

Procedures for Processing Samples 5.2.2.C for Carbon Analysis

Basic procedures are described below for: (1) processing the sample for analysis of total particulate carbon (TPC), particulate inorganic carbon (PIC), and particulate organic carbon (POC)¹; and (2) processing the sample for analysis of dissolved organic carbon (DOC). The specific method to be used depends on the target analyte and the choice of filter type and filtration equipment, which are to be documented on field forms and in field notes.

- ▶ **TPC (Total Particulate Carbon), PIC (Particulate Inorganic Carbon), and POC (Particulate Organic Carbon).**—Filtration of the sample requires a 25-mm glass-microfiber filter (see *UPDATE NOTE*). Particulate organic carbon (POC) is determined by subtracting the laboratory-analyzed concentrations of particulate inorganic carbon from total particulate carbon; that is, $POC = TPC - PIC$.
- ▶ **DOC (Dissolved Organic Carbon).**—Filtration of the sample requires either a disposable capsule unit with a polysulfone pleated filter or a 25-mm glass-microfiber filter (GF/F) (see *UPDATE NOTE*).

UPDATE NOTE: The NFM-5/99 version of this section (5.2.2.C) entitled "Gas-Pressurized Filter Procedures for Processing Samples for Analysis of Dissolved and Suspended Organic Carbon," which was based on a field method using silver filters, has been moved to Appendix A5-D. That method is no longer used in USGS studies as a standard procedure because of the decreasing availability of the silver filters (Office of Water Quality Technical Memorandum 2000.08).

¹POC is identified separately from the suspended organic carbon (SOC) analysis determined from organic carbon residue on a silver filter because SOC is determined by direct analysis rather than by calculation. USEPA method 440.0 is used for laboratory analysis of the TPC and PIC samples and also provides direct determination of total particulate nitrogen (TPN) concentration.

Equipment and equipment-cleaning protocols

The equipment needed to process samples for TPC and PIC analysis depends on whether the pressure-filtration method (table 5-6a) or the vacuum-filtration method (table 5-6b) will be used. The equipment options for processing samples for analysis of DOC are given in table 5-6c. (Refer to Appendix A5-D if the silver-filter method will be used to process samples for TOC, SOC, or DOC analysis.)

Equipment should be cleaned while still wet from sampling, preferably before leaving the field site. Immediately after each use, rinse the carbon-processing equipment at least three times with DIW and store in a plastic bag until it is ready to be cleaned.

- ▶ Clean the carbon-collection and carbon-processing equipment according to the standard procedures described in NFM 3.3.4.C. **Do not use methanol or any other organic solvent to clean this equipment** (see TECHNICAL NOTE).
- ▶ If it is necessary to return to the office before cleaning the equipment, be sure to field rinse the equipment onsite immediately after use and then place it into a clean plastic bag for transport.
- ▶ After the equipment has been cleaned, double-wrap all apertures and the filter apparatus with aluminum foil and store them inside a sealable plastic bag for transport to the next site.

TECHNICAL NOTE: Check the Comments/Errata page under NFM Chapters A3 and A5 periodically for an update or any changes in equipment-cleaning procedures. If a circumstance arises in which methanol-cleaned equipment must be used to collect samples for DOC analysis, it is necessary to (1) record and report the total volume of water that has passed through the equipment before the DOC sample is collected, (2) collect a field blank sample for laboratory analysis by passing organic-grade blank water through the equipment, (3) collect a source-water blank for laboratory analysis, and (4) compare the laboratory results of carbon concentrations for the environmental and quality-control (QC) samples and document the results in field notes and in any report in which the DOC data are presented.

Table 5-6a. Equipment and supplies used to process samples for analyses of total particulate carbon and particulate inorganic carbon using the pressure-filtration method

[FEP, fluorinated ethylene-propylene; mm, millimeter; μm , micrometer; $^{\circ}\text{C}$, degrees Celsius; mL, milliliter; in, inch; oz, ounce; lbs/in^2 , pounds per square inch; DOC, dissolved organic carbon]

