STANDARD OPERATING PROCEDURE
Analysis of Whole Sample Suspended Sediments in Water

KEY WORDS

Suspended sediment concentration, vacuum pump, glass fiber microfilter, Buchner filter funnel, drying oven, analytical balance

APPROVALS

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1.0 INTRODUCTION

1.1 PURPOSE

The purpose of this standard operating procedure is to describe the procedure used by the California Department of Pesticide Regulation, Environmental Monitoring Branch, to measure the whole sample suspended sediments in water. This method is also used to provide filtrate for measuring dissolved organic carbon (DOC) for CDPR SOP METH011).

1.2 DEFINITIONS

Whole sample suspended sediment in water is referred to as suspended sediment concentration (SSC). SSC measures all particles less than 1000 microns in size in a 1-L water sample. Macro-organic debris and other particles greater than 1000 microns (1 mm) in size (e.g., pebbles, filamentous algae, and plant debris) are not considered suspended sediments and are removed prior to analysis (Guo 2006). SSC method is more accurate than total suspended solid (TSS) analysis for natural waters, especially when sand-sized material composes a sizeable percentage of the sediment of a sample (Gray et al. 2000, Guo 2006). TSS measures an aliquot of SSC. For samples that have high levels of sediment, as edge-of-field runoff, TSS analysis (using US EPA Method 160.2; ALS Environmental 2011) may be more appropriate than SSC analysis because filters can become quickly overloaded and sediments often bypass the filter, going down the side of funnel. This SSC method is derived from US EPA Method 160.2.

2.0 MATERIALS

2.1 Vacuum pump with tubing

2.2 Buchner filter funnels

2.3 Erlenmeyer flasks (1000 mL)

2.4 Beakers (50 mL)

2.5 Glass microfiber filters (Whatman GF/F 1825-090, 0.7 micron)

2.6 Wash bottle

2.7 Deionized water

2.8 Forceps

2.9 Gloves

2.10 Safety glasses or goggles

2.11 Desiccator with desiccant

2.12 Drying oven (set at 103-105°C)

2.13 Analytical balance (accurate to 0.001g)

3.0 PROCEDURES

3.1 Water Samples

Store field collected water samples promptly in the walk-in refrigerator at 4°C. For analysis, move a set of water samples to the laboratory.

3.2 Preparation

3.2.1 Wipe the shelves of the drying oven with a wet towel or sponge to remove dust and dirt (if trays are used, replace aluminum foil). Turn the drying oven on and heat to 103-105°C.

3.2.2 Replace lab bench paper (if dirty) and clean the area where you will be working. Set up lab bench paper with the absorbent side facing up.

3.2.3 Attach a Buchner filter funnel to an Erlenmeyer flask, then attach the tubing to the Buchner filter funnel (Figure 1).

3.2.4 If using a rotary vane vacuum pump (e.g., Adixen pump), let the pump warm up one hour prior to use. Also prior to using, ensure oil in the pump is clean and full.

3.3 Pre-rinsing filters

3.3.1 With gloved hands and forceps, place a glass microfiber filter in a Buchner filter funnel with bumpy (wrinkled) side facing up.

3.3.2 To seal the glass microfiber filter to the Buchner filter funnel, squirt deionized water onto the filter until completely wetted (use wash bottle).

3.3.3 Apply suction with the vacuum pump by opening the “open/shut” handle. Gently lift the Buchner filter funnel to ensure that it is sealed.

3.3.4 Rinse the Buchner filter funnel with deionized water thrice, applying ~20 mL with each rinse. Allow the vacuum to remove all traces of water.

3.3.5 Release the suction by closing the “open/shut” handle. Using the forceps, remove the filter from the apparatus and place in the drying oven (bumpy side up). Lift and rotate the filters frequently during the first minute to prevent the filter form sticking to the oven rack or tray. Discard any filters that stick.

