

Sample Containers, Preservation Techniques, and Holding Times For Aqueous Matrices

Bacteriological Analyses

<u>Determination</u>	<u>Method</u>	<u>Container/ Volume (mL)</u>	<u>Preservative</u>	<u>Holding Time¹</u>
Coliform, Total aqueous/30hrsDW	SM9221B,SM9223	P,G/Sterile/100	recom.<10°C ¹²	8hrs nonDW
Coliform, Fecal aqueous/30hrsDW	SM9221E,SM9223	P,G/Sterile/100	recom.<10°C ¹²	8hrs nonDW
Enterococcus aqueous/30hrsDW	SM9230B	P,G/Sterile/100	recom.<10°C ¹²	8hrs nonDW
aqueous/30hrsDW	ASTM D650399	P,G/Sterile/100	recom.<10°C ¹²	8hrs nonDW
Heterotrophic Plate Ct. aqueous/30hrsDW	SM9215B	P,G/Sterile/100	recom.<10°C ¹²	8hrs nonDW
Streptococcus, Fecal aqueous/30hrsDW	SM9230B	P,G/Sterile/100	recom.<10°C ¹²	8hrs nonDW

Bacteriological Analyses-Food and Bottled Beverages

<u>Determination</u>	<u>Method</u>	<u>Container/ Volume (mL)</u> ***	<u>Preservative</u>	<u>Holding Time¹</u>
Yeast & Mold	AOAC 997.02	P,G/Sterile/100	None	NA
HPC/APC	AOAC 966.23/	P,G/Sterile/100	None	30hrsDW/NA
others	AOAC 990.12			
Acidophiles	CMMEF, 4 th Ed.	P,G/Sterile/100	None	NA
E. Coli	BAM-FDA 8 th Ed.	P,G/Sterile/100	Sodium Thiosulfate	30hrsDW/NA
other				
Coliform, Total	AOAC 966.24	P/G/Sterile/100	Sodium Thiosulfate	30hrsDW/NA
other				
Listeria	AOAC 999.06	P/G/Sterile/ 25grams	None	NA
Salmonella	AOAC 996.08	P/G/Sterile/ 25grams	None	NA
E. Coli 0157:H7	AOAC cert #010504	P/G/Sterile/ 25grams	None	NA

Inorganic Wet Chemistry Analyses

<u>Determination</u>	<u>Method</u>	<u>Container/ Volume (mL)</u>	<u>Preservative</u>	<u>Holding Time¹</u>
Alkalinity*	SM2320B	P,G/500	≤6°C	14 days
Ammonia	SM4500NH3G	P,G/100	≤6°C H ₂ S ₀ ₄	28 days
Asbestos	100.2	P/1000	≤6°C	48 hours ¹³
BOD*	SM5210B	P,G/1000	≤6°C	48 hours
Boron	200.7	P/500	HN ₀ ₃ ⁸	6 months
Bromate	300.1	P,G/100	EDA ¹⁷	28 days
Bromide*	300.1	P,G/100	None	28 days