Item	Description/Comments	Supplier or USGS One Stop Shopping Item Number ¹
FEP pressure-filtration apparatus (DOC-25)	Holds a 25-mm filter	Q444FLD
Ring stand and clamp	Holds the DOC-25 filtration unit	Open market
Filter, in-line vent, 50 mm	0.2- μm pore size; pre-filter to remove airborne particulates	Q445FLD
Peristaltic pump	Adjustable flow rate	Open market
C-Flex tubing	For use with pump	Q432FLD or Open market
Precombusted (baked) glass-microfiber filters (GF/F)	25 mm, 0.7- μm pore size, laboratory-baked at 400 $^{\circ}\text{C}$ (3 filters are required)	Q441FLD
Metal forceps, two pair	Standard metal forceps for handling filter media	Q347BACT
Glass cylinder	100-mL graduated cylinder, cleaned	Open market
Aluminum foil squares	6 in x 6 in	Q443FLD
Whirl-Pak bags	6 oz	Q22FLD
Whirl-Pak bags	18 oz	Q21FLD
Aluminum foil	Heavy duty	Open market
Cooler and ice	Standard; check with shipper for size and weight restrictions	Open market
Replacement filter-support screen	25 mm, either stainless steel or polysulfone	Pall Gelman Laboratory Part nos. 79791 or 87265
Pressure gage (optional)	Glycerin-filled, 0-30 lb/in^2 , to be inverted into side of a plastic tee that is positioned in-line between the peristaltic pump and the DOC-25 filtration unit.	Cole Parmer catalog no. P-07370-70 or equivalent
Organic grade water (if using this method for DOC processing)	Laboratory analysis of the water must certify a concentration of organic carbon that is less than the long-term laboratory reporting limit for DOC (currently <0.16 mg/L). Check the laboratory analysis for the lot number to confirm that it can be used.	N1590 (Pesticide-grade blank water) or N1580 (VOC-grade blank water)

¹The equipment that is supplied exclusively for USGS studies through the USGS internal One Stop Shopping is designated by the letters Q or N preceding a unique number. The USGS supplies and performs quality-control checks for such equipment exclusively for USGS water studies. Such equipment can be obtained for non-USGS studies on the open market or other source specified by the user. "Open market" designates equipment to be obtained from a retail or other vendor.

Table 5-6b. Equipment and supplies used to process samples for analyses of total particulate carbon and particulate inorganic carbon using the vacuum-filtration method

[mL, milliliter; mm, millimeter; μm , micrometer; °C, degrees Celsius; in, inch; oz, ounce; FEP, fluorinated ethylene-propylene]

Item	Description/Comments	Supplier or USGS One Stop Shopping Item Number ¹
Filtration flask	Polypropylene, 500 or 1,000 mL	Open market
Filter funnel	Polysulfone, 25 mm with 200-mL reservoir ²	Open market
Peristaltic pump or hand pump	Adjustable flow rate	Open market
C-Flex tubing	For use with pump	Q432FLD or Open market
Baked glass-microfiber filters (GF/F)	25 mm, 0.7- μm pore size, laboratory-baked at 400°C (3 filters are required)	Q441FLD
Metal forceps, 2 pair	Standard metal forceps for handling filter media	Q347BACT
Glass cylinder	100-mL graduated cylinder	Open market
Aluminum foil squares	6 in x 6 in	Q433FLD
Whirl-Pak bags	6 oz	Q22FLD
Whirl-Pak bags	18 oz	Q21FLD
Aluminum foil	Heavy duty	Open market
Cooler and ice	Standard: check with shipper for size and weight restrictions	Open market

¹The equipment that is supplied exclusively for USGS studies through the USGS internal One Stop Shopping is designated by the letters Q or N preceding a unique number. The USGS supplies and performs quality-control checks for such equipment exclusively for USGS water studies. Such equipment can be obtained for non-USGS studies on the open market or other source specified by the user. "Open market" designates equipment to be obtained from a retail or other vendor.

²The filter-support screen can be replaced with a stainless-steel screen like the one used in the FEP pressure-filtration apparatus. Contact Pall Gelman Laboratory, 600 Wagner Road, Ann Arbor, MI, 48103-9019; phone (734) 665-0651.