3.3.6 Dry the filters overnight at 103-105°C.

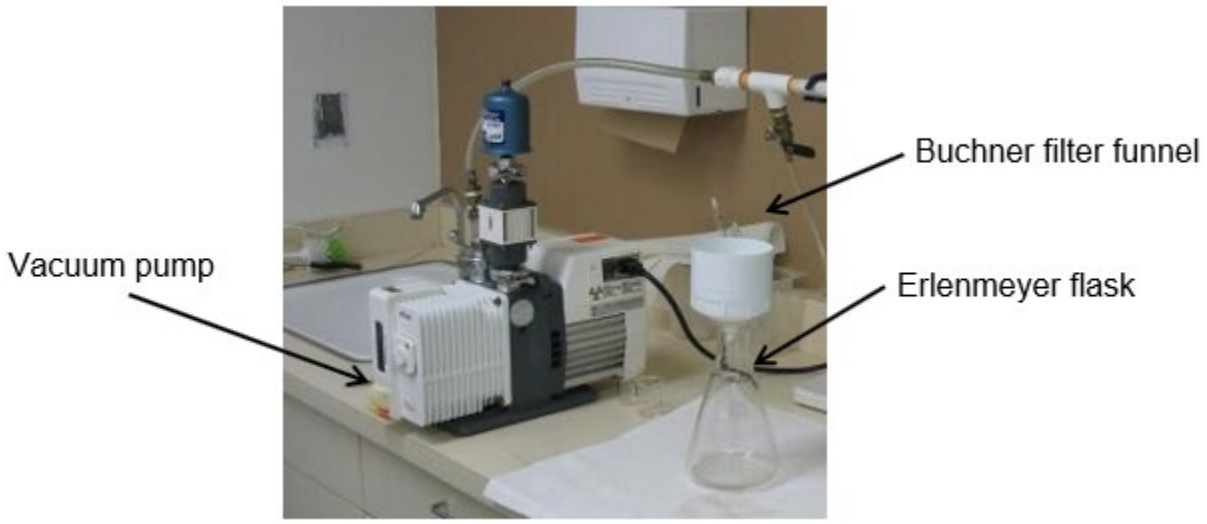

Figure 1. Filtering apparatus with Adixen vacuum pump, Buchner filter funnel, and Erlenmeyer flask.

3.4 Filtering the water samples

3.4.1 Remove the filters from the drying oven and place in a desiccator. The desiccant should be blue; replace it when it begins to turn purple.

3.4.2 Take a cooled glass microfiber filter from the desiccator and number it with an extra-fine point permanent marker (e.g., Sharpie®).

3.4.3 Using the analytical balance, weigh the glass microfiber filter (do not use a disposable weigh boat but ensure analytical balance weigh pan is clean). Record the weight and filter number on a data sheet. Use forceps

to carry filter paper to and from the analytical balance. Weigh boats can contain a static charge which may affect the final filter weight.

3.4.4 Using forceps, place a glass microfiber filter in the center of the Buchner filter funnel and seal with a small volume deionized water from the wash bottle.

3.4.5 In preparation for obtaining a sample for DOC analysis, do not shake sample bottles that are also being filtered for DOC. Only the dissolved portion of the sample is to be filtered for DOC.

3.4.6 Pour the content of one bottle into a 1-L graduated cylinder; record the volume on a data sheet.

3.4.7 Apply suction by opening the “open/shut” handle. Ensure that the filter is sealed.

3.4.8 Slowly pour the water sample into the Buchner filter funnel. With forceps, remove any macro-organic debris (particles > 1 mm in size; e.g., pebbles, filamentous algae, and plant debris).

3.4.9 For samples that are being analyzed for DOC, pour approximately 35 ml of the filtrate to a vial for organic carbon analysis (DPR SOP METH011). Then rinse sample bottle and graduated cylinder thrice with ~20 mL of deionized water (each time), filtering the rinse water into the Buchner filter funnel.

3.4.10 Using the wash bottle, rinse Buchner filter funnel with deionized water. Remove all traces of water with suction on.

3.4.11 Release the suction by closing the “open/shut” the handle. Using forceps, remove the glass microfiber filter from the Buchner filter funnel and place in the drying oven. Lift and rotate the filters frequently during the first minute to prevent the glass microfiber filter from sticking to the oven rack. Discard any filters that are damaged or that stick to the oven rack.

3.4.12 Repeat steps 3.4.2–3.4.11 to process the remaining samples. To avoid contamination and dilution of samples, use clean and dry Buchner filters and Erlenmeyer flasks for each sample.

3.4.13 Dry all filters overnight at 103-105°C.

3.5 Weighing the glass microfiber filters with sediment

3.5.1 Turn off oven.

3.5.2 Using forceps, remove the glass microfiber filters from the drying oven and place in a desiccator. Ensure the desiccant is blue (dry condition).

3.5.3 When cooled, weigh filters with an analytical balance and record weights. Do not use a disposable weigh boat when weighing filters, and ensure the filter is entirely on the balance weighing pan and that the pan is clean.

3.5.4 After weighing, store filters in the desiccator until data are approved by study director.

4.0 CLEANING AND MAINTENANCE

4.1 Clean drying oven, oven racks, and trays with a damp sponge after use. Remove dirty aluminum foil from trays, if used.

4.2 Disconnect Buchner filter funnels from the vacuum tubing. Fully dissemble funnels into three components prior to washing. Wash any glassware (beakers, Buchner filter funnels, Erlenmeyer flasks) and place on drying rack. Remove items when dry.

4.3 Replace oil in vacuum pump when it becomes dirty, is not full, or becomes contaminated with air. Follow manufacturer's recommendation for changing the oil.

5.0 QUALITY CONTROL

For QC, a 1-L sample of DI water will be filtered with each batch of samples. SCC data for the field samples will be acceptable if the QC DI sample is within three standard deviations of the continuous lab DI mean.

6.0 CALCULATIONS

SSC is the weight of the sediment expressed as mg/L. Use this simple equation to calculate SSC:

$$SSC = \frac{(A - B) \times 1000}{C}$$

where A = the weight of the filter + sediment residue (expressed in mg), B = the weight of the filter (in mg**)**, and C = the volume (expressed in mL) of the filtered sample.

7.0 SAFETY

7.1 Personal protective equipment needed when filtering: gloves and safety glasses with side shields or goggles.

7.2 Personal protective equipment needed when using the oven: insulated oven mitts may be needed.

8.0 REPORTING REQUIREMENTS

Record the data (including sample number, date sample was collected, date water sample was filtered, clean filter weight, 'dirty' filter weight, and appropriate notes) in an Excel spreadsheet and forward to the study's lead scientist.

9.0 REFERENCES

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