Inorganic And Wet Chemistry Analyses continued

<u>Determination</u>	<u>Method</u>	<u>Container/ Volume (mL)</u>	<u>Preservative</u>	<u>Holding Time¹</u>
Cations(Ca,Mg,Na,K)	200.7	P,G/500	HNO ₃ ⁸	6 months
COD	SM5220D	P,G/100	≤6°C, H ₂ SO ₄	28 days
Chloride*	300.0	P,G/100	None	28 days
Chlorine demand	SM2350B	P,G/1000	None	15 minutes
Chlorine dioxide*	SM4500ClO ₂ D	P,G,/100	None	15 minutes
Chlorine, residual*	SM4500ClG	P,G/100	None	15 minutes
Chlorate	300.1	P,G/100	EDA ^{17, 22}	28 days
Chlorite	300.1	P,G/100	≤6°C, EDA ¹⁷	14 days
Chromium-Hexavalent	SM3500CrD	P,G/100	≤6°C, NH ₄ Buffer ¹⁵	DW/WW 28 days
Chromium-Hex.(low level)	7199	P,G/100	≤6°C	24 hours
	218.6 218.7	P,G/100	≤6°C ²² , Na ₂ CO ₃ , NaHCO ₃ & (NH ₄) ₂ SO ₄ ¹⁸ OR (NH ₄) ₂ SO ₄ & NH ₄ OH	14 days
Color*	SM2120B	P,G/100	≤6°C	48 hours
Cyanide	SM4500CN C E G	P,G/250	≤6°C NaOH ¹⁴	14 days
Dissolved Oxygen	SM4500 O C	G/300	Fixed on site	8 hours
Flashpoint	1010	G/500	None	Not Specified
Fluoride*	SM4500 FC	P/100	None	28 days
Hardness (Total)	200.7	P,G/500	HNO ₃ ⁸	6 months
Metals ICP (incl. Cations)	200.7,6010B	P,G/500	HNO ₃ ⁸	6 months
Metals ICPMS	200.8,6020	P,G/500	HNO ₃ ⁸	6 months
Copper/Lead Rule	200.8	P,G/1000	None ¹⁶	6 months
Mercury	7470,7471,200.8	P,G/500	HNO ₃ ⁸	28 days
	SM3112B	P,G/500	HNO ₃ ⁸	28 days
Nitrate*	300.0	P,G/100	≤6°C	48 hours
Nitrite*	SM4500NO ₂ B	P,G/100	≤6°C	48 hours
Nitrogen–Total Kjeldahl	351.2	P,G/500	≤6°C, H ₂ SO ₄	28 days
Odor	SM2150B	P,G/100	≤6°C	48 hours
Oil & Grease	1664	G-A/500 ¹⁰	≤6°C, H ₂ SO ₄	28 days
PCBSA*	300.0	P,G/100	None	28 days
Perchlorate	314	P,G/100 ²³	≤6°C	28 days
Perchlorate (low level)	332.0/6860	P,G/100sterile or P,G/100 ²³	≤6°C, filter with syringe ≤6°C	28 days 28 days
pH*	SM4500H+B	P,G/100	None	15 minutes
Phenols	420.4	G-A/250	≤6°C ⁶ , H ₂ SO ₄	28 days
Phenols (low level)	420.4	G-A/1000	≤6°C, H ₂ SO ₄	28 days
Phosphates – Ortho*	SM4500P E	P,G/100	≤6°C	48 hours
Phosphorus, Total (as P)	SM4500P E	P,G/100	≤6°C, H ₂ SO ₄	28 days
Silica, Reactive*	SM4500 SiO ₂ C	P/500	≤6°C	28 days
Silica Total	200.7	P/500	HNO ₃ ⁸	6 months
Solids-Dissolved-TDS*	SM2540C	P,G/500	≤6°C	7 days
Solids-Suspended-TSS*	SM2540D	P,G/500	≤6°C	7 days
Solids-Total*	SM2540B	P,G/500	≤6°C	7 days
Solids-Settleable *	SM2540F	P,G/2000	≤6°C	48 hours
Solids-Volatile*	160.4	P,G/500	≤6°C	7 days
Specific Conductance-EC*	SM2510B	P,G/100	≤6°C	28 days
Sulfate*	300.0	P,G/100	≤6°C	28 days

Sulfide, dissolved	SM4500S D	P,G/100 ⁹	≤6°C zero headspace	15 min/7 flocc ZnAc
Sulfide, total	SM4500S D	P,G/100	≤6°C NaOH,ZnAcetate	7 days
Surfactants (MBAS)*	SM5540C	P,G/500	≤6°C	48 hours
Turbidity*	SM2130B	P,G/100	≤6°C	48 hours
Uranium	200.8	P,G/500	HNO ₃ ⁸	6 months
UV-254	SM ^{20th} 5910B	G-TLC-A/250	≤6°C	2 days
Volatile Acids	SM5560C	P,G/500	<6°C	7 days

Radiochemistry Analyses

<u>Determination</u>	<u>Method</u>	<u>Container/ Volume (mL)</u>	<u>Preservative</u>	<u>Holding Time¹</u>
Gross Alpha	900.0,9310	P,G/1000	HNO ₃ ⁵	6 months
Gross Beta	900.0,9310	P,G/1000	HNO ₃ ⁵	6 months
Uranium	908.0	P,G/1000	HNO ₃ ⁵	6 months
Radium 226	903.1	P,G/1000	HNO ₃ ⁵	6 months
Radium 228	904.0,9320	P,G/2000	HNO ₃ ⁵	6 months
Radon	913	G-TLC-A /2 x 250 ¹¹	≤6°C	4 days
Strontium 90	905.0	P,G/1000	HNO ₃ ⁵	6 months
Tritium	906.0	G/1000	None	6 months