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

Table 5-6c. Equipment and supplies used to process samples for analysis of dissolved organic carbon

[μm , micrometer; GF/F, glass microfiber filter; mm, millimeter; oz, ounce; mL, milliliter; DOC, dissolved organic carbon; $^{\circ}\text{C}$, degrees Celsius; <, less than; mg/L, milligrams per liter; *N*, normal; VOC, volatile organic compound]

Item	Description and Comments	Supplier or USGS One Stop Shopping Item Number ¹
Gelman Sciences Supor capsule filter	Pleated polysulfone filter medium, in disposable polypropylene casing, 0.45- μm pore size	Q398FLD
-----or----- Precombusted (baked) glass microfiber filters (GF/F)	-----or----- 25-mm diameter, 0.7- μm nominal pore size, laboratory baked at 400 $^{\circ}\text{C}$ FEP pressure-filtration apparatus or filtration flask with funnel and associated equipment is required, as indicated in table 5-6a and table 5-6b, respectively	-----or----- Q441FLD
4-oz amber glass bottle, baked	Bottles (125 mL) supplied for DOC samples have been pre-cleaned and baked at 400 $^{\circ}\text{C}$ and quality-controlled to meet a detection limit criterion for organic carbon of <0.1 mg/L	Q28FLD
Sulfuric acid (H ₂ SO ₄) preservative	4.5 <i>N</i> -H ₂ SO ₄ , supplied in 1-mL vials	Q438FLD
Organic-grade water	Laboratory analysis of the water must certify a concentration of organic carbon that is less than the long-term laboratory reporting limit for DOC (currently <0.16 mg/L). Check the laboratory analysis for the lot number to confirm that it can be used.	N1590 (Pesticide-grade blank water) or N1580 (VOC-grade blank water)
Aluminum foil	Heavy duty	Open market
Cooler and ice	Standard; check with shipper for size and weight restrictions	Open market
Foam bottle sleeve	Individual bottles are slipped into foam sleeves to protect from breakage.	Q137FLD
¹ The equipment that is supplied exclusively for USGS studies through the USGS internal One Stop Shopping is designated by the letters Q or N preceding a unique number. The USGS supplies and performs quality-control checks for such equipment exclusively for USGS water studies. Such equipment can be obtained for non-USGS studies on the open market or other source specified by the user. "Open market" designates equipment to be obtained from a retail or other vendor.		

TPC, PIC, and POC sample processing

The sample-processing options described below involve use of either the pressure-filtration or vacuum-filtration method. The equipment and supplies needed are listed in tables 5-6a and 5-6b, respectively. Particulate analytes (TPC, PIC, POC, SOC) are reported in units of mass per volume (mg/L), and therefore **the volume of sample passed through each filter must be measured accurately and recorded on the field and the Analytical Services Request (ASR) forms.**

- D The amount of water to be filtered to obtain a sufficient quantity of material for the analysis depends on the suspended-sediment concentration and/or on the concentration of humic and other substances (such as organic and inorganic colloids that cause color in water).
- D A graph of the historical stream stage plotted against suspended-materials concentration can aid in estimating concentrations of suspended materials. Suspended-material concentrations can be used to help select the volume of sample to be filtered for a POC determination (table 5-6d).
- D Be mindful to determine and record the filtrate volume passed through each filter used for particulate analysis. **This is critical for calculation of POC concentrations.**

Table 5-6d. Guidelines for selecting the volume needed for filtration of samples for analysis of suspended and particulate 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

For TPC and PIC samples, be sure to record the total volume of water that passed through each GF/F filter.

To process samples for analysis of TPC and PIC:

- + 1. **Sampling location and collection:** Study objectives and site characteristics determine where the sample will be collected. Follow guidelines for: (1) preventing sample contamination as described in NFM 4.0, (2) using the appropriate isokinetic or nonisokinetic method as described in NFM 4.1, and (3) preparing composites and subsamples or discrete samples as described in NFM 5.0 through 5.1.1.² Avoid use of methanol-rinsed equipment.
2. Select one of the following three options (note that the actual volume of sample needed is determined by the concentration of particulates for the specific site and not by the bottle volume):
- **Discrete sample collected with a weighted bottle sampler at centroid of flow** (see NFM 4, section 4.1.1.A, VCF method)—Load the sampler with baked 125-mL DOC bottles or a 1-L baked pesticide bottle, depending on the type of sampler being used. Cap all bottles securely after they are filled with sample.
 - **Collect and composite sample processed through a cone splitter**—Using procedures described in 5.1.1.B, collect the TPC/PIC subsample from the methanol-free cone splitter into a baked, 1-L pesticide bottle or into three to four baked 125-mL DOC bottles. Cap all bottles securely.
 - **Collect and composite sample processed through a churn splitter**—Using procedures described in 5.1.1.A, collect the TPC/PIC subsample from the churn splitter into a baked, 1-L pesticide bottle or into three to four baked 125-mL DOC bottles. Cap all bottles securely.
- + TECHNICAL NOTE: An experiment to test the effect of sand in the polyethylene churn splitter on particulate carbon concentrations showed that, under most sampling conditions, the abrasion of material from the churn by sand particles will result in negligible bias in the analytical results. Caution is recommended in situations where very large concentrations of sand particles coincide with carbon concentrations that are close to the analytical minimum reporting limit (MRL).

