glass and plastic bottles (various sizes)
- preservatives (acids, bases, and/or ice)

6.0 REAGENTS

Reagents required for preservation of samples are specified in Table 1. The preservatives required are specified by the analyses to be performed. Decontamination solutions are specified in the Standard Operating Procedures for Sampling Equipment Decontamination.

7.0 PROCEDURES

Once aqueous samples are collected, add the appropriate preservative to reach the desired pH, then immediately cool samples to 4°C. Check with the analytical lab to determine if preservative is required and for the proper amount of preservative to be added to the sample if the laboratory does not provide sampling containers with preservative added.

8.0 ADDITIONAL INFORMATION SOURCES

Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities, NIOSH/OSHA/USCG/EPA, October, 1985.

U.S. Environmental Protection Agency, 1988, Environmental response branch standard operating safety guides, July.

TABLE 1 (cont.)

Parameter	Matri x²	Container³	Volume to be Collected	Preservative	Holding Times
Petroleum Hydrocarbons	S	G	224 grams	Cool (4 ⁰ C)	14 days
Pesticides/PCBs ⁴	W	G (amber)	2 x 1 liter	Cool (4 ⁰ C)	7 days until extraction, 40 days after extraction
Pesticides/PCBs	S	G	224 grams	Cool (4 ⁰ C)	14 days until extraction, 40 days after extraction
Phenols	W	G	1 liter	Cool (4 ⁰ C), H ₂ SO ₄ , pH<2	7 days until extraction, 40 days after extraction
Phenols	S	G	224 grams	Cool (4 ⁰ C)	14 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons ⁴	W	G	2 x 1 liter	Cool (4 ⁰ C)	7 days until extraction, 40 days after extraction
Polynuclear Aromatic Hydrocarbons	S	G	224 grams	Cool (4 ⁰ C)	14 days until extraction, 40 days after extraction
Reactivity (RCRA) (Cyanide)	W	P	1 liter	Cool (4 ⁰ C), Na OH, pH>12	?
Reactivity (RCRA) (Sulfide)	W	P	1 liter	Cool (4 ⁰ C), 4.0 ml zinc acetate solution	?
Reactivity (RCRA) (Cyanide/Sulfide)	S	G (amber)	224 grams	Cool (4 ⁰ C)	?
Corrosivity (RCRA)	W	P	500 ml	Cool (4 ⁰ C)	?
Ignitability (RCRA)	W	G (amber)	500 ml	Cool (4 ⁰ C)	?
Ignitability (RCRA)	S	G (amber)	224 grams	Cool (4 ⁰ C)	?
TCLP-VOCs ⁵	W	G	3 x 40 ml vial	Cool (4 ⁰ C)	?
TCLP-BNAs	W	G (amber)	2 x 1 liter	Cool (4 ⁰ C)	?
TCLP-Pesticides/ Herbicides	W	G (amber)	2 x 1 liter	Cool (4 ⁰ C)	?
TCLP-Inorganics	W	P	1 liter	Cool (4 ⁰ C), HNO ₃ , pH<2	?
TCLP-Nonvolatile Extratction ⁶	S	G	448 grams	Cool (4 ⁰ C)	?
TCPL-Volatile Extration ⁶	S	G	448 grams	Cool (4 ⁰ C)	?
TOC	W	P or G	500 ml	Cool (4 ⁰ C), H ₂ SO ₄ , pH<2	28 days
TOC	S	G	224 grams	Cool (4 ⁰ C)	?

