FILTERED SAMPLES 5.2

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Filtration is the physical process used to separate the particulate and aqueous fractions of a water sample. Samples are filtered for several purposes; for example, to remove microorganisms in order to help preserve ambient analyte concentrations, to remove suspended materials that interfere with specified analytical procedures, and to determine chemical speciation and fractionation of trace elements for geochemical studies.

Study objectives and the analytes targeted for study dictate the filtration method and equipment to be used. Ambient concentrations of filtered analytes typically can be near the limit of detection; therefore, field personnel must pay strict attention to possible sources of contamination from sampling and processing equipment, construction material of the chamber frame and of the filtration equipment, and the way the equipment is handled. (Equipment and supplies used to filter water samples are described in detail in NFM 2.)

- Check the composition and pore size of the filter medium and the effective filtration area of the filter; these can affect the quality and accuracy of the data and can compromise data-quality requirements.
- To minimize airborne contamination,
  - Filter samples within a processing chamber.
  - Add chemical treatments to samples within a separate preservation chamber.

Filter samples during or immediately after sample collection.

5.2.1 INORGANIC CONSTITUENTS

Most filtration systems currently used by the USGS are appropriate for filtering wholewater samples, if the limitations of each system are taken into account. **Standard USGS procedure is to filter inorganic-constituent wholewater samples through a 0.45-micrometer (µm) pore-size disposable capsule filter.** Filtration through media with pore sizes other than 0.45 µm or with other equipment (such as tangential-flow devices) depends on the use and interpretation of the data and can yield substantially different results for trace-element concentrations.

Data-quality requirements for interpretive studies of ground-water and surface-water chemistry can dictate filtering the sample through a nominal pore size of ≤ 0.2 µm. The quality-assurance procedures used for samples filtered through the 0.45-µm nominal-pore-size capsule, plate, or other filtration equipment also are required for the ≤ 0.2-µm filters. If concentrations of target analytes are analyzed at sub-parts-per-billion levels, more stringent QA/QC measures are needed. Such samples can be filtered through a plate filter or other filtration equipment (for example, a 47-mm-diameter vacuum-filter unit) as long as the equipment used is approved by the study or program, data-quality requirements are met, and additional quality-control samples are collected. For additional information on filtration artifacts, procedures, and equipment, see Kennedy and others (1976), Salonen (1979), McCarthy (1988), McCarthy and Zachara (1989), Puls and Barcelona (1989), Ward and Harr (1990), Horowitz and others (1992, 1994), Williams and others (1993), Robards and others (1994), and Koterba and others (1995).

Cleaning and conditioning of various filter media used for inorganic constituents are summarized in table 5-3. Contamination during sample filtration can be reduced by following the instructions given for cleaning, conditioning, and handling of the filter media.

Table 5-3. Field cleaning and conditioning procedures for media used to filter samples for inorganic-constituent analysis

[µm, micrometer; mL, milliliter; sample, the water to be sampled; µg/L, microgram per liter; mm, millimeter; HNO_3, 1 molar solution of ultrapure-grade nitric acid; HCl, 1 molar solution of ultrapure-grade hydrochloric acid; nutrients, nitrogen and phosphorus species; DIW, District- or laboratory-produced deionized water of known quality, ASTM Type-1 grade or better; IBW, laboratory-produced inorganic-grade blank water; *N*, normal; >, greater than]

Description	Filter media	Field cleaning/ conditioning	Application
Disposable capsule filter[1] (Polypropylene)	Polysulfone, pleated membrane, 0.45-µm or 0.2-µm pore size	Clean with 1,000 mL DIW and remove residual DIW[2] Condition with 25 mL sample	Major ions and nutrients; trace elements with concentrations > 1 µg/L; radio-chemicals and isotopes
Plate filter — 142 mm (Polycarbonate or acrylic)	Cellulose nitrate, tortuous path (0.45 and 0.1 µm are most commonly used pore sizes)	Clean with 500 mL DIW and extract residual DIW Condition with 100 mL sample	Major ions and nutrients; trace elements if concentrations > about 100 µg/L
Cartridge or hand-pressure filter assembly—47 mm (Polypropylene or fluorocarbon polymer)	Cellulose nitrate, tortuous path (0.45, 0.2, and 0.1 µm are most commonly used pore sizes)	Clean with 100 mL DIW and remove residual DIW Condition with 20 mL IBW or 10 mL sample	Major ions and nutrients; trace elements with concentrations at about 1 µg/L or greater
Cartridge or hand-pressure filter assembly—47 mm (Fluorocarbon polymer)	Polycarbonate (such as Nuclepore), direct path (0.40 and 0.1 µm are most commonly used pore sizes)	Soak in HNO_3 rinse with IBW.[3] Remove residual IBW Condition with 20 mL IBW or 10 mL sample	Major ions and nutrients; trace elements with concentrations at about 1 µg/L or greater

[1]Example: Gelman Sciences 12175 (0.45 µm); 600 square-centimeter filtration area. Other disposable capsule filters are available that have different effective filtration area, media type, and media pore size.

[2]For trace-metal analyses at nanogram-per-liter concentration levels, first acid rinse with 500 mL of 1-*N* HCl (polysulfone membranes cannot withstand HNO_3).

[3]Substitute HCl for HNO_3 if sampling includes nutrients.

- Before filtering, designate one member of the processing team as Clean Hands (CH) and another member as Dirty Hands (DH) if using the CH/DH method (NFM 4).
- Wear appropriate, disposable, powderless gloves throughout the process. Vinyl gloves are adequate for inorganic-constituent sampling.
- Filter the samples within a processing chamber to minimize the possibility of contamination.

5.2.1.A Capsule-Filter Procedure

The capsule filter is a disposable, self-contained unit composed of a pleated filter medium encased in a plastic housing that can be connected in-line to a sample-delivery system (such as a submersible or peristaltic pump) that generates sufficient pressure (positive or negative) to force water through the filter. Filter media are available in several other pore sizes, but 0.45 µm is the pore size used routinely for most studies at this time. The capsule filter is required for most studies when filtering samples for trace-element analysis and is recommended when filtering samples for major-ion or other inorganic-constituent analyses.

The following instructions implement Clean Hands/Dirty Hands (CH/DH) techniques and the other QA procedures that are required for trace-element samples with analyte concentrations at the parts-per-billion (ppb) level and that are recommended as good field practice for all samples.

- The DH team member performs operations that are outside of the processing chamber and the CH team member performs operations inside the chamber. DH and CH must wear appropriate disposable, powderless gloves (gloves).
- Preclean capsule filters (step 5 below) before leaving for the field to save field time.

Fill bottles for filtered samples in this sequence: FA (trace elements) ➜ FAM (mercury) ➜ FA and FU (major ions) ➜ FCC or FCA (nutrients) ➜ FAR and all other samples.

To prepare the work space, sample bottles, and capsule filter:

1. *CH*/*DH*: Put on one or several layers of gloves.
2. *CH*: Assemble processing chamber, attach chamber cover, and change gloves. Place capsule filter and sample bottles into chamber, and run discharge end of peristaltic pump tubing into the chamber. Open DIW[7] container and cover it with a plastic bag to prevent contamination from airborne particulates.
3. *CH*/*DH*: (*CH*) Insert intake end of peristaltic pump tubing through the plastic covering and into a l-L container of DIW.
   a. (*DH*): Attach tubing to peristaltic pump head and pump DIW to fill tubing.
   b. Discharge waste rinse water through a sink funnel or a toss (waste) bottle.
4. Discard DIW stored in DIW-prerinsed sample bottles. If sample bottles were not DIW-prerinsed by field personnel:
   a. Wearing gloves, rinse off exterior of each bottle.
   b. Pour DIW into bottle until about one-tenth full.
   c. Cap bottle and shake vigorously about five times.
   d. Uncap and empty bottle.
   e. Repeat b–d of step 4 twice (for a total of three times).
   f. Recap bottles until ready to field rinse.