²The guidelines described were designed for stream sampling. These procedures can be adapted for the collection of TPC, PIC, and TPN in ground-water samples, if necessary.

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ALERT! Do not field rinse the baked DOC or pesticide bottles.

3. Cover the bench or table with a sheet of aluminum foil to make a clean work surface. Put on disposable, powderless gloves. Assemble necessary equipment and supplies on the clean work surface.
 - a. Fold into thirds the aluminum foil square(s) into which the filters will be placed.
 - b. To remove airborne particulates, attach an in-line, 0.2- μm pore-size filter to the inlet side of a dry pump hose between the filtration apparatus and the peristaltic or hand pump.
 - c. Attach pump tubing to pump.
 - d. Remove the aluminum foil wrapping from the equipment.
 - e. Change gloves.
4. Prepare the filtration apparatus.
 - **Pressure-filtration method:**
 - a. Open the bottom of the DOC-25 filtration unit.
 - b. Using metal forceps, place a 25-mm, 0.7- μm pore size, GF/F onto the support screen in the base of the DOC-25 apparatus.
 - c. Push the bottom white ring that holds the filter base up against the filter unit and screw it onto the base of the filtration-apparatus barrel by screwing the blue top ring down onto the bottom white ring.
 - Finger-tighten only. Turning the bottom white ring will cause the outer edge of the filter to be cut off, making removal of the filter difficult.
 - Take care not to wrinkle or tear the GF/F.
 - d. Place the DOC-25 apparatus into the ring-stand clamp. Place a bottle or beaker under the DOC-25 filtration unit.
 - e. Shake the sample vigorously to suspend all particulate matter and immediately pour an aliquot of the sample into the barrel of the DOC-25 apparatus. While pouring, ensure that the particulates remain suspended.
 - f. Screw the top part of the DOC-25 apparatus onto the barrel and attach the peristaltic pump tubing.

- **Vacuum-filtration method:**

- a. Place the filter funnel on the filter flask.
 - b. Lift the top part of the filter funnel.
 - c. Using metal forceps, place the GF/F onto the base of the filter funnel. **Make sure the filter is not wrinkled or torn.**
 - d. Place the top part of the filter funnel back on the base.
 - e. Shake the sample vigorously to suspend all particulate matter and immediately pour an aliquot of the sample into the filter funnel. While pouring, swirl sample to ensure that the particulates remain suspended (top of filter flask can be covered with aluminum foil while swirling sample).
 - f. Attach the pump tubing from the peristaltic pump or hand pump to the vacuum flask.
5. Apply pressure (pressure filtration) or suction (vacuum filtration) to start the flow of sample water through the filtration apparatus.
- If using a peristaltic pump to pressurize the DOC-25, install a pressure gage in the line.
 - a. Do not exceed about 15 lbs. of pressure.
 - b. During pumping, a drop in pressure will signal when the last of the sample water has passed through the filter.
6. After an aliquot of sample has been filtered, tap the bottom of the filter apparatus and increase the pressure slightly to dislodge the remaining drops of sample water. When no more filtrate comes out:
- a. Depressurize the filtration apparatus carefully.
 - **Pressure-filtration method:** Remove the tubing to release the pressure and then remove the top of the DOC-25 apparatus. Check that there is no water on the filter and that the filter is covered with particulates. The particulate cake should not be extremely thick.
 - **Vacuum-filtration method:** Remove the foil cover and look into the top of the filter funnel. Check that there is water on the filter and that the filter is covered with particulates.
 - b. If the filter is dry but not covered with particulates, add another aliquot of sample by repeating steps 4e-f, 5, and 6a until the filter is loaded to capacity. **It is important that all the water in the barrel be passed through the GF/F, leaving the filter "dry."**
 - c. After the filter is dry and covered with particulates, go to step 7.

