
WASTEWATER, PHARMACEUTICAL, AND ANTIBIOTIC COMPOUNDS 5.6.1.F

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The USGS differentiates between samples collected for analysis of wastewater compounds and those collected for analysis of pharmaceutical and antibiotic compounds, based on the analytical schedule for the laboratory method.¹ Currently, only the wastewater laboratory method for field-filtered samples (SH1433) is an approved, routine (production) method. (The unfiltered wastewater method LC 8033 also is available but requires a proposal for custom analysis.) At this time, analysis of samples for pharmaceutical and antibiotic compounds is confined to research studies and is available only on a custom basis.

To collect and process surface-water and ground-water samples that will be analyzed for concentrations of wastewater, pharmaceutical, and antibiotic compounds, the standard USGS procedures for collecting and filtering organic compounds are used (see NFM 4, 5.1, 5.2.2). However, special considerations related to personal safety and to sample contamination are required.

Samples collected for analysis of these compounds may be collected directly from sources of raw or treated wastewater. Sources of wastewater include treated and untreated domestic sewage, leaking septic systems or sewer lines, sanitary sewer overflows, and runoff from animal feeding operations. **Handling of such samples can expose personnel to pathogenic microorganisms, and therefore requires strict adherence to safety protocols.**

¹The compounds included in a given laboratory schedule are subject to change and should be checked when planning a sampling activity that involves collecting data on wastewater, pharmaceutical, or antibiotic compounds.

Samples collected for analysis of wastewater, pharmaceutical, and antibiotic compounds are susceptible to contamination because they are ubiquitous in daily use. **To ensure sample integrity, field personnel should avoid contact with or consumption of products that contain the compounds targeted for analysis, and must adhere scrupulously to equipment-cleaning, equipment-handling, sample-processing, and sample-handling protocols.**

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CAUTION: Raw or treated wastewater samples can contain microorganisms harmful to human health—use safe handling protocols.

► **Adhere to safety protocols.** The following precautions must be followed by field personnel when collecting, processing, or handling raw or treated wastewater samples for analysis of wastewater, pharmaceutical, or antibiotic compounds:

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- Be familiar with the basic procedures to minimize exposure to and effects from contaminated water, as described in NFM 9.7.
- Receive proper immunizations before engaging in field activities and consult with your safety officer on this issue.
- Avoid direct contact with sewage and other types of wastewater and with equipment still contaminated through contact with the sample or source water.
- Avoid breathing in sewage and wastewater fumes or mist.
- Do not use workspace surfaces or equipment that have come into contact with polluted water until they have been decontaminated. Use only those decontamination procedures that are described below under “Prevent sample contamination.”

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► **Prevent sample contamination:**

- + — On the day of sampling activities, avoid contact with or consumption of the products listed below. Where contact with or consumption of these products is unavoidable, the collection of field blanks is strongly recommended.

Wastewater compounds

- Soaps and detergents, including antibacterial cleansers
- DEET (active ingredient in most insect repellents)
- Fragrances (cologne, aftershave, perfume)
- Sunscreen
- Animal or human urine or excrement
- Caffeine (coffee, tea, colas)
- Tobacco

Pharmaceutical compounds

- + —
- Prescription drugs, medications, and hormonal substances
 - Over-the-counter medications
 - Selected human antibiotics

Antibiotics

- Human antibiotics
 - Veterinary antibiotics
- + — Wear powderless nitrile laboratory gloves during sampling and processing. Change to clean gloves with each change in activity or potential glove contamination.
- Avoid breathing directly over open samples/equipment.
- + — Avoid direct contact between yourself (including clothing) and the sample, sampling device, and processing equipment. Clothing is a source of detergents, fragrances, and fire retardants.

- Thoroughly field rinse and seal in a plastic bag all reusable equipment that comes in contact with sewage until the equipment can be properly decontaminated and disinfected (NFM 3.2.2). +
- Clean scrupulously all workspace surfaces that come into contact with sewage—use a non-antibacterial soap² and water, followed by wiping all potentially contaminated surfaces with a clean, disposable isopropyl alcohol (70-90 percent) pad.
- Avoid any actions at the field site that result in the disposal or release of wastewater and pharmaceutical substances.

► **Implement quality control.** Quality-control samples are a required, integral part of water-quality investigations. As previously noted, samples for analysis of wastewater, pharmaceutical, and antibiotic compounds are vulnerable to contamination.

- Check your quality-control plan for instructions on the collection of field blanks and replicates for these sample types. Although the specific type, number, and distribution of quality-control samples are determined by the design and data-quality requirements of the study (NFM 4.3), field blanks are processed more frequently for these samples than for most other sample types. +
- When using a custom analysis, consult with the laboratory analysts for quality-control recommendations.
- Process an initial field blank to evaluate the potential for contamination associated with the field methods, materials used, and sampling environment. Distribute subsequent field blanks areally and temporally to meet data-quality requirements of the project.
- Use either pesticide- or VOC-grade blank water as the source solution for the field blanks. +

²The laboratory analysis of wastewater includes triclosan, an active ingredient in most antibacterial soaps. Triclosan is also commonly found in some deodorants, toothpastes, mouthwashes, skin creams, lotions, laundry detergents, and dish soaps.

- + — The analytical methods used for pharmaceuticals and antibiotics are currently unapproved and are available only on a custom basis for research projects. It is reasonable to assume that blank water tested and certified contaminant free for approved organic constituent methods is an appropriate source solution for field blanks for these unapproved methods; however, a more rigorous approach would be to include a source solution blank as an integral part of the project quality-control plan.
- Process the field blanks in the same manner and under the same environmental conditions as the environmental sample (NFM 4.3.1.B).