[7]Office of Water Quality Technical Memorandum 92.01 describes the quality required of the deionized water.

5. **Clean the capsule filter.** If the capsule filter was precleaned, go to the sections that follow on "To filter a composite sample" or "To filter a pumped sample," as appropriate. The steps below comprise sufficient precleaning of the filter for inorganic analytes at the parts-per-billion (ppb) concentration level. More rigorous precleaning procedures that include rinsing with trace-metal-grade hydrochloric acid are required for samples containing ppb concentrations of target analytes (table 5-3). **Only CH touches those portions of tubing that will be in direct contact with the DIW or capsule filter.**

   a. *CH*: In the processing chamber, remove capsule filter from protective bags.
      - Attach pump tubing to inlet connector of capsule filter, keeping tubing as short as possible. **Make sure direction of flow through capsule filter matches the direction-of-flow arrow on the side of the capsule.**
      - To help minimize aeration of the sample (usually for ground-water samples), secure a short length of clean fluorocarbon polymer tubing onto capsule filter outlet to extend into the sample bottle so the bottle can be filled from the bottom up.

   b. *CH/DH*: Pump 1 L of DIW through capsule filter; discharge waste rinse water through a sink funnel or to a toss bottle.
      - *DH* operates the pump at a low speed.
      - *CH* inverts the capsule filter so the arrow on the housing is pointing up. (This expels trapped air from the capsule during initial filling; do not allow water to spray onto the chamber walls.)

   c. *DH*: Remove tubing from DIW reservoir and continue to operate pump in forward mid-range speed position to drain as much of the DIW that remains in the capsule filter as possible. While pump is operating, shake capsule filter to help remove any entrained DIW.

   d. *CH*: Detach capsule filter from peristaltic pump tubing, put it into a clean, sealable plastic bag, and place in a corner of the processing chamber until ready for use.

Filtration procedures differ somewhat, depending on how the sample is collected. If the sample is collected using discrete collection equipment, such as the surface-water bag or bottle sampler or ground-water bailer, use the procedures described below in "To filter a composite sample." If the sample is collected by pumping it directly from the source, use the procedures described below in "To filter a pumped sample." Ground-water samples usually are not collected as a composite. If samples are to

be withdrawn from a well using a bailer, consider using a bailer to which the capsule filter or other filtration device can be connected inline to the bailer bottom-emptying device. Pouring a sample from the top of the bailer into another receptacle aerates the sample and therefore is not a generally recommended procedure for processing ground-water samples.

To filter a composite sample (generally for surface water):

1. Field rinse peristaltic pump tubing with the water to be sampled.
    a. *CH:* Rinse the outside of each end of the peristaltic pump tubing.
    b. *CH*: Transfer intake end of peristaltic pump tubing into composite sample. If a churn splitter is used, transfer intake end of peristaltic pump tubing through churn funnel and reseal plastic bag around the tubing.
    c. *DH*: Start peristaltic pump to slowly pump sufficient sample to completely fill pump tubing.
    d. *CH*: Discard rinse water through the sink funnel or into a toss bottle or other receptacle and dispose of appropriately. Prevent water from ponding in the processing chamber.
    e. *DH*: Stop peristaltic pump after tubing is field rinsed.
2. Field rinse capsule filter:
    a. *CH*: Remove cleaned capsule filter from plastic bag and attach discharge end of the peristaltic pump tubing to the inlet connector on the capsule filter.
        - A clean, small plastic hose clamp may be used to secure the discharge end of the tubing to the capsule filter inlet connector.
        - Check that the direction of sample flow through the capsule filter matches the direction of the arrow on the capsule.
    b. *DH:* Operating the pump at low speed, pump sample through the tubing to the capsule filter.
    c. *CH:* Turn capsule filter so that the outlet is pointing up (arrow on capsule housing is pointing up) and flow of the sample forces trapped air out of the capsule filter while it is filling. **Do not let sample spray onto chamber cover.**
        - The chamber cover must be changed if sample has sprayed onto it.
        - If some water that sprayed onto the chamber cover has dripped into the sample bottle, discard the bottle, change the cover, and collect a new sample.

d. *DH:* Stop the peristaltic pump as soon as the capsule filter is full of sample and all air in the capsule filter has been expelled.

> TECHNICAL NOTE: The goal is to minimize clogging the filter medium with suspended materials by minimizing the volume of sample that will be used to field-rinse the filter.

3. Collect sample filtrate.

a. *CH*: Check that there is a tight connection between the pump tubing and the capsule filter.

DH: Check that the intake tube is properly inserted in the sample and start the pump.

CH: Collect a maximum of 25 mL of the water to be sampled as it discharges through the filter. **Do not exceed 25 mL**.

CH: Field rinse a precleaned 250-mL FA bottle for trace-element sample only with sample filtrate.

DH: Stop the pump in time to prevent losing filtrate to waste.

CH: Cap bottle, shake vigorously, and then discard rinse water into appropriate receptacle.

b. *DH:* Start pump and resume flow from pump to the filter.

CH: **Filter only the next 200 mL of the sample** into the trace-element FA bottle (fill to top of upper lip of standard 250-mL polyethylene bottle). Cap bottle securely and set aside for chemical treatment.

c. *DH:* Stop the pump after the trace-element FA bottle is filled.

d. If a filtered mercury sample is required, restart pump and repeat steps 3a–c, substituting a FAM bottle for the FA bottle.

e. *CH:* Field rinse any remaining sample bottles for inorganic analyses. **Use no more than a total of 100 mL of filtrate per capsule filter to field rinse any remaining bottles for filtered sample.**

f. Fill remaining bottles in the following order: (1) major cations, (2) nutrients and major anions (including alkalinity), (3) radiochemicals (Appendix A5-A), and (4) stable isotopes. Cap each bottle immediately after filling.

To filter a pumped sample (usually ground water):

Ground-water samples usually are withdrawn from a well by means of a submersible pump. Note that this method might be appropriate for some surface-water samples. The capsule filter or other filter assembly is connected inline with the sample tubing in order to collect samples directly from the well.

- When sampling ground water, DH should check that the turbidity values recorded at the end of purging have remained stable. Equipment changes or adjustments that disrupt sample flow can affect sample turbidity and should be avoided. If sample flow is disrupted, pump for several minutes until ambient turbidity values are reestablished.
- **Maintain a smooth, uniform flow.** Do not stop pump or divert flow from capsule filter or other filter assembly during bottle field rinse or filtration, if possible.

  TECHNICAL NOTE: If using a three-way valve, changing the setting to divert the flow of sample being pumped to the filter with a submersible pump can cause air bubbles to form, can air-block the filtration equipment, and can cause changes in pumping rate that could result in increased turbidity. These effects should be avoided to preserve sample integrity; therefore, flow to the filter should not be stopped until all filtration is complete.

1. Field rinse the capsule filter with sample water:

   a. *CH:* Ensure that the sample line is full of sample and free of bubbles; then attach the discharge end of the sample line to the inlet connector on the capsule filter.
      - Practice your technique for attaching the capsule filter to the tubing carrying flowing water so that water does not spray onto chamber walls.
      - Check that the direction of flow matches the direction of the arrow on the capsule.

   b. *DH:* Adjust the sample flow through the sample line to the capsule filter, keeping a slow rate of flow.

   c. *CH*: Turn the capsule filter so the outlet is pointing up (arrow on capsule housing is pointing up) and the flow of sample forces trapped air out while the capsule filter is filling.
      - Do not allow water to spray onto chamber walls.
      - The capsule filter should be full of sample. No air should be left in the capsule filter.

d. Field rinse bottles for inorganic-constituent filtered samples with sample filtrate (section 5.0.3). Use bottles that were already rinsed three times with DIW. Determine whether the potential clogging of pores in the filter medium is of concern for your samples (see TECHNICAL NOTE below).

CH: Fill a 250-mL FA bottle for trace elements with 25 mL of sample filtrate; cap, shake vigorously, and discard rinse water into appropriate receptacle.

