GREENWAY ENGINEERING

Sample Containers, Preservation and Holding Times

H2SO4 – Sulfuric Acid (conc.) HNO3 – Nitric Acid (conc.) NaOH – Sodium Hydroxide Zinc Acetate

Parameter/Analysis	Preservative	Sample Holding Time	Minimum Volume	Type of Container	Comments
Alkalinity & Acidity	< 4ºC	24 hours recommend 14 days regulatory (6 hours if Biological Activity is present)	250 mL	Polyethylene or Borosilicate glass	Fill container completely and cap tightly. Avoid sample agitation and prolonged exposure to air. G
Ammonia-Nitrogen	<4ºC + H_2SO_4 pH<2 DECHLOR	7 days recommend 28 days regulatory	500 mL	Plastic or glass	G or C
Biochemical Oxygen Demand and CBOD5	Analyze 2 hours or cool to <4ºC.	6 hours recommend 48 hours regulatory	1000 mL	Plastic or glass	G o C
Boron	HNO3 to pH <2	28 days recommended 6 months regulatory	250 mL	Plastic (PTFE) or Quartz	G or C
Bromide	None required	28 days	125 mL	Plastic or glass	G or C
Chemical Oxygen Demand	H_2SO_4 pH<2 + <4ºC	7 days recommended 28 days regulatory	100 mL	Plastic or glass	Analyze ASAP G or C
Chloride	< 4ºC	28 days	125 mL	Plastic or glass	G or C
Chlorine	None, analyze immediately	15 minutes / In situ	500 mL	Plastic or glass	G
Chromium VI	<4ºC	24 hours	1000 mL	Plastic or glass (acid-washed with 1:1 HNO3)	G
Color	<4ºC	48 hours	500 mL	Plastic or glass	Warm samples to room temperature before measurement G or C
Copper by colorimetry	<4ºC	24 hours	250 mL	Plastic or glass (acid-washed with 1:1 HNO3)	G or C
Conductivity	<4ºC	28 days	500 mL	Plastic or glass	G or C
Cyanide, Amenable	0.6 g Ascorbic acid if chlorine present and refrigerate	14 days regulatory 24 hours if sulfide present	125 mL	Plastic or Glass	G or C
Cyanide, Total	NaOH to pH > 12, refrigerate in dark	24 hours recommend 14 days regulatory 24 hours if sulfide present	125 mL	Plastic or Glass	G or C
Dissolved Oxygen	None, analyze immediately	15 min/ In situ	300 mL	BOD bottle	G
Fecal Coliform & E. coli	<4ºC (1 hour) + 10% $Na_2S_2O_3$ (0.1 mL)	6 hours and 2 hours to process	100 mL	Sterile - Non reactive borosilicate glass or plastic	Leave air space G
Fluoride	None required	28 days	125 mL	Plastic	G or C
Hardness	Add HNO_3 or H_2SO_4 to pH <2	6 months	100 mL	Plastic or glass	G or C
Iodine	None, analyze immediately	15 min / In-situ	500 mL	Plastic or glass	G
Iron	Conc. nitric acid (2 mL/L) to pH <2	6 months at room temperature	250 mL	Plastic or glass (acid-washed with 1:1 HNO3)	G or C
Lead	Conc. nitric acid (2 mL/L) to pH <2	6 months at room temperature	250 mL	Plastic or glass acid-washed 1:1 HNO3	G or C
MBAs	<4ºC	48 hours	250 mL	Plastic or glass	G or C
Mercury (Hg)	Add HNO3 to pH <2 + <4ºC	28 days	250 mL	Plastic or glass (acid-washed with 1:1 HNO3)	G or C
Metals, general (except Hg)	HNO3 to pH <2 For dissolved metals, filter immediately.	6 months at room temperature	250 mL	Plastic or glass acid-washed with 1:1 HNO3	G or C
Nitrate (chlorinated)	<4ºC	28 days	250/500	Plastic or glass	G or C

