CONTAINERS, PRESERVATION, AND HOLDING TIMES FOR SELECTED PARAMETERS

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Water Data Management & Environmental Quality

St. Louis District
U. S. Army Corps of Engineers

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1 INTRODUCTION

1.1 WATER QUALITY SAMPLE CONTAINERS, PRESERVATION AND HOLDING TIMES

This Standard Operating Procedure (SOP) is intended to serve as a field reference guide and applies to surface water quality sample containers, sample preservation and holding times for selected parameters. It describes container types, preservation solutions and methods, and holding times required for chemical analysis as used by the U. S. Army Corps of Engineers, St. Louis District personnel who collect water quality samples on all Corps Projects.

1.2 PURPOSE AND SCOPE

This SOP is directed specifically toward field personnel to facilitate the collection of consistent and representative water quality data. To help ensure that data collected on Corps Projects meet established data quality objectives, it is imperative that proper field procedures are followed during sample collection. Following the procedures provided in this SOP will ensure consistent and representative data of known quality are collected.

1.3 APPLICABILITY

This SOP applies to all surface water quality and related data collection efforts implemented by the Water Data Management & Environmental Quality Section with the St. Louis District's Environmental Quality Section.

1.4 DEFINITIONS AND ACRONYMS

AHA – Activity Hazard Analysis. A Corps document containing all the possible hazards about a specific job function.

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Holding Time - the length of time a sample can be stored after collection and prior to analysis without significantly affecting the analytical results.

MSDS – Material Safety Data Sheets. MSDS' are available for all hazardous material and contain basic information needed to insure the safety of the user.

QA/QC – Quality assurance/quality control
SAP – Sampling Analysis Plan
SOP – Standard Operating Procedure

2 HEALTH AND SAFETY WARNINGS

The Corps' Activity Hazard Analysis (AHA) program requires a review of all field activities followed by a summary report listing all accident potentials resulting in that area of work. Personnel should review the HAA for all water sample collection duties and inform the supervisor if a report for an activity is not available.

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When sampling possibly contaminated sites personnel are required to follow any Corps, EPA, OSHA and other specific health and safety procedures. All chemicals, cleaners and compounds carried in Corps trucks or used by Corps personnel must be accompanied by a Material Safety Data Sheet (MSDS) document. A file of all MSDS documents is available to personnel at each Project Office and from safety officers.

When working in or near water, personal flotation devices are mandatory and personnel must follow all applicable safety precautions.

Sample preservation requires the use of nitric and sulfuric acids. Both acids are capable of producing severe burns and blindness therefore wearing safety glasses and carrying eye rinse are required and acid proof gloves are recommended.

3 CAUTIONS

Acid preservatives are stored in glass vials and are released by removing screw top and inverting it. When the top of the ampoule is snapped off acid can be ejected upward toward the samplers face, the need for safety glasses is imperative.

Field offices should have an acid spill clean up kit available for large spills and personnel should be familiar with clean up procedures.

4 INTERFERENCES

Interferences that affect data quality are broken into two general groups (1) contamination and (2) improper handling.

4.1 CONTAMINATION

Contamination is the inadvertent addition of material to the water sample. The following lists several possible avenues of sample contamination:

- Sample containers and equipment need to be stored properly during the sample trip. All containers need to be closed securely and sampling equipment needs to be stored so neither will come into contact with contaminants.
- Inadvertently adding the wrong acid preservative can render the sample unusable for analytical analysis of targeted parameters.

4.2 IMPROPER HANDLING

Improper sample handling can greatly reduce the quality of data generated from collected samples. The following lists several improper handling concerns that need to be avoided.

- Improper Cooling. Most samples require cooling, which slows biological and chemical reactions once the sample is collected, as part of the preservation process. Failure to cool the samples shortly after collection or packing the samples with insufficient ice when shipping can significantly impact the quality of the samples.

- Missing the Holding Time. Each analyte has a specific time it can be stored before it can be analyzed. Delaying the shipment of samples to the lab or shipping problems can result in samples exceeding the holding time (Table 1.)