CH: Fill a FA bottle for trace elements with about 200 mL of sample filtrate (to top of upper lip of 250 mL bottle). Cap bottle and set aside for chemical treatment.

CH: If a mercury sample is required, field rinse and fill a FAM bottle using the same procedure as for the 250-mL FA bottle.

CH: Field rinse remaining bottles, trying to use no more than an additional 100 mL of sample filtrate.

> TECHNICAL NOTE: Depending on sample turbidity and composition, the nominal pore size of filter media tends to decrease as the volume of sample passed through the filter increases because pores are clogged by sediment loading or mineral precipitation on the filter (Horowitz and others, 1994). Ground water with turbidity ≤5 NTU should not affect filter pore size appreciably. To minimize the chance of filter clogging, limit the volume of sample passed through the filter by eliminating the field rinse—be sure that you use clean bottles and fill them one after the other. For ground-water sampling, do not stop the pump during the field-rinse and sampling process.

e. *CH:* Collect sample filtrate immediately into any remaining bottles in the following sequence (flow rate should be slow enough to avoid splashing sample out of the bottle): (1) major cations, (2) major anions and nutrients (including alkalinity sample for field titration), (3) radiochemicals (check Appendix A5-A for bottle-rinse and filtration requirements), (4) stable isotopes.

f. *CH*: Cap each bottle immediately.

Rinse FA, FU, FAM, FCA, and FCC bottles with filtered sample—not with raw sample.

After collecting filtered samples:

1. *CH*: If samples require chemical treatment, place FA bottles in the preservation chamber and go to section 5.4.
2. For filtered samples that do not require chemical treatment:
   a. *CH*: Set samples outside processing chamber.
   b. *DH*: Check that information on the bottle label is correct and complete.
   c. *DH:* Pack samples that require chilling in ice or refrigerate immediately.
   d. *DH:* Pack remaining samples for shipping (section 5.5).
3. Rinse all reusable equipment with DIW immediately—before equipment dries.
   - *CH*: If equipment will be reused at another site before returning to the office, rinse immediately with DIW and field clean tubing and other sample-wetted parts of the equipment using the prescribed cleaning procedures (NFM 3).
   - *CH*: If equipment or tubing will not be reused before returning to the office, rinse immediately with DIW and store rinsed tubing and equipment in plastic bags for office or laboratory cleaning.
4. **Discard the capsule filter after filtering each sample—do not reuse.**
5. Document the filtration procedures used on field forms and in field notes.

Use of the 0.45-µm disposable capsule filter for trace-element samples is required for many USGS programs.

5.2.1.B Plate-Filter Procedure

The filtering procedure using a 142-mm-diameter plastic plate-filter assembly is described below. The procedure remains basically the same for plate-filter assemblies of different diameters.

Prepare and precondition plate-filter assembly:

The following instructions pertain to either a 142-mm-diameter or a 47-mm-diameter plastic plate-filter assembly and require that the assembly components have been rigorously cleaned (NFM 3). To avoid recleaning in the field, prepare a set of filtration equipment for each well or surface-water sampling station. (Ignore Step 3 below if plate-filter assembly has been rinsed in the office.)

1. *CH*: Put on gloves. In a processing chamber, open a clean plate-filter assembly and load with the filter.
   a. Using nonmetallic forceps, place the bottom retaining screen on the base of the filter assembly. **Do not interchange bottom and top retaining screens.**
   b. Place the filter on top of bottom retaining screen using clean, blunt plastic or ceramic forceps. Do not touch the filter with hands (gloved or ungloved).
      - Be sure that only one filter is transferred from its original container directly to the plate of the filter assembly. Take care not to transfer the paper liner that separates each filter.
      - The filter should never be removed from the original container until each is transferred to the plate-filter assembly for use. (Exception: polycarbonate (Nuclepore) filter medium is precleaned with acid solution. If transferring one of these, hold the filter with forceps and rinse off acid with inorganic blank water (IBW) dispensed from wash bottle.)
   c. Using forceps, place the top retaining screen on top of the filter.

      TECHNICAL NOTE: If filtering sediment-laden water, a prefilter can be placed between the filter and the top retaining screen.

   d. Dispense 10 to 20 mL of DIW from a wash bottle onto the filter.

e. Close the plate-filter assembly by aligning the top and bottom plates and lightly tightening the plastic bolts, followed by finger tightening opposite pairs of bolts. **Overtightening can cause the plate-filter assembly to warp and leak.** Check that O-rings are in place before closing the assembly. Change gloves.

2. *DH/CH*: Pass the discharge end of the pump tubing through the hole in the side or top of the processing chamber. **Only the CH team member touches sections of tubing that will be in direct contact with the plate-filter assembly.**

   - Keep tubing as short as practical.
   - Attach a short piece of clean tubing to outlet connector of plate-filter assembly.

3. *DH/CH*: Rinse the plate-filter assembly with DIW, using a peristaltic pump, as follows **(rinsing must be repeated each time a clogged filter is replaced with a new filter)**:

   a. *CH*/*DH*: Place intake end of peristaltic pump tubing into a 500-mL container of DIW. Turn pump on low speed.

   b. *CH*: Open the air-vent valve on top of the plate-filter assembly. Tilt the filter assembly slightly to the side and squeeze the outlet tube closed to force trapped air out through the vent. Release the outlet tube. (Venting trapped air is necessary because air bubbles will reduce the effective filtering area by preventing sample from passing through the filter.)

   c. *CH*: Close valve when top is filled with sample.

   d. *CH*: Pump sample through the plate-filter assembly and discard this field-rinse water through the sink funnel or into the toss bottle to prevent the water from ponding in the bottom of the processing chamber.

   e. *CH*/*DH*: Remove intake end of the pump tubing from the DIW container and continue to pump, draining as much of the remaining DIW from the plate-filter assembly as possible.

4. If using a peristaltic pump to transfer the sample to the processing chamber (go to step 5 if sample delivery is with a submersible ground-water pump):

   a. *CH*: Rinse intake end of the peristaltic pump tubing with the water to be sampled.

   b. *CH*: Transfer intake end of the peristaltic pump tubing into the container of sample. If a churn splitter is used, transfer the intake end through the churn funnel and reseal the plastic bag around the tubing.

c. *CH*: Remove peristaltic pump tubing from the inlet connector of the plate-filter assembly and hold the end of the tubing over the sink funnel or toss bottle.

d. *DH/CH*: Start the peristaltic pump in the forward position at slow speed and pump sufficient sample to fill and rinse all pump tubing. Stop the pump after the tubing is rinsed.

5. *CH*: Attach the discharge end of the peristaltic-pump or submersible pump tubing to the inlet connector of the plate-filter assembly.

   - Keep tubing as short as practical.
   - A clean, small, plastic hose clamp can be used to secure the discharge tubing to the inlet connector.

6. *DH*: Start sample flow to the plate-filter assembly.

7. *CH*: Vent trapped air and rinse plate-filter assembly as instructed in steps 3 b–d above.

   - If using a peristaltic pump, turn pump on low speed.
   - If using a submersible pump, maintain a slow and steady flow rate.

8. *CH*: Rinse appropriate sample bottles once with filtrate. Filter no more than 100 mL of sample for the final rinse of all sample bottles that require rinsing.

9. Filter samples, filling bottles in the following order, as applicable to study objectives and sample designation:

   a. Trace elements

      TECHNICAL NOTE: Study objectives and data-quality requirements govern procedures to be used if the filtered trace-element sample is to reflect concentrations of analytes in true solution (the dissolved fraction). Such interpretive studies of ground-water or surface-water chemistry commonly use ≤0.1-µm filter media and plate-filter assembly or a tangential flow method of phase separation. Note that any deviation from the standard procedure for collecting filtered trace-element samples through the 0.45-µm capsule filter must be documented and reported with the analytical results.

   b. Major cations

   c. Nutrients, major anions, and alkalinity sample

   d. Radiochemicals

   e. Isotopes

10. *CH*: If the filter medium clogs before the needed volume of water is filtered, carefully remove the filter and replace with a new filter. Repeat steps 1 through 7. Cap each bottle immediately after filling.