Wastewater, pharmaceutical, and antibiotic samples are vulnerable to contamination and thus require rigorous quality control, usually by processing field and source-solution blanks.

- + **► When planning sampling activities that will result in a request for a custom analysis:**
 - Contact the NWQL in Lakewood, Colorado, (<http://nwql.usgs.gov/>) before collecting any samples for unfiltered wastewater-compound analysis or pharmaceutical-compound analysis.
 - Check One-Stop Shopping for supplies for USGS projects.
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 - Contact the Organic Geochemistry Laboratory in Lawrence, Kansas, (<http://ks.water.usgs.gov/Kansas/reslab/>) before collecting any samples for antibiotics that will be analyzed using this laboratory's methods.

To collect and process samples:

1. Order and assemble all equipment. Select sampling and processing equipment made of fluorocarbon polymers, glass, aluminum, or stainless steel. Avoid equipment made of Tygon[®], polyethylene, or other plastics. +
2. Clean equipment thoroughly before use, following the general protocols for organic-compound samples described in NFM 3.2.2, but with the following caveats:
 - Use non-antibacterial detergents.
 - Take extra care to ensure that equipment is copiously rinsed with deionized water (DIW) after the detergent wash—detergents are a source of interference in the analysis of pharmaceutical compounds and may include a target analyte (triclosan) of the wastewater analytical method.
 - Follow the DIW rinse with a methanol rinse. Collect the used methanol solution into an appropriate container for disposal. +
 - Do not clean or field-rinse the baked-glass sample bottles obtained from OWQRL or another laboratory.
3. Collect and process the samples using methods appropriate for organic compounds, as described in NFM 4 and 5.2.2. Use laboratory-baked, brown (amber) glass sample bottles.
 - For wastewater and pharmaceuticals being shipped to the NWQL for analysis, use 1-L GCC bottles.
 - For antibiotic samples being shipped to the OWQRL for analysis, use 1-L GCC bottles.
 - For antibiotic samples being shipped to the Lawrence Organic Geochemistry Laboratory for analysis, use two 125-mL baked-glass bottles with Teflon[®] caps per sample. +

- + 4. Label bottles with site ID (identifier), date, time, sample type (“filtered” or “unfiltered”), and laboratory code or schedule number.
- a. For samples being shipped to the NWQL:
- Wastewater, field-filtered – Label sample bottle “GCC--SH 1433”
 - Wastewater, raw – Label sample bottle “GCC-LC 8033” (add this laboratory code to the Analytical Services Request (ASR) form: **currently this is a custom analysis**)
 - Pharmaceuticals – Label sample bottle “LC9003”
- b. For antibiotic-analysis samples being shipped to the OWQRL – Label sample bottle “Antibiotics”
- c. For antibiotic-analysis samples being shipped to Lawrence, Kansas – Label sample bottles “LC-AN”
- + 5. If collecting wholewater samples for custom wastewater analysis – Fill the 1-L GCC bottle to the shoulder.
6. When collecting filtered samples for wastewater, pharmaceutical, or antibiotic analyses – Filter the samples at the field site.
- Pass samples through a 0.7- μm nominal pore-size glass microfiber plate filter (GF/F grade), following the procedures for organic compounds described in NFM 5.2.2.A.
 - Fill the 1-L GCC or two 125-mL baked-glass bottles to the shoulder.
7. For samples collected from polluted water, decontaminate the exterior of bottles:
- a. Rinse bottles with copious amounts of water.
- b. Wipe each bottle with a clean, disposable isopropyl alcohol (70-90 percent) pad.
- + c. Rinse off each bottle with water.

8—PROCESSING OF WATER SAMPLES

8. Keep the samples chilled to 4°C or less until they are prepared for shipping. +
9. After sample collection and processing, use a non-antibacterial soap and water to thoroughly clean any workspace surfaces that have come into contact with polluted water. Follow the cleaning by decontaminating the workspace with isopropyl alcohol. **Do not use a bleach solution for decontamination of surfaces that come in contact with samples, since any bleach residue will degrade target analytes.**

To ship the sample(s):

1. For raw or treated wastewater, use the following precautions when packing and shipping, as these samples can pose a health hazard to field and laboratory personnel.
 - a. Decontaminate any sample bottles containing raw or treated wastewater as described in step 7 above.
 - b. Check bottle cap to ensure a tight closure. +
 - c. Place each bottle inside a foam sleeve and then place bottles into a ziplock bag along with four 3M high-capacity chemical sorbent pads (pads are available from Lab Safety and Supply at www.labsafety.com). Seal the bag.
 - d. Place the sealed bag inside two additional ziplock bags (a total of three bags) and seal each bag.
 - e. Clearly note in the "Login Comments" section of the ASR form "SAMPLE CONTAINS RAW OR TREATED SEWAGE. HANDLE WITH CARE." Sampling staff or shipping staff should notify the laboratory login staff by telephone or e-mail of the expected delivery of these samples, in advance of their arrival at the lab. +

2. For all other samples,
 - a. Check that sample bottles are securely capped.
 - b. Check that sample bottles are correctly labeled and match the information shown on the ASR form.
 - c. Pack all samples in ice to maintain a temperature of or below 4°C without freezing, using standard procedures (NFM 5.5.3 - 5.5.4). Check that the ASR form has been properly sealed in doubled plastic bags and included with the samples.
3. Ship all samples to the laboratory via overnight delivery.

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