7. Pour the filtrate into a graduated cylinder and measure and record the volume on the field form and on the "Comments to NWQL" line of the ASR form. +
8. Using organic-grade water, rinse any remaining particles from the sides of the DOC-25 barrel or the sides of the filtration funnel. **Do not include the rinse water in the measured volume.**
9. Discard filtrate. **Do not send this filtrate to the laboratory for analysis of DOC.**
10. After all the organic-grade water filtrate has passed through the DOC-25 filtration unit:
 - a. Remove the DOC-25 apparatus from the ring stand.
 - b. Continue pumping, rotating the DOC-25 apparatus at a slight angle while tapping the side of the filtration unit to evacuate any remaining water droplets that are clinging to the sides of the filtration unit. This procedure moves droplets toward the center of the filter surface for final passage through the filter.
 - c. The procedure is complete when filtrate droplets are entirely evacuated and have passed through the filter-holder funnel.
11. After completing the rinse, depressurize the filtration apparatus. Change gloves. +
12. Lift the top off the filter funnel to check that the filter is dry before proceeding to carefully remove the bottom of the DOC-25 apparatus.
 - a. Open the previously folded aluminum foil square and place it on the clean work surface.
 - b. Gently remove the filter from the filter holder with metal forceps. Do not touch the filter with your fingers. Using two metal forceps:
 - i. Place the filter so that it is centered on one of the creases in the aluminum foil square; start the fold with the forceps, then press the foil down on top of the filter to complete the fold.
 - ii. Fold it in half with suspended material on the inside. Do not lose any suspended material.

Wear safety glasses when pressurizing of depressuring a filter apparatus.

- + 13. Repeat steps 4-12 two more times until a total of three filters (two for TPC and one for PIC) have been processed.
- **If the same volume of sample water was filtered through all three filters**, place them all, side by side, into one aluminum-foil envelope.
 - **If different volumes have been filtered**, use either three separate properly labeled aluminum foil envelopes or use a single packet and write the volume for each filter on the outside of the foil in which each of the filters is located.
14. Close the other flap of the aluminum foil, turning the ends up carefully.
- i. Label the aluminum foil envelope(s) with site identification, date and time, total filtered volume of sample, laboratory sample-designation code, and the laboratory schedule requested.
 - ii. Do not use tape and, if a preprinted label is used, do not let it wrap around the ends of the envelope. (The envelope will be opened and used at the laboratory when drying the filters.)
- + 15. Place labeled aluminum foil envelope(s) into small (6 oz) Whirl-Pak bag(s) and seal the bag(s).
16. Place the 6-oz Whirl-Pak bag(s) inside a large (18 oz) Whirl-Pak bag and seal the large bag.
17. Place the 18-oz Whirl-Pak package into an ice-filled cooler and maintain the samples at or below 4°C during storage and shipment to the laboratory.

**For TPC and PIC analyses, record the
TOTAL VOLUME of sample that passed
through each filter.**

DOC sample processing

The sample-processing options described below involve filtering the sample either through a GF/F and pressure-filtration apparatus (the pressure-filtration method), or a capsule filter (the capsule-filter method) (Office of Water Quality Technical Memorandum 2000.08). The pressure-filtration and capsule-filter methods are described below and the equipment needed for each method is listed in tables 5-6a and 5-6c, respectively. The silver-filter method and equipment are described in Appendix A5-D.

- ▶ Use organic-grade water when collecting an equipment blank or field blank for quality control. Organic-grade water is deionized water that has been certified by laboratory analysis of the lot to have an organic-carbon concentration that is less than the laboratory reporting limit (currently at 0.16 mg/L for DOC).
- ▶ Each equipment or field blank designated for carbon analysis should be accompanied by a source blank collected from the same lot of organic-grade water as is used for the equipment and field blanks.

TECHNICAL NOTE: A laboratory study to compare the results of the DOC analysis in same-source water that was filtered through 0.45- μm pore-size silver-filter media, the Gelman 0.45- μm SuporTM capsule filter, and 0.7- μm GF/F determined no statistically significant differences in DOC concentrations (Charles Patton and George Aiken, U.S. Geological Survey, written commun., 2001).

