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.

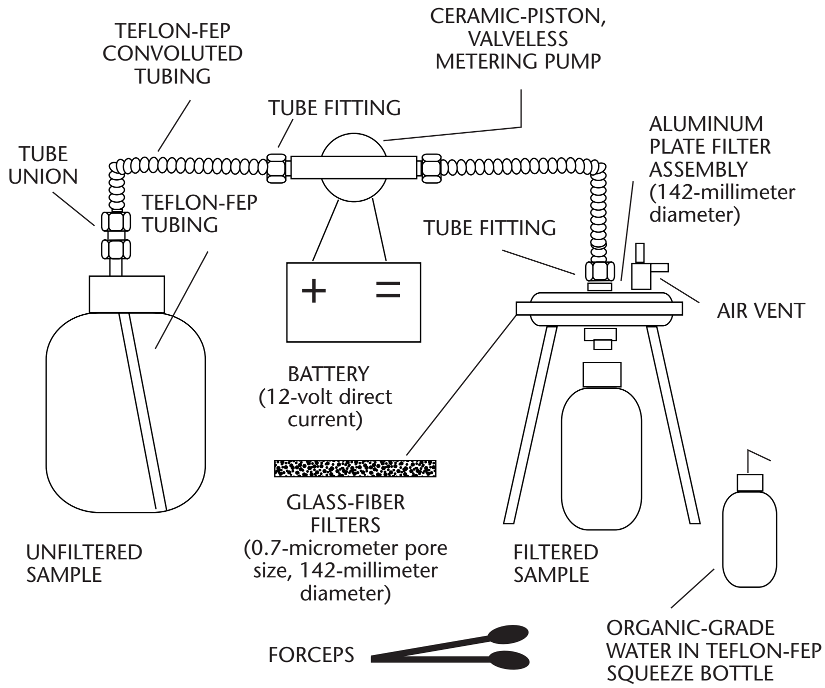

Figure 5-1. An equipment system suitable for filtering samples for analysis of organic compounds (from Sandstrom, 1995).

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.