11. **If samples require chemical treatment ➜ Go to section 5.4.**

12. *DH*: After filtration,

    a. Check that information on the bottle label is complete and set the samples aside for shipping (section 5.5). Samples that must be chilled need to be refrigerated or packed in ice as quickly as possible and maintained at 4°C without freezing.

    b. Disconnect and disassemble the plate-filter assembly. **Discard the used filter.**

    c. Rinse all equipment with DIW immediately after use and before it dries. Equipment that has dried after sampling without being rinsed or cleaned needs to be cleaned vigorously with a detergent and rinsed with DIW before the next use. Nonmetallic equipment must also be acid rinsed.

    d. Put rinsed tubing in a plastic bag for cleaning at the office laboratory.

    e. If equipment is to be used at the next site, field clean all the equipment using the procedures described in NFM 3. Field cleaning between sampling sites is carried out while still at the sampling site.

13. Document on field forms and in field notes any modifications to the filtration procedures used.

ORGANIC COMPOUNDS 5.2.2

Standard procedure for phase separation of general trace-organic compounds involves the use of a stainless steel or aluminum 142- (or 293-) mm-diameter plate-filter assembly with glass-fiber filter media and a valveless piston or fluorocarbon polymer diaphragm-head metering pump (section 5.2.2.A). Equipment and procedures differ when filtering samples for dissolved and suspended organic carbon (section 5.2.2.C) and optionally for organonitrogen herbicide analyses (section 5.2.2.B). Required conditioning for filter media is discussed below and summarized in table 5-4.

Table 5-4. Field conditioning requirements for media used to filter samples for organic-compound analysis

[mm, millimeter; mL, milliliter; PBW, pesticide-grade blank water; sample, the water to be sampled; methanol, pesticide-grade methanol; DIW, deionized water]

Filtration equipment *Application*	**Construction materials**	**Filter media**	**Filter cleaning and conditioning**[1]
Plate-filter assemblies: 142 or 293 mm *General trace organic compounds*	Stainless steel or aluminum	Glass-fiber filter[2]	Wet with PBW: 10-20 mL (142 mm) or 50-75 mL (293 mm) Condition with 100-125 mL sample
Disposable capsule filter: 25 mm *Organonitrogen herbicides*	Polypropylene	Nylon	Rinse with 10 mL of methanol No conditioning
Pressure filter apparatus: 47 mm *Dissolved and suspended organic carbon*	Stainless steel or fluorocarbon polymer	Silver metal	Rinse with 100 mL PBW or District-prepared organic-grade DIW Condition with 10-15 mL sample

[1]Do not reuse filters.

[2]Use only glass-fiber filters that have been adequately baked.

The procedures for filtering samples for analysis of trace-organic compounds, including volatile organic compounds, pesticides, and base-neutral compounds, are summarized from Sandstrom (1995). CH/DH techniques and associated QA procedures for inorganic analytes with parts-per-billion concentrations are not required for organic analytes but are recommended as good field practices to maintain the integrity of sample chemistry. Field personnel must wear disposable, powderless gloves (gloves). These gloves must be able to withstand any solvents or other chemicals that will be used during sample processing and equipment cleaning. Equipment and supplies used to filter different types of organic compounds are described in NFM 2. Additional information about organic-compound filtration can be found in Ward and Harr (1990), Manning and others (1994), Shelton (1994), and Koterba and others (1995).

Plate-Filter Procedure 5.2.2.A

Read through the procedures described in Sandstrom (1995) and presented in tables 5-4 and 5-5 and in figure 5-1. Obtain the equipment needed (table 5-5), test equipment operation, and collect an equipment blank if needed. Filtering samples for organic-compound analysis inside a processing chamber and using Clean Hands (CH)/Dirty Hands (DH) techniques is not mandatory but is recommended.

Table 5-5. Equipment for filtration of water-sediment samples for determination of organic compounds

[Modified from Sandstrom (1995); FEP, fluorinated ethylene-propylene; mm, millimeter; mL/min, milliliter per minute; L, liter; µm, micrometer; °C, degree Celsius]

Item	Description of equipment
	Container for unfiltered sample. Clean, laboratory-grade glass bottles with fluorocarbon polymer-FEP-lined lids.
	Fluorocarbon polymer-FEP tubing, 6.35-mm outside diameter.
	Union, 6.35-mm tube (Swagelok Company, Solon, Ohio, No. SS-400-6 or equivalent).
	Fluorocarbon polymer-FEP convoluted tubing, 6.35-mm outside diameter (Cole-Parmer Instrument Company, Chicago, Ill., No. L-06486-02 or equivalent).
	Tube fitting, 6.35-mm diameter tube to 6.35-mm diameter pipe thread (Swagelok Company, Solon, Ohio, No. SS-400-1-4 or equivalent).
	Pump, ceramic-piston, valveless, with 12-volt direct current motor, capable of pumping from 0 to 500 mL/min (Fluid Metering, Inc., Oyster Bay, N.Y., Model QB-1 CSC or equivalent).
	Battery, 12-volt direct current.
	Tube fitting, 6.35-mm diameter tube to 9.53-mm diameter pipe thread (Swagelok Company, Solon, Ohio, No. SS-400-1-6 or equivalent).
	In-line plate-filter assembly, aluminum (or stainless steel), 142-mm diameter (Geotech Environmental Equipment Inc., Denver, Colo., No. 0860 or equivalent).
	Glass-microfiber filter media, binder-free, 142-mm diameter, 0.7-µm nominal pore size (Whatman Inc., Clifton, N.J., GF/F grade, No. 1825C142 or equivalent). Note: The filters must be baked at 400°C for at least 2 hours and kept wrapped in aluminum foil before use.
	Bottle for filtered samples, amber borosilicate glass, 1 L with fluorocarbon polymer-FEP-lined cap.
	Fluorocarbon polymer-FEP squeeze (wash) bottle for organic-grade blank water.
	Stainless-steel forceps for handling the filters.

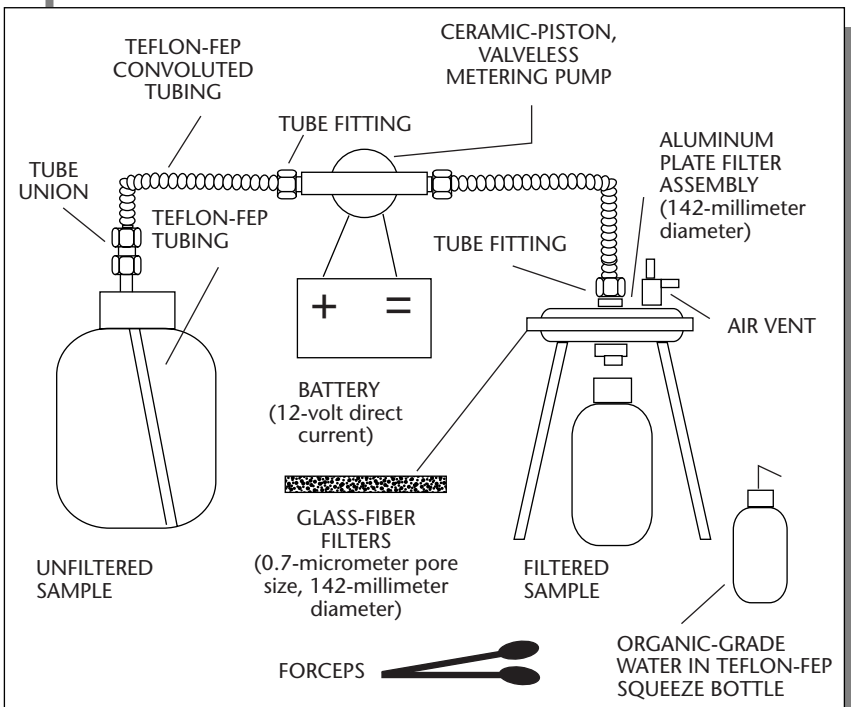

Figure 5-1. An equipment system suitable for filtering samples for analysis of organic compounds (from Sandstrom, 1995).