Organic Analyses

Determination	Method	Container/ Volume (mL)	Preservative	Holding Time¹ Extraction/Analysis
Semivolatiles, N.P.Pest.	525	G-TLC-A/1000	≤6°C ³ , HCl	14/30 days
Base/Neutrals/Acid	625	G-TLC-A/1000	≤6°C ³	7/40 days
	8270	G-TLC-A/1000	≤6°C ³	7/40 days
Carbamates	531.2	VOA-G-A/3 x 40 vials	≤6°C, Na ₂ S ₂ O ₃ , PDC ⁴	28 days
Chlorinated pests/PCB's	505	G-TLC-A/2 X 40 vial	≤6°C ³ , Na ₂ S ₂ O ₃	7/1 days ⁷
Chlorinated pests/PCB's	508	G-TLC-A/1000	≤6°C ³	7/14 days ⁷
Chlorinated pesticides	608,8081	G-TLC-A/1000	≤6°C ³	7/40 days ⁷
Polychlorinated Biphenyls	608	G-TLC-A/1000	≤6°C	7/40 days
	8082	G-TLC-A/1000	≤6°C	7/40 days
Chlorinated Herbicides	515.3	G-TLC-A/1000	≤6°C ³	14/14 days
	8151	G-TLC-A/1000	≤6°C ³	7/40 days
Diesel Range Organics	8015B	VOA-G/4 x 40 vials, TB ²	≤6°C, HCl or H ₂ SO ₄	14 days recom.
	522	G-TLC-A/100	≤6°C ²² , Na ₂ SO ₃ /NaHSO ₄ ¹⁸	28/28 days
1,4-Dioxane	522	G-TLC-A/100	≤6°C ²² , Na ₂ SO ₃ /NaHSO ₄ ¹⁸	28/28 days
Dioxins	1613B	G-A/1000	≤6°C ³	30 days
Diquat	549.1	P/1000, A	≤6°C ³ , H ₂ SO ₄ ²⁴	7 days for ext ¹³
EDB and DBCP	504,8011	VOA-G-A/3x40vials, TB/2	≤6°C, Na ₂ S ₂ O ₃	14 days
Endothall	548.1	G-A/500	≤6°C ³	7/14 days
Ethylene Glycol	GCFID/MS(8015-Mod)	G-TLC-A/1000	≤6°C	40 days
Gasoline Range Orgs.	8015B	VOA-G/4 x 40 vials	≤6°C, HCl	14 days recom.
Glyphosate	547	VOA-G/3 x 40 vials	≤6°C, Na ₂ S ₂ O ₃	14 days ⁷
Haloacetic Acids	SM6251B	VOA-G/4 x 40 vials, TB	≤6°C, NH ₄ Cl	9/21 days
Haloacetic Acids	552.3	VOA-G/4 x 60 vials, TB	≤6°C, NH ₄ Cl	14/28 days
Hormones	539	G-TLC-A/1000, TB	≤6°C ²² , Na ₂ S ₂ O ₃ & 28/28 days	
			2-mercaptopyridine-1-oxide, Na ²¹	
Organophos. Pests.	8270C	G-TLC-A/1000	≤6°C ³	7/40 days ⁷
Perfluorinated Compounds	537	PP/ 250, TB	≤6°C ²² , Trizma ²⁰	14/28 days
Total Organic Carbon	SM5310B	P,G/4 x 40 vials	≤6°C, H ₂ SO ₄	28 days
Total Organic Halogen	SM ^{20th} 5320B	G-TLC-A/250	≤6°C ³ , H ₂ SO ₄	28 days
TPH	418.1	G-TLC-A/1000	≤6°C, H ₂ SO ₄	28 days
Trihalomethanes	524.2	VOA-G-A/4 x 40 vials, TB	≤6°C, Na ₂ S ₂ O ₃	14 days
			(or HCL + ascorbic acid)	
Formation Potential	524.2	VOA-G-A/4 x 40 vials, TB	≤6°C	10 days
Maximum Potential	524.2	VOA-G-A/4 x 40 vials, TB	≤6°C	7 days
Volatile Organics	524.2, 624, 8260	VOA-G/4 x 40 vials, TB ²	≤6°C, HCl ³	14 days
			≤6°C ²² , Maleic, ascorbic acid ¹⁹	14 days

Notes:

G=Glass, P=Polyethylene (plastic), PP= Polypropylene G-A=Amber Glass, VOA=Vial with Teflon-lined septum – zero head space, G-TLC-A=Amber Glass with Teflon-lined cap, Recom.=recommended, NA=Not Applicable, TB=Travel Blank, °C = degrees, floc = flocculate, EDA = Ethylenediamine DW = drinking water, GW = groundwater, SW = storm water, WW = wastewater

SM refers to Standard Methods for the Examination of Water and Wastes, 22nd Edition unless otherwise noted. All other methods referenced are EPA numbers, AOAC Method Numbers or BAM-FDA reference methods.