Parameter/Analysis	Preservative	Hold Time	Volume	Type of Container	Comments
Nitrate (non chlorinated)	<4ºC, non-acidified	48 hours	250 or 500 mL	Plastic or glass	Analyze ASAP G or C
Nitrite	<4ºC	48 hours	125 mL	Plastic or glass	Analyze ASAP, Store only if necessary G or C
Nitrate + Nitrite	<4ºC + H_2SO_4 pH<2	1-2 days recommend 28 days regulatory	250 mL	Plastic or glass	G or C
Odor	<4ºC	6 hours	500 mL	Glass	Analyze ASAP G
Oil and Grease	<4ºC + H_2SO_4/HCl (10 mL 6N HCl)pH<2	28 days	1000 mL	Glass, wide mouth, calibrated	G
Orthophosphate (always reported "as P")	Filter, <4ºC	48 hours	125 mL or 250 mL	Glass (acid-washed with 1: 1 HCl)	Warm samples to 15-25ºC before analysis. For dissolved phosp., filter immediately G
pH	None	15 minutes/in-situ	50 mL	Plastic or glass	Analyze immediately G
Pesticides	<4ºC, add 1000 mg ascorbic acid/L if TRC present	7 days until extraction	2000 mL	Glass (solvent-cleaned), PTFE-lined cap	G or C
Phenols	<4ºC + H_2SO_4 pH<2	28 days until extraction		Plastic or glass, PTFE-lined cap	G or C
Semivolatile Organic Compounds (SVOCs/SVOAs)	<4ºC	7 days until extraction	2000 mL	Amber glass (solvent-rinsed/baked)	G or C
Solids (TS)	<4ºC	7 days	250 mL	Plastic or glass	G or C
Sulfate	<4ºC	28 days	125 mL	Plastic or glass	G or C
Sulfide	4 drops 2N Zn Accetate/100 mL, NaOH to pH>9. Cool to <4ºC	7 days	125 mL	Plastic or glass	G or C
Temperature	None	Immersion Stab.	1 L	Plastic or glass	Analyze immediately
Total Dissolved Solids	<4ºC	5 days from receipt at laboratory	250 mL	Plastic or glass	G or C
Total Kjeldahl Nitrogen (TKN)	<4ºC + H_2SO_4 pH<2 DECHLOR	7 days recommended 28 days regulatory	125 mL	Plastic or glass	G or C
Total Organic Carbon (TOC)	Analyze immediately; or refrigerate and add HCl, H3PO4, or H2SO4 to pH<2 + <4ºC	7 days recommended 28 days regulatory	100 mL	Glass (borosilicate)	G or C
Total Phosphorus (always reported "as P")	4ºC + H_2SO_4 pH<2	28 days	125 mL	Plastic or glass (acid-washed with 1:1 HCl)	Warm samples to 15-25ºC and neutralize with 5.0 N NaOH before analysis if acid was added. Correct for volume additions. G or C
Total Suspended Solids (TSS)	<4ºC	7 days	250-1000 mL	Plastic or glass	G or C
Turbidity	<4ºC	24 h. recommended 48 h. regulatory	100 mL	Plastic or glass	Analyze same day, store in dark up to 24 hours G or C
Volatile Organic Compounds (VOCs)	Add HCl to pH<2 + <4ºC.	7 days recommended 14 days regulatory	3 x 40 mL vials	Glass, PTFE-lined cap	Add 1000 mg ascorbic acid/L if TRC present. G

*Information taken from Standard Methods 20th Edition: p. 1-33: Table 1060:I. *Summary of Special Sampling and Handling Requirements.*
Sample volumes have been adapted to comply with Greenway Engineering Environmental Laboratory specific sample volume protocols.

Collection and Preservation of Samples

Fill sample containers without pre-rinsing with sample; prerinsing results in loss of any pre-added preservative and sometimes can bias results high when certain components adhere to the sides of the container. **Depending on the determinations to be performed, fill the container full (ORGANIC ANALYSES) or leave room for mixing (MICROBIOLOGICAL AND INORGANIC ANALYSES).** Except when sampling for analysis of volatile organic compounds, leave an air space equivalent to approx. 1% of the container volume to allow for thermal expansion during shipment.

Zero head-space is important in preservation of samples with volatile organic compounds and radon or other gases. Avoid loss of volatile materials by collecting sample in a completely filled container (there should be no "air bubbles" when turning the container upside down). Achieve this by carefully filling the bottle so that the top of the meniscus is above the top of the bottle rim. **It is important to avoid spillage or air entrapment if preservatives such as HCl or ascorbic acid have already been added to the bottle.** After capping or sealing bottle, check for air bubbles by inverting and gently tapping it; if one or more air bubbles are observed, then, if practical, discard and repeat refilling bottle with new sample until no air bubbles are observed (this cannot be done if bottle contained preservatives before it was filled).

"It is an old axiom that the result of any testing method can be no better than the sample on which it was performed." The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled. This objective implies that the relative proportions or concentrations of all pertinent components will be the same in the samples as in the material being sampled, and that the sample will be handled in such a way that no significant changes in composition occur before the tests are made."

Sample carefully to ensure that analytical results represent the actual sample composition. Important factors affecting results are the presence of suspended matter or turbidity, the method chosen for removing a sample from its container, and the physical and chemical changes brought about by storage or aeration). **Sample splitting and sub-sampling must be performed carefully. The sample must be homogenous prior to splitting a sample or prior to removing an aliquot of sample – this can be achieved by thorough shaking and mixing of the sample prior to splitting or sub-sampling. Sufficient headspace (at least 1'') should be present in microbiological and inorganic containers to facilitate mixing.** Samples with volatile compounds should have no headspace.

To minimize the potential for volatilization or biodegradation between sampling and analysis, keep samples as cool as possible without freezing. Preferably pack samples in crushed or cubed ice or commercial ice substitutes before shipment. **(SEE "TIPS FOR PACKING YOUR COOLER" FOR MORE DETAILS).**

Reference: Clesceri, Lenore S, et al. Standard Methods for the Examination of Water and Wastewater, 20th edition. Washington, DC: American Public Health Association (1998).