To filter sample for analysis of general trace-organic compounds in solution:

1. *CH/DH*: Wear appropriate (latex or nitrile) gloves throughout sample processing. Change gloves after setting up equipment. (Wearing several layers of gloves can save time.)
2. *CH*: Load the filter onto the plate-filter assembly within the processing chamber.
   a. Open precleaned plate-filter assembly.
   b. Place one stainless steel support screen on the base of the plate-filter assembly—Use stainless steel forceps.
   c. Place one clean 0.7-µm pore-size glass microfiber filter on top of the screen. **Do not touch the filter with fingers; use stainless steel forceps.**
   d. Wet the filter with a few drops of pesticide-grade blank water (PBW) from a fluorocarbon polymer wash bottle to help keep the filter in place as the unit is assembled.

e. Close plate-filter assembly—Align top and bottom plates. **Lightly tighten** the locking bolts or locking ring. Attach a short length of fluorocarbon polymer tubing to the outlet of the plate-filter assembly to channel filtrate to a toss bottle, sink funnel, or drain.

f. Add 10 to 20 mL of PBW rinse water through the inlet in the upper plate to wet the filter completely before tightening the clamps. (This rinse also helps prevent damage to the filter: a dry filter might rupture when the plate-filter assembly is tightened.)

g. Tighten the locking bolts or ring by hand. **Overtightening can cause the plate-filter assembly to warp and leak and the filter to rupture.**

3. *CH/DH*: Rinse the pump tubing (from a metering pump) or the sample tubing (from a submersible ground-water pump) with the water to be sampled. Discard rinse water into a sink funnel or toss bottle.

4. Set up the pump for filtration.

   - *CH*: If using a metering pump, place intake end of tubing into the container holding the sample. Attach discharge end of pump tubing to the inlet connector of the plate-filter assembly. Use a stainless steel compression fitting of the appropriate size to secure the discharge hose to the inlet connector.
   - *CH*: If using a submersible pump, attach discharge end of the sample tubing from the pump to the plate-filter assembly, keeping tubing as short as practical. Use a stainless steel compression fitting of the appropriate size to secure the discharge hose to the inlet connector.

5. *CH*: Rinse and condition the filter. The total volume of sample passed through the filter, including rinse water, needs to be accurately determined to ±1 mL and recorded in the field notes.

   a. Turn on the metering pump at low speed or open the sample tubing from the submersible pump and operate at a low flow rate.

   b. Open the air-vent valve located on top of the plate-filter assembly. Tilt the assembly slightly to the side to allow all trapped air to escape (vent).

   c. Close the air-vent valve when water discharges through the valve.

d. Pass 100 mL of sample through the filter to remove any residual liquids from the cleaning or prewetting procedures. If concentration of organic compounds in suspended-material phase is to be determined:

   i. Capture the rinse water in a dry, clean, graduated cylinder.

   ii. Measure and record the actual volume of sample passed through the filter.

e. Discard rinse water to a sink funnel or toss bottle.

6. *DH*: Tare the weight of a clean, baked, glass sample bottle. (First check to see if this is required for the analytical procedures to be used.)

   a. Set up, level, zero, and check the accuracy of the balance with a reference weight. Record accuracy in field notes.

   b. Tare the weight of a dry, clean, capped 1-L amber bottle, and record the weight. Remove the bottle cap.

7. Filter and weigh each sample. (Do not field rinse baked, glass sample bottles.)

   a. *CH*: Resume the flow of sample through the plate-filter assembly.

   b. *CH*: Place the appropriate sample bottle under the outlet of the plate-filter assembly.

   c. *CH*: Collect approximately 1 L of filtered sample for each analytical schedule, but leave headspace in each bottle. If the filter medium becomes too clogged to proceed, go to step 13 below.

   d. *DH*: Cap the bottle(s) and pass sample(s) out of chamber. Wipe the bottle dry with a lint-free laboratory tissue, such as Kimwipe™, to remove any condensation from the outside of the sample bottle.

   e. *DH*: Weigh and record the amount of sample filtered (total weight minus tare weight of bottle).

   f. Chill samples immediately and maintain at or below 4°C without freezing for shipment to the laboratory (section 5.5).

8. *CH*: Remove as much water as possible from the inside of the plate-filter assembly by using the metering pump to pump air through the sample tubing, or by pulling water out through the outlet nozzle with a peristaltic pump, or by using a syringe to apply positive air pressure to the inlet connector. This removes any residual sample and prevents spilling the water-sediment slurry when the plate-filter assembly is disassembled.

9. *CH*: If sediment collected on the filter is to be analyzed for organic compounds:

    a. Carefully disassemble the top of the plate-filter assembly.

    b. Using metal forceps, carefully fold the filter in half and then in half again (quarters).

    c. Transfer the filter to a baked, wide-mouth glass jar with a fluorocarbon-polymer-lined cap.

    d. Record on the jar label and on field forms the total volume of sample that passed through the filter.

    e. Chill and maintain the sediment sample at or below 4°C for shipment to the laboratory (section 5.5)

10. *DH/CH*: If sediment on the filter will not be analyzed, disassemble the top of the plate-filter assembly and remove the filter with forceps. Discard the filter appropriately. Rinse the plate-filter assembly components and tubing immediately after the filter has been removed.

11. *DH/CH*: **If the equipment is to be used at a subsequent site, field clean all equipment while equipment is still wet and before going to the next site.** Clean with detergent solution, rinse with DIW, and final rinse with methanol—do not use methanol on equipment used for TOC, DOC, or SOC samples (NFM 3). If the plate-filter assembly will not be reused before returning to the office, rinse all components with DIW. Put rinsed components and tubing in a resealable bag for cleaning at the office laboratory.

12. Document on field forms and in field notes the filtration procedures used.

13. **If the filter medium becomes clogged before the required volume of sample has been collected,** stop the metering pump or divert the sample flow from the submersible pump (see TECHNICAL NOTE below) and replace the filter with a new filter as indicated in steps a through f below.

    TECHNICAL NOTE: Diverting the flow of sample being pumped with a submersible pump by use of a three-way valve can result in a temporary increase in turbidity (NFM 4). Allow turbidity to clear after reestablishing flow through the sample tubing and to the plate-filter assembly.

    a. Remove as much water as possible from inside the plate-filter assembly. The stainless-steel or aluminum plate-filter assembly does not have an upper support screen, so the filter cannot be backflushed. Remove the inlet tubing to the metering pump from the sample and either attach tubing from a peristaltic pump to the outlet and pull residual water out, or use a syringe to apply positive air pressure to the inlet connector.

    b. Remove the clogged filter with forceps. **If sediment collected on a filter is to be analyzed for organic compounds, follow directions in step 9**.

    c. Load the plate-filter assembly with a new filter and reassemble the unit as described in step 2.

    d. Prepare the filter as described in steps 2f and 5a–d, allowing the first 125 mL of sample to remove any sediment particles that may have moved below the filter during the replacement procedure. Use a graduated cylinder to measure volume.

    e. Record the volume of sample rinsed through the plate-filter assembly if sediment collected on the filter is to be analyzed for organic compounds. Volume accuracy should be ± 1 mL.

    f. Place a tared sample bottle under the plate-filter assembly outlet, resume the flow of sample through the filter, and continue to collect the sample filtrate.

Capsule-Filter Procedure for Processing Samples for Analysis of Organonitrogen Herbicides (Optional) 5.2.2.B

The capsule-filter procedure for filtering samples for organonitrogen-herbicide analysis described below is provided if the option to process these samples onsite is selected. The steps that follow are taken from Sandstrom (1995), which includes more detailed instructions and description of the equipment, including the 25-mm-diameter disposable nylon-media filter capsule (nylon filter):

1. Before leaving for the field site, clean the nylon filter.
   a. Put on appropriate, disposable, powderless gloves (gloves).
   b. Place intake end of the metering pump tubing into the methanol.
   c. Pump about 10 mL through the nylon filter to a used-methanol disposal container.

