Standard Operating Procedure (SOP) 2.3.4

Adopted from: Field Guide for Surface Water Sample and Data Collection

Sample Filtering

1.0 Introduction

Many samples require filtration either in the field or the laboratory. Filtration separates constituents dissolved in the water from detritus and other solids that may alter the chemistry of the sample before it can be analyzed. However, filtration in the field adds the possibility of sample contamination if not done carefully. Program needs and the nature of the water being sampled determine whether the greater risk is from field filtration or from sample degradation before filtration at the laboratory. Samples containing large amounts of suspended solids or organic matter and samples analyzed for trace constituents (such as **dissolved iron**) are the most susceptible to degradation before filtration at the laboratory. If samples are filtered in the field it is possible to check for potential contamination by processing (filtering) and bland sample of deionized (DI) water to determine whether field filtration is associated with any contamination.

As with bottles, each batch of filters must be checked to assure that they do not contaminate the samples. For the same reasons discussed for bottles, it is most efficient if the laboratory supply and quality assure any filters that are used for field filtration. Care should also be taken to avoid contamination of the filter and filter apparatus from fingerprints and soil. The filter and apparatus need to be rinsed well with either DI water or sample water prior to filling the sample bottles. Because filtering apparatuses vary greatly in size it is impossible to give universal guidelines on the amount of rinsing needed. Thus, it is most efficient if the laboratory specifies or provides or recommends any filtering apparatus that should be used and determines an appropriate rinse protocol for that apparatus. Filters commonly used are made of polycarbonate or are cellulose-based, e.g., cellulose acetate. The pore size of the filter is critical in determining what actually is analyzed in filtered samples. A pore size of 0.45 microns is commonly used for many constituents, 0.1 microns is used for many metals such as aluminum; however, smaller pore sizes may be needed for the analysis of some trace metals if colloidal material is present in significant amounts. This is best determined by comparing a series of aliquots collected with filters of differing pore sizes, for example, 0.4 and 0.1 micron filters. This needs to be done only in studies that need the best estimates of dissolved trace metal concentrations.

2.0 Ortho-P & Total-P Filtering

2.1. Sample Collection

Sample types may be: Grab (surface run-off), and Auto samples (Composite sample). The water samples are prepared and processed for analysis of Ortho-P and Total- P.

2.1.1. Equipments and Materials

- Dipper, sampling pole
- Large sampling jars
- Cooler with ice pack

- Marker
- Field notebook
- Pen

2.1.2 Procedure

- Make sure jars are properly labeled before start of sampling.
- Rinse the dipper 3 times with sample.
- Rinse the jars with cap three times with sample water.
- Fill the jar to it's brim and cap it.
- Take Normal (N) sample
- Take Field Duplicate (FD) samples

3.0 Sample Processing

3.1 Surface Grab Samples

A chart showing the processing procedure for grab samples is below:



Figure 1. Surface grab sample processing chart.

3.1.1 Filter Equipments and Cleaning

Materials needed for filtering include

- Disposable gloves
- Phosphorus- free detergent
- Tooth brush
- Filter holders
- O-ring
- Filter paper (0.45 microns)

3.1.2. Steps for Cleaning filter holders

- 4 buckets arranged in the following sequence:
- First bucket should contain soap solution made of phosphorus-free detergent and tap water
- Second bucket should contain clean tap water

- Third bucket should have DI water
- Last bucket should also have DI water
- Open filter holder by rotating anti-clockwise (sideways)
- Remove dirty / used filter papers (discard)
- Wash both parts of filter holders with brush in the Phosphorus-free soap solution (1st bucket)
- Rinse the filter holder in clean tap water (2nd bucket)
- Then rinse in the first DI water (3^{rd} bucket)
- Rinse finally in the second DI water (4^{th} bucket)
- Check to ensure there is O-ring in at least one part of the filter holder
- Place filter paper in the bottom part of the filter holder (use caution not to tear or kink)
- Attach the top part of the filter to the bottom

3.1.3 Filtering

- Grab Samples intended for Ortho-P, analysis must be filtered with 0.45 um (fine filter). However, samples for Total-P should not be filtered.
- Properly place filters in filter holders
- Use syringe to take samples from jar and filter into vials
- Rinse syringe with DI water (3x) before using on subsequent samples
- More than 100ml filtered sample is needed for Ortho-P laboratory analysis
- 100ml unfiltered sample plus 5 drops of sulfuric acid (H_2SO_4) acid is

required for Total-P laboratory analysis

3.1.4 Preservation

- This is done to maintain the chemical integrity of the samples until they are analyzed in the lab.
- Samples for TP are preserved with sulfuric acid (H_2SO_4).
- However, no acid should be added to the samples for Ortho-P analyses.
- Use dropper to take acid from the acid container
- Put five (5) drops of acid on 100ml samples in each bottle intended for TP analyses.
- Make sure the pH of sample is 2 by dipping a pH paper in the sample and comparing the color with the standard pH colors.
- Do not over acidify the samples to a pH less than 2.

3.1.5. Storage

- Check sample container for leaks by squeezing tightly.
 - Label vials using COC

Check accuracy of labeling twice before storage.

Samples should be processed immediately after coming back from the field. The samples should be delivered to the laboratory within 48 hours after collection.

3.2.1 Preservation

Composite Samples are already preserved with H₂SO₄ during the sampling.

- Check pH by dipping a pH paper in the sample and comparing the color with the standard pH colors. Make sure pH is at 2.
- Do not over acidify the samples to a pH less than 2.

Tip: When provided with an expiration date on the filters being used, use filters prior to their expiration date.

Reference: Turk, John. 2001 Field Collection for Surface Water Sample and Data Collection. USFS. USDA www.fs.fed.us/waterdata/PDFfiles/FieldGuide