- ▶ USGS designations and preservation treatment for various filtered samples are listed below. The general order of preservation is to acidify all samples requiring HCl treatment first, followed by those for H₂SO₄ treatment if nutrient samples are to be acidified, and then those for HNO₃ treatment. Whole-water samples are preserved along with their filtered counterparts. The chamber cover is changed with each change in the acid treatment.
 - FAM: filtered, acidified with HCl, for mercury analysis.
 - FCC: filtered and chilled to ≤ 4 °C for nitrogen and phosphorus nutrient analysis.
 - FCA: filtered, acidified with H₂SO₄, and chilled to ≤ 4 °C for nitrogen and phosphorus nutrient analysis.
 - DOC: filtered, acidified with H₂SO₄, for dissolved organic carbon analysis.
 - FA: filtered, acidified with HNO₃, for trace-element and major-cation analysis.
 - FAR: filtered, acidified with HNO₃, for radiochemical analysis.

Capsule-filter method:

- + 1. Collect samples: Set up a clean capsule filter and sample bottle(s) within a protective chamber using the Clean Hands method (NFM 4.0.1).
- a. **Surface Water:** Follow guidelines for: (1) preventing sample contamination as described in NFM 4.0, (2) using the appropriate isokinetic or nonisokinetic method as described in NFM 4.1, (3) preparing composites and (or) subsamples or discrete samples as described in NFM 5.0 through 5.1.1, and (4) equipment selection and quality control as described in the TECHNICAL NOTE below.
- b. **Ground Water:** Follow standard guidelines for: (1) well purging (NFM 4.2), (2) sampling (NFM 4.0, 4.2, 5.0, and 5.1.2), and (3) equipment selection and quality control as described in the TECHNICAL NOTE below. Use a clean bailer that has not contacted methanol if other sampling equipment has been methanol-rinsed. Sample collection and filtration can be conducted in the same protective chamber.

+ TECHNICAL NOTE: Process the DOC sample after other filtered samples have been processed. To prevent methanol contamination of the sample, do not use methanol-rinsed collection and processing equipment, and use a new capsule filter. Collection and analysis of field-blank and source-blank samples is recommended. If methanol-rinsed equipment must be used, collection of these blanks to correlate with each DOC sample is required, regardless of the volume of water passed through the system before DOC sample collection. Taking these quality-control measures does not remove the possibility of methanol contamination of the sample, however.

- + 2. Change gloves. Place a 125-mL baked glass amber bottle under the capsule filter outlet.
- Do not field rinse the DOC bottle.
 - Do not splash sample water.
 - Pass 1 L of organic-grade water (certified for organic-carbon concentration of less than the laboratory reporting limit through the capsule filter).
 - Pass at least 1 L of sample water through the capsule filter before collecting the DOC sample.
- + 3. Fill the bottle to its shoulder.
- + 4. Cap the bottle and transfer it to the preservation chamber.

5. Change gloves. Open the DOC bottle in the preservation chamber. Add the contents of a 1-mL H₂SO₄ vial to the DOC sample.
6. Cap the DOC bottle securely. Shake the sample bottle vigorously to mix the sample and H₂SO₄.
7. Remove the DOC bottle from the preservation chamber.
8. Check that the bottle is labeled correctly and completely. Place the bottle in a foam sleeve and then into an ice-filled shipping container.
9. Maintain the sample at or below 4°C without freezing (NFM 5.5).

Pressure-filtration method:

1. Collect sample(s).
 - a. **Surface Water:** Follow guidelines for: (1) preventing sample contamination as described in NFM 4.0, (2) using the appropriate isokinetic or nonisokinetic method as described in NFM 4.1, (3) preparing composites and (or) subsamples or discrete samples as described in NFM 5.0 through 5.1.1, and (4) equipment selection and quality control as described in the TECHNICAL NOTE below.
 - b. **Ground Water:** Follow standard guidelines for: (1) well purging (NFM 4.2), (2) sampling (NFM 4.0, 5.0, and 5.1.2), and (3) equipment selection and quality control as described in the TECHNICAL NOTE below.

TECHNICAL NOTE: To prevent methanol contamination of the sample, do not use methanol-rinsed collection and processing equipment. If methanol presence is of concern, process the DOC sample either before introduction of any methanol-rinsed equipment or well after the work area has been cleared of methanol-rinsed equipment and residue of methanol fumes. Collection and analysis of field-blank and source-blank samples is recommended. If methanol-rinsed equipment must be used, collection of these blanks to correlate with each DOC sample is required, regardless of the volume of water passed through the system before DOC sample collection. Taking these quality-control measures does not remove the possibility of methanol contamination of the sample, however.