- Improperly Stored or Secured Sample Bottles. Glass sample bottles should never be stored next to each other without a separator during transport as they can easily break. Sample containers packed in ice shift and rub against each other and the ice. If the sample container lid is not tight this abrasion can loosen or remove the cap.

5 PERSONNEL QUALIFICATIONS

Personnel should be knowledgeable and experienced in sample collection.

6 EQUIPMENT AND SUPPLIES

The following list defines those items discussed in this SOP

6.1 PRESERVATION

• Acid carrying case
- Nitric acid vials
- Sulfuric acid vials
- Eye wash
- Cooler (s)
- Ice or frozen gel packs

6.2 SAMPLE CONTAINERS

- 1 L polyethylene wide mouth
- 250 ml polyethylene wide mouth
- 500 ml polyethylene wide mouth
- 1L amber glass, Teflon lined lid wide mouth
- 500 ml clear glass wide mouth
- 25 ml glass test tube with screw cap
- 1 L brown plastic wide mouth
- 1 L and 500 ml Nalgene wide mouth
- 40 ml VOA vial
- 50ml Fecal bottle?

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7 CONTAINERS, PRESERVATIVES, AND HOLDING TIMES

Samples that are not analyzed at the sampling site must be stored, preserved and transported to the appropriate laboratory within specific time limits.

7.1 SAMPLE CONTAINERS

The St. Louis District Environmental Quality Section uses only certified pre-cleaned polyethylene, plastic and amber glass bottles for sample collection with the exception of chlorophyll a and invertebrates. The chlorophyll a samples are placed in a new 1L brown plastic wide mouth screw top bottles. Invertebrates are placed in a new Nalgene 1 L wide mouth bottle.

Containers are purchased sealed and should only be opened when filling with the sample.

7.2 Sample Preservation

Methods of preservation include chemical addition for pH control, "fixing" biological organisms, and cooling. Preservation is intended to reduce biodegradation, volatilization, oxidation, sorption, precipitation, and other physical, chemical and biological processes. Complete preservation of samples is impossible because complete stability for every chemical and biological constituent can never be achieved.

Preservatives used by the Environmental Quality Section include:

- Nitric Acid
- Sulfuric Acid
- Hydrochloric Acid
- Formaldehyde
- Ethanol
- Cooling to 4^{o}C

7.3 Holding Times

Holding times are the length of time a sample can be stored after collection and prior to analysis without significantly affecting the analytical results. Holding times vary with the analyte sample matrix, and analytical methodology used to quantify the analytes concentration. Maximum holding times are defined by the analytical method used.

Table 1 presents sample category, specific parameters, container types, preservatives and holding times. The category column corresponds to those found on the Water Sample Labels (See SOP WQ- , Collection of Surface Water Samples, for labeling requirements).

Analyte	Container	Preservatives	Holding Time	Minimum Sample
Alkalinity	Polyethylene	4°C	14 d	200ml
BOD	Polyethylene	4°C	48 hrs	1000ml
Chlorophyll	Polyethylene	4°C	30 d	500ml
Ammonia-Nitrogen	Polyethylene	4°C, H_2SO_4	28 d	500ml
Iron, Total	Polyethylene	4°C, HNO_3	6m	250ml
Maganese, Total	Polyethylene	4°C, HNO_3	6m	250ml
Nitrate-Nitrogen	Polyethylene	4°C, H_2SO_4	28 d	500ml
Ortho-Phosphate	Polyethylene	4°C	48h	250ml
Pesticides	Amber Glass, TFE lined cap	4°C	7d	1000ml
Phosphate, Total	Polyethylene	4°C	48h	100ml
Suspended Solids, Total	Polyethylene	4°C	7d	200ml
Volatile Suspended Solids, Total	Polyethylene	4°C	7d	200ml
Total Kjeldahl Nitrogen	Polyethylene	4°C, H_2SO_4	28d	500ml
Volatile Organic Compounds	Glass Vial	4°C, HCL	7d	40ml
Invertebrates		10% Formaldehyde or 20% ethanol	Indefinite	

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8. References

U.S. Army Corps of Engineers. 2010. Sampling and Analysis Plan for 2010 Monitoring of St. Louis District USACE Reservoirs.

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