* All of these analyses can be performed out of one 1/2 gallon plastic container.

*** Food and Bottled Beverage samples for Bacteriological analyses are submitted in sealed product containers provided by client. Container size depends on product container.

1. Holding times per 40 CFR 141 for drinking waters, and CFR 136.3 for wastewaters. Preservative, as indicated, must be present for holding time to be valid.
2. Travel Blank (also preserved with HCl).
3. If Chlorine Residual is present, ascorbic acid (524.2) or sodium sulfite (525) or sodium thiosulfate (all other methods) is needed to neutralize free chlorine. Dechlorinator must be added prior to additional preservation. Nonvolatile samples, suspected of containing chlorine, are screened for chlorine and additional dechlorinator is added as needed. Consult method.
4. Potassium dihydrogen citrate (PDC) is added at the ratio of 0.37g per 40 mL sample.
5. Sample preserved at lab after Electrical Conductivity is checked.
6. Preserved sample is screened for chlorine as necessary and treated at lab. See SOP A06 for more details.
7. See method exceptions.
8. Sample can be preserved at lab in its original container and must be held > 24 hrs. prior to analysis. (> 16 hrs. for UCMR3)
9. Collect grab sample in 1 quart plastic container, fill completely, eliminating all headspace.
10. Grab sample only.
11. Consult laboratory for special instructions.
12. With Sodium thiosulfate
13. Analysis is subbed out. Please allow extra time for short holding time analyses.
14. Client submits unpreserved sample which is screened for sulfide and chlorine as necessary and preserved to pH>12 with NaOH upon receipt to the laboratory. See SOP A06 for more details.
15. Wastewater samples should be filtered in the field (within 15 minutes) and preserved by the laboratory within 24 hours. Drinking water samples should be preserved in the field and filtered at the bench. Preservation includes addition of NH₄ buffer to pH 9.0-9.5.
16. For client safety, sample is preserved at lab with nitric acid, in its original container, and held > 28 hrs. prior to analysis.
17. Sparge sample with an inert gas (helium, argon, nitrogen) prior to preservation if chlorine dioxide is present.
18. The NH₄OH/(NH₄)₂SO₄ preservative serves as buffer and dechlorinating agent. May be added to sample bottles prior to shipment. Apply the preservative at the rate of 1 mL per 100 mL of sample.
19. For 40-mL vial, add 25 mg of ascorbic acid and 200 mg of maleic acid.
20. Trizma crystals are added to each sample bottle prior to shipment to the field (or prior to sample collection). Add 1.25 g per 250mL.
21. Add preservation reagents to each sample bottle prior to shipment to the field (or prior to sample collection). Add 80 mg Sodium Thiosulfate and 65 mg 2-mercaptopyridine-1-oxide Na per Liter.
22. For UCMR3 sample temperature requirements, refer to "Sample Containers, Preservation and Holding Times for UCMR3" table listed below.
23. Sample must be submitted with headspace. Fill sample container no more than half full to ensure sample is aerobic.
24. Samples which are biologically active must be preserved by adding sulfuric acid to pH 2 to prevent adsorption of method analytes by the humectant material.

Basic Sampling Guidelines

- A. Always utilize proper sampling containers and preservatives.
- B. For organic analytes, all bottles should have Teflon lined lids, vials should have Teflon lined septa.
- C. Soil samples are typically collected in brass or steel tubes and wide mouth jars (500ml) with Teflon-lined caps. Sludges should be collected in wide mouth jars, not brass or steel tubes. Store at <6°C
- D. Aqueous samples for volatile analyses should not have head space between the sample matrix and septum, or bubbles within the sample.
- E. Samples requiring organic analyses should never be handled with plastic implements, latex gloves, or stored in plastic containers. Glass is the only acceptable container (except EPA 549).
- F. Always use trip blanks when samples require volatile analyses. Fill completely, eliminate all headspace.
- G. Keep samples isolated from all possible sources of contamination (i.e., gasoline refueling operations, solvents, paints, lacquers, and adhesives).
- H. Always complete a Chain-of-Custody form.
- I. Use blue ice packs in coolers when possible.
- J. Deliver samples directly to the laboratory as soon as possible.