

TOP:020.4

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TECHNICAL OPERATING PROCEDURE

PROCEDURE TITLE:

Procedures for Filtering Fine Particulate Matter from Water Samples That Contain TFM

APPLICABILITY:

This procedure is applied when conducting analysis for TFM on water samples that contain colloidal clay or other fine particulate matter not removed by standard filtration. Millipore filtration (vacuum or syringe) is used before all measurements of TFM concentration if the absorbance of a water sample filtered through Whatman 2V or equivalent filter paper differs by more than .020 absorbance units from the absorbance of the same sample after filtration through a 0.45 um filter.

PRINCIPLE:

Colloidal clay and other suspended particulate matter interfere with the spectrophotometric analysis for TFM by partially blocking the passage of light through water samples. Filtration through a Whatman paper filter removes most particulate matter, but water samples containing fine particles require additional filtration through a smaller pore-size filter.

SAMPLE COLLECTION AND PRESERVATION:

See TOP:018.x; Procedures for Conducting Spectrophotometric Analysis for TFM in Stream Water

EQUIPMENT REQUIRED:

Millipore or equivalent vacuum filtration funnel
Erlenmeyer vacuum flask with heavy-wall vacuum hose
Vacuum pump
Syringe with Lure-lock tip
Filter-disc holder
47 mm filter circles with 0.45 μm pore size
Disposable syringe filters with 0.45 μm pore size

POTENTIAL INTERFERENCES:

Water samples with heavy loads of particulate may quickly obstruct filters. Pre-filter samples through a Whatman 2V paper filter (or equivalent).
Some syringe filters (e.g.: Target brand) remove TFM from the water sample. New filters must be tested.

SAFETY:

No special safety precautions are required.

DISPOSAL:

Wastes from field laboratories are collected and emptied into the stream receiving treatment.

REAGENTS:

No special reagents required.

PROCEDURES:

- I. Preparation of sample
 - A. The sample is buffered and pre-filtered according to procedures in TOP:018.x.
 - B. A sub-sample of about 100 mL or greater is necessary for vacuum or syringe filtration.
- II. Filtration of sample
 - A. Vacuum filtration
 1. Place a filter disc in the vacuum funnel and insert the funnel into the Erlenmeyer flask.
 2. Attach the hose to the flask and to the vacuum port or directly to the vacuum pump.
 3. Pour about 30 - 40 mL of pre-filtered sample into the funnel and turn on the vacuum pump.
 4. When the funnel is empty of sample, turn off the pump, rinse the sides of the flask with the filtered sample, and then discard the rinse.
 5. Again place the funnel into the flask, fill the flask with pre-filtered sample, and turn on the vacuum pump.

6. When the funnel is empty of sample, turn off the pump, and pour the sample into a metal beaker for heating and subsequent analysis.
7. Repeat the process for additional samples until the speed of filtration slows from particulate collected on the filter disc.
8. Disassemble the vacuum filter funnel, replace the disc, and continue with the filtration process.

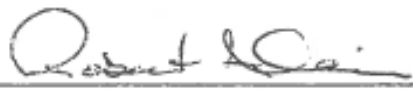
B. Syringe filtration

1. New shipments of filters must be tested to detect loss of TFM to the filter.
 - a. Measure absorbance of a TFM standard.
 - b. Filter the standard through the new filter and again measure absorbance.
 - c. Compare absorbencies to detect adsorption of TFM onto filter.
 - d. Differences of $>.005$ absorbance units indicates unsuitability of filters.
2. Place a filter disc in the syringe filter holder and attach the holder to the syringe or attach disposable syringe filter.
3. Pour about 10 - 20 mL of pre-filtered sample into the syringe, insert the plunger, shake the syringe to rinse the sides, and slowly force the sample through the filter. Discard the filtrate.
4. Pour about 30 - 40 mL of pre-filtered sample into the syringe, insert the plunger, and slowly force the sample through the filter into a metal beaker for heating and subsequent analysis.
5. Change filter discs or disposable filters between samples to avoid clogging.

REFERENCES:

None

This procedure has been reviewed and approved by the undersigned representatives of the U.S. Fish and Wildlife Service and Fisheries and Oceans Canada.

REVIEWED/APPROVED  DATE 2-9-11
Field Supervisor (U.S.)

REVIEWED/APPROVED  DATE 20 9, 2011
Division Manager (Canada)