TABLE 1

EXAMPLES OF SAMPLE CONTAINERS, VOLUMES TO BE COLLECTED, PRESERVATIVES AND HOLDING TIMES BY PARAMETER AND MATRIX¹

Parameter	Matrix ²	Container ³	Volume to be Collected	Preservative	Holding Times
Acidity/Alkalinity	W	P or G	1 liter	Cool (4 ⁰ C)	14 days
Acidity/Alkalinity	S	P or G	224 grams	Cool (4 ⁰ C)	14 days
BNA	W	G (amber)	2 x 1 liter	Cool (4 ⁰ C)	7 days until extraction, 40 days after extraction
BNA ⁴	S	G	224 grams	Cool (4 ⁰ C)	14 days until extraction, 40 days after extraction
BOD	W	G	1 liter	Cool (4 ⁰ C)	2 days
COD	W	P or G	1 liter	Cool (4 ⁰ C), H ₂ SO ₄ , pH<2	28 days
Cr ⁺⁶	W	P	200 ml	Cool (4 ⁰ C)	24 hours
Creosotes ⁴	W	G	2 x 1 liter	Cool (4 ⁰ C)	?
Creosotes	S	G	224 grams	Cool (4 ⁰ C)	?
Cyanide ⁴	W	P	1 liter	Cool (4 ⁰ C), NaOH, pH>12	14 days
Cyanide	S	G	224 grams	Cool (4 ⁰ C)	14 days
Dioxin/Furans	W	G	2 x 1 liter	Cool (4 ⁰ C)	7 days until extraction, 40 days after extraction
Dioxin/Furans	S	G	448 grams	Cool (4 ⁰ C)	?
Herbicides ⁴	W	G	2 x 1 liter	Cool (4 ⁰ C)	7 days until extraction, 40 days after extraction
Herbicides	S	G	224 grams	Cool (4 ⁰ C)	14 days until extraction, 40 days after extraction
Mercury (Hg)	W	P or G	1 liter	Cool (4 ⁰ C), HNO ₃ , pH<2	28 days
Mercury (Hg)	S	P or G	224 grams	Cool (4 ⁰ C)	28 days
Metals (except Cr ⁺⁶ and Hg)	W	P or G	1 liter	Cool (4 ⁰ C), HNO ₃ , pH<2	6 months
Metals (except Hg)	S	G	224 grams	Cool (4 ⁰ C)	6 months
Oil & Grease	W	G	2 x 1 liter	Cool (4 ⁰ C), H ₂ SO ₄ , pH<2	28 days
Oil & Grease	S	G	224 grams	Cool (4 ⁰ C)	28 days
Petroleum Hydrocarbons ⁴	W	G	2 x 1 liter	Cool (4 ⁰ C), H ₂ SO ₄ , pH<2	14 days

TABLE 1 (cont.)

Parameter	Matri x²	Container³	Volume to be Collected	Preservative	Holding Times
TOX	W	G	300 ml	Cool (4 ⁰ C)	28 days
TOX	S	G	224 grams	Cool (4 ⁰ C)	?
VOCs ⁶	W	G	3 x 40 ml vial	Cool (4 ⁰ C) ⁷	14 days
VOCs	S	G	40 ml vial	Cool (4 ⁰ C)	14 days

¹ Subcontract laboratory requirements may vary. Verify prior to sample collection.

² W - water, S - soil/sediment

³ P - polyethylene, G - glass

⁴ For one sample of every batch of 10 (or less) samples, collect two additional 1 liter volumes for MS/MSD analysis.

⁵ Avoid excessive turbulence when filling the sample container. The container must be sealed so that no air bubbles are entrapped. No headspace allowed.

⁶ For one sample of every batch of 10 (or less) samples, collect two additional 16-ounce volumes for MS/MSD analysis.

⁷ For drinking water samples, if residual chlorine is present, the sample should be preserved with 0.008 percent sodium thiosulfate. U.S. Environmental Protection Agency Methods 330.4 and 330.5 may be used for measurement of residual chlorine. Field-test kits are commercially available for this purpose.

Figure 1. Chain-of-Custody Record

South Dakota Department of Environment and Natural Resources
Joe Foss Building, Pierre, South Dakota 57501

CHAIN OF CUSTODY RECORD

PROJ. NO.	PROJECT NAME					NO. OF CONTAINERS	REMARKS					
SAMPLERS (Signature)												
STAT. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION							
Relinquished by: (Signature)							Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)	
Relinquished by: (Signature)							Date/Time	Received by: (Signature)	Relinquished by: (Signature)	Date/Time	Received by: (Signature)	
Relinquished by: (Signature)							Date/Time	Received for Laboratory by: (Signature)	Date/Time	Remarks:		

Distribution: Original Accompanies Shipment, First Copy to Coordinator Field Files, Second Copy to Representative of Inspected Facility

Spot Sample
 Accepted Declined _____ Signature

End of Section

Figure 2. Chain-of-Custody Paper Seal Example

**SOUTH DAKOTA DEPARTMENT OF
ENVIRONMENT AND NATURAL RESOURCES
PIERRE, SOUTH DAKOTA 57501**

DATE: _____ **SAMPLER:** _____
SAMPLE: _____