CAUTION: Do the following if using methanol or other organic solvent:

- **Work under a fume hood or in a well-ventilated area, NOT in the field vehicle.**
- **Wear protection against skin and eye contact and do not inhale fumes.**
- **Collect methanol rinse waste into proper disposal containers and dispose of according to local regulations.**

2. At the field site, cover the field bench or table with a sheet of aluminum foil or Teflon™ to prepare a clean work surface.
3. Place equipment and supplies on the clean work surface. Remove foil or other wrapping from precleaned equipment. Change gloves.
4. Remove the nylon filter from the plastic bag. Rinse the discharge end of the pump tubing with methanol. Discard used methanol to a proper waste container. Attach the metering-pump tubing to the capsule inlet; keep tubing as short as possible.
5. If filtering with a metering pump, transfer the intake end of the pump tubing to the sample. If using a submersible pump to collect the ground-water sample, redirect the sample flow to and from the nylon filter as needed, using a manifold flow-valve system.
6. Purge air from the sample tubing. Before connecting the nylon filter, allow ground-water sample to flow through the tubing at a very low rate. This will require just a few milliliters of sample if a metering pump is used. With sample flowing, connect tubing to the nylon filter. (Use a Luer™ connector of appropriate size to secure the discharge hose to the inlet connector.)
7. Collect at least 100 mL of filtrate in a 125-mL baked amber glass sample bottle. Do not completely fill the bottle. Allow 2–3 cm of headspace. The headspace leaves space for matrix spike standards to be added (if required) and prevents sample loss if the sample freezes.
8. If the nylon filter medium becomes clogged before a sufficient amount of sample has been filtered, replace it with a new nylon filter and repeat steps 6 and 7 until at least 100 mL have been collected.
9. When filtering is complete, cap the bottle firmly. Chill and maintain the sample at or below 4°C without freezing during storage and shipment to the laboratory (section 5.5).
10. Discard the nylon filter. Field clean the pump and tubing as described in NFM 3 before using the equipment at the next site.
11. Document on field forms and in field notes the filtration procedures used.

Gas-Pressurized Filter Procedures for Processing Samples for Analysis of Dissolved and Suspended Organic Carbon 5.2.2.C

The standard filtration procedures for samples for analysis of dissolved organic carbon (DOC samples) and suspended organic carbon (SOC samples) use 47-mm-diameter, 0.45-µm pore-size, silver-metal filter media. A gas-pressurized filter assembly (SOC/DOC filter apparatus) constructed of either stainless steel or fluorocarbon polymer is required (NFM 2). In addition, either a peristaltic pump, a hand-air pump, or compressed gas (usually organic-free nitrogen gas) is used to pressurize the SOC/DOC filter apparatus and force the sample through the silver filter. Filtration procedures are identical for ground-water and surface-water samples.

A different set of procedures and separate silver filters are used to process the SOC and the DOC samples, unless suspended-material concentrations are low (up to about 30 mg/L). This section describes methods for (1) filtration of SOC samples only, (2) combined SOC/DOC sample filtration, and (3) filtration of DOC samples only.

- **If sample contains a large amount of suspended materials, at least two filtrations must be performed: one for SOC and one for DOC.**
- If sample contains low concentrations of suspended materials, filtration procedures can be combined using the same silver filter.
- Unless the study plan dictates an additional sample for quality control, only one silver filter should be needed for the SOC filtration.
  - The SOC filtration requires a minimum of 0.5 mg of suspended material in the 125-mL sample.
  - If filter clogging is a problem, or if it is difficult to obtain the 125-mL volume of sample needed for the SOC analysis, 64 mL of sample or multiple 64-mL samples can be substituted.

- Immediately after each use, rinse the filter apparatus several times with organic-grade DIW.
  - Field clean the filter apparatus while still wet if it is to be used at the next site. Otherwise, rinse, bag, and return the apparatus to the office laboratory for cleaning.
  - Thoroughly rinse the white (fluorocarbon polymer) O-ring and any other fluorocarbon polymer parts.
  - After cleaning, double-wrap all apertures and the filter apparatus with aluminum foil and place filter apparatus inside a sealable plastic bag.
- Blank water (VBW or PBW) from a freshly opened bottle should be used for quality-control samples for the DOC analysis. This blank water can also be used for prerinsing the silver filters. Once the bottle has been opened, the VBW or PBW must not be used for collection of future quality-control samples.
- Document on field forms and in field notes the filtration procedures used.

Do not use methanol or any other solvent to clean SOC, DOC, or TOC equipment (NFM 3).

TECHNICAL NOTE: Use 64-mL or 125-mL baked glass bottles (available from QWSU) instead of a graduated cylinder to measure sample volume for the DOC or combined SOC/DOC analysis. The advantage of using the baked glass bottles to measure volume is that they are certified as clean, whereas graduated cylinders can be difficult to clean adequately, especially under field conditions (Burkhardt and others, 1997).

- Bottles for DOC samples must have been baked at 400°C and meet a detection limit criterion for organic carbon of <0.1 mg/L detection limit for DOC. Do not bake the graduated cylinder because calibration will be lost.
- Volumetric accuracy of the 125-mL and 64-mL baked glass bottles is about ±1 mL.

SOC sample processing:

SOC analysis of the suspended material left on the silver filter requires that the volume of sample passed through the silver filter be measured and recorded. Determination of the volume of sample to be filtered for SOC analysis can depend on the concentrations of suspended materials; however, the concentration of humic and other substances that cause colored water, such as organic and inorganic colloids, will affect the volume that can pass through the silver filter. The sample volume that can pass through the silver filter decreases as the concentration of suspended materials increases. A graph of the historical stream stage compared to a graph of the suspended-material concentration will aid in estimating suspended-material concentrations at a given surface-water site. Guidelines for selecting the volume of sample to be filtered for SOC analysis, based on suspended-material concentrations, are shown in table 5-6.

1. Collect the SOC sample(s) in a baked glass bottle, either at the centroid of the streamflow (NFM 4) or as a subsample from the churn or cone splitter. The data-quality requirements of the study and site characteristics determine where to withdraw the sample. If collecting sample at the centroid of flow with a weighted-bottle sampler, fill the bottle to the top; this is not necessary if subsampling from the churn or cone splitter. Cap the bottle securely.

    - Use a 125-mL baked glass bottle for water with relatively small concentrations of suspended materials (concentrations approximately less than 250 to 300 mg/L) (table 5-6).
    - 64-mL baked glass bottles are recommended for samples that are colored or particulate laden.
    - A clean, graduated cylinder may be used when the volume of sample to be filtered is less than 64 mL.

Table 5-6. Guidelines for selecting the volume needed for filtration of samples for analysis of suspended organic carbon

[Guidelines are based on sand-sized materials; other physical property factors and chemical composition were not taken into account. mg/L, milligrams per liter; mL, milliliters; >, greater than]

Approximate suspended-materials concentration (mg/L)	Volume of sample to be filtered (mL)
1 - 30	250
> 30 - 300	100
> 300 - 1,000	30
> 1,000	10

2. Cover the bench or table with a sheet of aluminum foil to make a clean work surface. Put on appropriate disposable, powderless gloves. Assemble necessary equipment on the clean work surface.
   a. To remove airborne particulates, attach an in-line, 0.2-µm pore-size filter (Acrodisc 50™) to the inlet side of a dry pump hose that goes to the filter apparatus when using peristaltic or hand pumps to pressurize the apparatus.
   b. Change gloves.
   c. Remove the aluminum foil wrapping from equipment.
3. Disassemble the clean filter apparatus.
4. Using metal forceps, place a silver filter on the base of the filter apparatus between the support screen and the fluorocarbon polymer gasket, and screw the barrel onto the filter base. (There is no gasket in the fluorocarbon polymer apparatus.)
5. Pour a minimum of 100 mL of ASTM Type II reagent water (Burkhardt and others, 1997) or VBW or PBW into the barrel. Analysis of the water used must indicate less than 0.1 mg/L of organic carbon.
6. Screw the top part of the filter apparatus onto the barrel and attach a clean, dry hose, either from a peristaltic pump, hand pump, or compressed gas cylinder (use a clean metal hose clamp to secure the discharge hose to the inlet connector). Set the filter apparatus into a stand.