- + 2. Cover the bench or table with a sheet of aluminum foil to make a clean work surface. Assemble the necessary equipment on the clean work surface, wearing disposable powderless gloves.
- When using the DOC-25 filtration apparatus, remove airborne particulates as follows: attach an in-line, 0.2- μm pore-size filter (table 5-6a) to the inlet side of a dry pump hose or to the outlet between the peristaltic pump and the DOC-25 unit. If attaching the DOC-25 on the inlet side, be sure to dedicate a piece of tubing for the sole purpose of channeling air flow.
 - Remove the aluminum foil wrapping from the precleaned equipment.
 - Change gloves.
3. Prepare the filtration apparatus:
- Remove the bottom barrel of the filtration apparatus.
 - With metal forceps, place a clean GF/F filter onto the support screen in the base of the filtration apparatus. **Make sure that the filter medium is not wrinkled or torn.**
 - Push the bottom white ring that holds the filter base up against the filter unit and screw it onto the base of the filtration-apparatus barrel by screwing the blue top ring down onto the bottom white ring.
 - + - Finger-tighten only. Turning the bottom white ring will cause the outer edge of the filter to be cut off, making removal of the filter difficult.
 - Take care not to wrinkle or tear the GF/F.
 - Open the top of the filtration-apparatus barrel and fill with approximately 100 mL of whole-water sample.
 - **For water with high concentrations of suspended materials** (usually in surface water), collect the sample into a clean baked glass bottle, cap it securely, place it on ice, and allow the suspended materials to settle; then, pour 100 mL of the clear supernatant into the filter barrel.
 - **For surface water**, the 100-mL wholewater sample can be either a subsample collected from the churn or cone splitter or the supernatant from the bottle(s) used in the weighted-bottle sampler.
 - **For ground water**, the 100-mL wholewater sample is collected directly from the pump discharge unless turbidity is high. For turbid samples, follow the procedure described above for water with high concentrations of suspended materials.
 - Screw the top part of the filter apparatus onto the barrel and attach the pump tubing.
- + +

- f. Apply pressure, regulated to less than 15 lb/in², to start the flow of sample water through the filter apparatus.
- g. Place a 125-mL clean, baked glass bottle under the discharge tube of the filtration apparatus. **Do not field rinse the DOC bottle.**
- h. Fill the DOC bottle to the shoulder with sample filtrate.
 - If the filter clogs before 100 mL of sample for DOC analysis can be filtered, depressurize the filtration unit, empty the remaining volume of wholewater sample from the barrel, and remove the clogged GF/F filter.
 - Using clean metal forceps, replace the old filter with a new GF/F filter, following the directions from steps 3b-h above for a pressure-filtered DOC sample.
4. After the DOC sample bottle has been filled to the shoulder, cap the bottle and transfer it to the preservation chamber.
5. Depressurize and dismantle the filtration apparatus. Clean the apparatus immediately (while still wet), following the USGS procedures prescribed in NFM 3.3.4.C. If the apparatus cannot be field-cleaned immediately, it must be cleaned the same day it is used and before being reused—rinse it copiously with DIW and place it in a plastic bag so that it will not dry before being cleaned.
6. Change gloves before working in the preservation chamber.
7. In the preservation chamber, open the DOC bottle. Add the contents of a 1-mL vial containing 4.5N H₂SO₄ preservative.
8. Cap the DOC bottle securely and shake vigorously to mix the sample. Remove the DOC sample bottle from the preservation chamber.
9. Check that the bottle is labeled correctly and completely. Place the bottle into a foam sleeve and into an ice-filled shipping container (see NFM 5.5 for correct shipping procedures).
10. Maintain the sample at or below 4°C without freezing (NFM 5.5).

Wear safety glasses when pressurizing or depressurizing the filter apparatus.

11. To collect a QC sample:

- a. Clean carbon-processing equipment as prescribed in NFM 3.4.
- b. Label bottles.
- c. Change gloves. Working in a clean processing chamber, carefully pour a sample of the organic-grade water into a clean, baked, amber DOC bottle. Cap securely and pass the bottle to the preservation chamber.
- d. Process a sample of organic-grade water through the cleaned carbon-sampling and carbon-processing equipment, following the identical steps prescribed for an environmental sample (steps 2-5 above). Cap securely and pass the bottle to the preservation chamber.
- e. Follow the same steps for sample preservation, handling, and shipping as for the environmental sample (steps 6-10).

Never increase the pressure in a filter apparatus to greater than 15 lb/in² in order to increase the rate of filtration.