7. Apply pressure to start the flow of rinse water through the filter apparatus, using either a peristaltic pump or hand pump, or by regulating the flow of compressed gas (usually nitrogen).

   a. The pump pressure must be regulated to less than 15 lb/in^2.

   b. If compressed gas (for example, organic-free nitrogen) is used, proceed as follows:

      i. **Make sure that the pressure regulator valve is closed.** Turn the handle on the pressure regulator counterclockwise for several turns until the pressure-regulator valve is closed.

      ii. Open the valve to the nitrogen cylinder.

      iii. Open the pressure-regulator valve by turning the handle clockwise until up to 15 lb/in^2 registers on the pressure gage. Do not exceed 15 lb/in^2 of pressure.

   c. Discard rinse water.

8. Depressurize the filter apparatus. **Always point the apparatus away from your body, face, and other people.** When using compressed gas,

   a. Close the valve to the pressure regulator after the pressure gage shows no pressure.

   b. Close the valve to the gas cylinder.

   c. Change gloves.

Wear safety glasses when pressurizing or depressurizing the filter apparatus.

9. Remove the top of the filter apparatus carefully.

10. Shake the sample vigorously (swirl if using a graduated cylinder) to suspend all particulate matter. (This is possible even if the bottle is filled to the top.)

11. Pour an aliquot of the sample immediately into the barrel of the filter apparatus, keeping particulates suspended.

12. Screw the top part of the filter apparatus onto the barrel and pressurize to filter the sample. Follow the instructions in step 7 (above) for pressurizing the filter apparatus.

13. After an aliquot of sample has been filtered or filtrate is being collected at less than one drop per minute:

    a. Depressurize apparatus (step 8).

    b. Remove the top of the filter apparatus.

    c. Check if there is water on the silver filter and if it is covered with particulates.

    - If the silver filter is dry but not covered with particulates, add another aliquot of sample by repeating steps 10–12.
    - After the silver filter is dry and covered with particulates, continue to step 14.

    TECHNICAL NOTES:

    It is important that all the water in the barrel be passed through the silver filter, leaving the filter "dry." To accomplish this, it might be necessary to filter the sample as separate aliquots, repeating steps 10–13 until the filter is loaded to capacity.

    Shake the sample to resuspend particulates before pouring each aliquot into the barrel.

    If using a 125-mL or 64-mL bottle, it is not necessary to empty the entire sample volume. Use of a clean, graduated cylinder also is acceptable.

    It is recommended (but not required) that the sides of the barrel of the filter apparatus be rinsed with organic-grade DIW.

14. Collect the filtrate in a 50-mL or other appropriately sized graduated cylinder.

    - If additional aliquots will be filtered through the same silver filter, collect all the filtrate in the graduated cylinder.
    - When the entire filtration is complete, record the total volume of filtrate on field forms and on the Analytical Services Request (ASR) form.
    - Discard filtrate in the graduated cylinder—**Do not send to laboratory for analysis.**

15. Depressurize (step 8) and disassemble the bottom of the filter apparatus.
    a. Use a pair of metal forceps to remove the silver filter.
    b. Fold the silver filter in half with suspended material on the inside, taking care not to lose any suspended material. **Do not wrap the silver filter in aluminum foil.**
    c. Place the folded silver filter into a petri dish for SOC analysis.
    d. Close the petri dish and label it with site identification, date and time, total filtered volume of sample, and laboratory sample designation code. (The total volume of filtered sample includes the volume used to precondition the silver filter(s).)
    e. Maintain SOC sample at or below 4°C during storage and shipment to the laboratory.

Combined SOC/DOC sample processing:

Procedures for a combined filtering of samples for SOC and DOC analysis are listed below. Additional information can be found in Burkhardt and others (1997).

1. Collect the sample for SOC/DOC analysis as instructed in NFM 4.
2. Cover the bench or table with a sheet of aluminum foil to make a clean work surface. Put on appropriate disposable, powderless gloves. Assemble necessary equipment on the clean work surface.
    a. To remove airborne particulates, attach an in-line, 0.2-µm pore-size filter (Acrodisc 50™) to the inlet side of a dry pump hose that goes to the filter apparatus when using peristaltic or hand pumps to pressurize the apparatus.
    b. Change gloves.
    c. Remove the aluminum foil wrapping from equipment.
3. Disassemble the clean filter apparatus.
4. Using metal forceps, place a silver filter on the base of the filter apparatus between the support screen and the fluorocarbon polymer gasket, and screw the barrel onto the filter base. (There is no gasket in the fluorocarbon polymer pressure-filter apparatus.)
5. Pour a minimum of 100 mL of ASTM Type II reagent water (Burkhardt and others, 1997) or VBW or PBW into the barrel. Analysis of the water used must indicate less than 0.1 mg/L of organic carbon.

6. Screw the top part of the filter apparatus onto the barrel and attach a clean, dry hose, either from a peristaltic pump, hand pump, or compressed gas cylinder (use a clean metal hose clamp to secure the discharge hose to the inlet connector). Set the filter apparatus into a stand.
7. Apply pressure to start the flow of rinse water through the filter apparatus, using either a peristaltic pump or hand pump, or by regulating the flow of compressed gas (usually nitrogen).

Wear safety glasses when pressurizing or depressurizing the filter apparatus.

   a. The pump pressure must be regulated to less than 15 lb/in^2.
   b. If compressed gas (for example, organic-free nitrogen) is used, proceed as follows:
      i. **Make sure that the pressure regulator valve is closed.** Turn the handle on the pressure regulator counterclockwise for several turns until the pressure-regulator valve is closed.
      ii. Open the valve to the nitrogen cylinder.
      iii. To pressurize the filter apparatus, open the pressure-regulator valve by turning the handle clockwise until up to 15 lb/in^2 registers on the pressure gage.
   c. Discard rinse water.

Do not exceed 15 lb/in^2 of pressure.

8. Depressurize the filter apparatus. **Always point the apparatus away from your body, face, and other people.** When using compressed gas,
   a. Close the valve to the pressure regulator after the pressure gage shows no pressure.
   b. Close the valve to the gas cylinder.
   c. Change gloves.
9. Remove the top of the filter apparatus carefully.
10. Condition the silver filter for the SOC/DOC sample:
    a. Select the volume of wholewater (either 64 mL or 125 mL) to be filtered based on the estimated suspended-materials concentration of the sample, and record the volume on the ASR and the field forms. The volume to be filtered can be based on the table 5-6 guidelines and on previous experience of filtering samples from the site.
    b. Shake the sample vigorously to resuspend settled particles and measure the sample volume using a clean, baked 64-mL or 125-mL bottle filled to the very top. **Do not field rinse baked glass bottles.** Immediately transfer the entire volume of the sample container to the barrel of the filter apparatus.
    c. Screw the top part of the filter apparatus onto the barrel and pressurize to filter the sample. Follow the instructions in step 7 (above) for pressurizing the filter apparatus.
    d. Condition the silver filter by passing 15 to 25 mL of sample water through the filter to waste. (Pass 15 mL of sample water through the silver filter if using a 64-mL volume of sample; 15 mL is the minimum volume of sample that should be used.) Record the total volume of water that was passed through the silver filter.

Do not field rinse DOC bottle.

11. Place a 125-mL baked glass bottle under the discharge tube of the filter apparatus and collect the sample filtrate for the DOC analysis (100 mL is recommended; a minimum of 50 mL is required). If the silver filter clogs before sufficient volume for the SOC analysis can be filtered, start the process over and filter a smaller volume of water; the 64-mL bottles are useful for such conditions. **If the silver filter clogs before the entire volume of the 64-mL bottle can be filtered, this combined SOC/DOC method cannot be used. Start over and filter SOC and DOC samples separately.**

    - If the volume needed for the SOC analysis is insufficient for a DOC analysis (less than 50 mL), two or more filtrations through separate silver filters can be combined into one DOC bottle. (Retain two of the filters if a duplicate SOC analysis is planned and record the total volume of sample that passed through each of the retained filters.)
    - Each time a new silver filter is used, repeat steps 3–10, rinsing and conditioning the silver filter as described. Discard the first 15 or 25 mL of sample filtrate to waste. Reposition the DOC bottle under the discharge tube and collect the sample filtrate. Record the total volume of sample that was passed through each silver filter.
    - If the volume needed for SOC analysis is greater than the 100 mL of sample to be used for DOC analysis, remove DOC bottle after filling with 100 mL of filtrate, but continue filtering until the entire volume needed for SOC analysis has been filtered. (Record total volume filtered and discard extra filtrate.)

12. After the DOC sample has been collected and the volume for SOC analysis has been filtered, cap the DOC bottle securely and check that the bottle is labeled correctly and completely. Place the bottle in a foam sleeve before placing in an ice-filled shipping container.

13. Depressurize the filter apparatus (step 8), then disconnect the hose from the filter apparatus cylinder and remove the top. When depressurizing the compressed-gas-operated apparatus:

    a. Close the valve to the pressure regulator only after the gage indicates no pressure.

    b. Close the valve to the nitrogen cylinder.

14. Using no more than a total of 20 mL of organic grade DIW:
    - Rinse residual suspended matter from the bottle that was used to measure sample volume and pour into the filter barrel.
    - Rinse any residual suspended matter from the sides of the filter barrel.
15. Reconnect the top of the filter apparatus. Attach the pressure hose and pressurize (step 7), passing the organic-grade DIW rinse water through the silver filter. Discard rinse water to waste. Depressurize the filter apparatus (step 8).
16. Disassemble the bottom of the filter apparatus and remove the silver filter.
    a. Use a pair of metal forceps when removing the silver filter.
    b. Fold the filter in half with suspended material on the inside, taking care not to lose any suspended material. **Do not wrap the silver filter in aluminum foil.**
    c. Place the folded filter in a petri dish for SOC analysis.
    d. Close the petri dish and label dish with site identification, date and time, total filtered volume of sample, and the laboratory sample designation code. (Include the volume used to precondition the silver filter(s) in the total volume of filtrate.)
    e. Place the labeled petri dish in a sealable plastic bag.
    f. Chill DOC and SOC samples and maintain at or below 4°C without freezing (section 5.5). For SOC samples submitted to NWQL, record the total volume of filtrate on the comment line of the ASR form.
    g. If more than one silver filter was needed for the SOC sample, place each silver filter into a separate petri dish that is labeled as described in step 16d. Place all the petri dishes for a single sample into one sealable plastic bag labeled with the site identification and the date and time of sample collection. This is submitted as a single sample.
        - Package the silver filter(s) for duplicate SOC analysis separately.
        - Ship samples for SOC analysis to the laboratory with a note on the ASR form stating the number of silver filters used.

For SOC analysis, record TOTAL VOLUME of sample that passed through each silver filter.

DOC sample processing:

Procedures for filtering a DOC-only sample are listed below. Additional information can be found in Burkhardt and others (1997).

1. Collect the sample for DOC analysis (NFM 4).
2. Cover a bench or table with a sheet of aluminum foil to make a clean work surface. Put on appropriate disposable, powderless gloves. Assemble necessary equipment on the clean work surface.
   a. To remove airborne particles, attach an in-line filter, 0.2-µm pore size, (Acrodisc 50™) to a dry pump hose in front of the filter apparatus when using peristaltic or hand pumps to pressurize the apparatus.
   b. Change gloves.
   c. Remove the aluminum foil wrapping from equipment.
3. Disassemble the clean filter apparatus.
4. Using metal forceps, place a silver filter on the base of the filter apparatus between the support screen and the fluorocarbon polymer gasket, and screw the barrel onto the filter base. (There is no gasket in the fluorocarbon polymer pressure-filter apparatus.)
5. Pour a minimum of 100 mL of ASTM Type II reagent water (Burkhardt and others, 1997) or VBW or PBW into the barrel. Analysis of the water used must indicate less than 0.1 mg/L of organic carbon.
6. Screw the top part of the filter apparatus onto the barrel and attach a clean, dry hose, either from a peristaltic pump, hand pump, or compressed gas cylinder (use a clean metal hose clamp to secure the discharge hose to the inlet connector). Set the filter apparatus into a stand.

7. Apply pressure to start the flow of rinse water through the filter apparatus, using either a peristaltic pump or hand pump, or by regulating the flow of compressed gas (usually nitrogen).

Wear safety glasses when pressurizing or depressurizing the filter apparatus.

   a. The pump pressure must be regulated to less than 15 lb/in^2.
   b. If compressed gas (for example, organic-free nitrogen) is used, proceed as follows:
      i. **Make sure that the pressure regulator valve is closed.** Turn the handle on the pressure regulator counterclockwise for several turns until the pressure-regulator valve is closed.
      ii. Open the valve to the nitrogen cylinder.
      iii. Open the pressure-regulator valve by turning the handle clockwise until up to 15 lb/in^2 registers on the pressure gage. Do not exceed 15 lb/in^2 of pressure.
   c. Discard rinse water.

8. Depressurize the filter apparatus. **Always point the apparatus away from your body, face, and other people.** When using compressed gas,
   a. Close the valve to the pressure regulator after the pressure gage shows no pressure.
   b. Close the valve to the gas cylinder.
   c. Change gloves.
9. Remove the top of the filter apparatus carefully.
10. Condition the prerinsed silver filter:
   a. Open the barrel of the filter apparatus and pour about 125 mL of wholewater sample into the barrel (or about 64 mL if silver filter media is expected to clog). For water with large concentrations of suspended materials, collect the sample first into a baked glass bottle, allow suspended materials to settle, and pour 125 mL of the clear supernatant into the filter barrel.
   b. Screw the top part of the filter apparatus onto the barrel.

11. Apply pressure to start the flow of sample through the filter apparatus (step 7).
    - **Do not exceed 15 lbs/in^2.**
    - If using compressed gas, open the pressure-regulator valve first, then the valve to release gas from the cylinder (tank).
12. Condition the silver filter media by passing about 25 mL of sample through the silver filter to waste.
13. Filter the sample:
    a. Place a 125-mL organic-free amber glass bottle under the discharge tube of the filter apparatus. **Do not prerinse the DOC bottle.**
    b. If the silver filter media clogs, depressurize the filter apparatus and replace the silver filter.
        i. Rinse the new filter with blank water as described in steps 5–9.
        ii. Fill a clean DOC bottle with the water to be sampled and let the suspended materials settle before decanting the sample into the barrel of the filter apparatus.
        iii. Condition the new silver filter by passing about 25 mL of sample through the filter to waste.
        iv. Reposition the DOC bottle under the discharge tube and continue to collect the filtrate.
    c. Fill the bottle until sufficient volume for DOC analysis has been collected (50 mL is the minimum requirement; 100 mL is recommended). Cap the bottle securely and check that the bottle is labeled correctly. Place the bottle in a foam sleeve before placing in an ice-filled shipping container.
14. Depressurize the filter apparatus (step 8).
15. Chill and maintain the DOC sample at or below 4°C without freezing (section 5.5).
16. Disassemble the bottom of the filter apparatus. Remove the silver filter with metal forceps and place the filter in a plastic bag for disposal or recycling. **Do not reuse silver filters.**

Never increase the pressure in a filter apparatus to greater than 15 lb/in^2 in order to increase the rate of filtration.