

COLLECTION AND PRESERVATION OF SAMPLES (1060)/Collection of Samples

TABLE 1060:I. SUMMARY OF SPECIAL SAMPLING AND HANDLING REQUIREMENTS*

Determination	Container†	Minimum Sample Size mL	Sample Type‡	Preservation§	Maximum Storage Recommended	Regulatory
Acidity	P, G(B), FP	100	g	Cool, $\leq 6^{\circ}\text{C}$	24 h	14 d
Alkalinity	P, G, FP	200	g	Cool, $\leq 6^{\circ}\text{C}$	24 h	14 d
BOD	P, G, FP	1000	g, c	Cool, $\leq 6^{\circ}\text{C}$	6 h	48 h
Boron	F, P (PTFE) or quartz	1000	g, c	HNO_3 to $\text{pH} < 2$	28 d	6 months
Bromide	P, G, FP	100	g, c	None required	28 d	28 d
Carbon, organic, total	G(B), P, FP	100	g, c	Analyze immediately, or cool $\leq 6^{\circ}\text{C}$ and add HCl, H_3PO_4 , or H_2SO_4 to pH	7 d	28 d
Carbon dioxide	P, G	100	g	Analyze immediately	0.25 h	N.S.
COD	P, G, FP	100	g, c	Analyze as soon as possible, or add H_2SO_4 to $\text{pH} < 2$; Cool, $\leq 6^{\circ}\text{C}$	7 d	28 d
Chloride	P, G, FP	50	g, c	None required	N.S.	28 d
Chlorine, total, residual	P, G	500	g	Analyze immediately	0.25 h	0.25 h
Chlorine dioxide	P, G	500	g	Analyze immediately	0.25 h	N.S.
Chlorophyll	P, G	500	g	Unfiltered, dark, $\leq 6^{\circ}\text{C}$ Filtered, dark, -20°C (Do not store in frost-free freezer)	24–48 h 28 d	N.S.
Color	P, G, FP	500	g, c	Cool, $\leq 6^{\circ}\text{C}$	24 h	48 h
Specific conductance	P, G, FP	500	g, c	Cool, $\leq 6^{\circ}\text{C}$	28 d	28 d
Cyanide Total	P, G, FP	1000	g, c	Analyze within 15 min. Add NaOH to $\text{pH} > 12$ if sample is to be stored, Cool, $\leq 6^{\circ}\text{C}$, in dark. Add thiosulfate if residual chlorine present	24 h	14 d; 24 h if sulfide present
Amenable to chlorination	P, G, FP	1000	g, c	Remove residual chlorine with thiosulfate and cool $\leq 6^{\circ}\text{C}$	stat	14 d; 24 h if sulfide present
Fluoride	P	100	g, c	None required	28 d	28 d
Hardness	P, G, FP	100	g, c	Add HNO_3 or H_2SO_4 to $\text{pH} < 2$	6 months	6 months
Iodine	P, G	500	g	Analyze immediately	0.25 h	N.S.
Metals	P(A), G(A), FP (A)	1000	g, c	For dissolved metals filter immediately, add HNO_3 to $\text{pH} < 2$	6 months	6 months
Chromium VI	P(A), G(A), FP (A)	250	g	Cool, $\leq 6^{\circ}\text{C}$, pH 9.3–9.7, ammonium sulfate buffer preservative as specified in method 3500-Cr to extend to 28 d HT	28 d	28 d
Copper by colorimetry	—*	—	g, c	—	—	—
Mercury	P(A), G(A), FP(A)	500	g, c	Add HNO_3 to $\text{pH} < 2$, Cool $\leq 6^{\circ}\text{C}$	28 d	28 d
Nitrogen						
Ammonia	P, G, FP	500	g, c	Analyze as soon as possible or add H_2SO_4 to $\text{pH} < 2$, Cool, $\leq 6^{\circ}\text{C}$	7 d	28 d
Nitrate	P, G, FP	100	g, c	Analyze as soon as possible; Cool, $\leq 6^{\circ}\text{C}$	48 h	48 h (14 d for chlorinated samples)
Nitrate + nitrite	P, G, FP	200	g, c	Add H_2SO_4 to $\text{pH} < 2$, Cool, $\leq 6^{\circ}\text{C}$	1–2 d	28 d
Nitrite	P, G, FP	100	g, c	Analyze as soon as possible; Cool, $\leq 6^{\circ}\text{C}$	none	48 h
Organic, Kjeldahl	P, G, FP	500	g, c	Cool, $\leq 6^{\circ}\text{C}$, add H_2SO_4 to $\text{pH} < 2$	7 d	28 d
Odor	G	500	g	Analyze as soon as possible; Cool $\leq 6^{\circ}\text{C}$	6 h	24 h (EPA Manual drinking water)
Oil and grease	G, wide-mouth calibrated	1000	g	Add HCl or H_2SO_4 to $\text{pH} < 2$, Cool, $\leq 6^{\circ}\text{C}$	28 d	28 d

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TABLE 1060:I. CONT.

Determination	Container†	Minimum Sample Size mL	Sample Type‡	Preservation§	Maximum Storage Recommended	Regulatory
Organic Compounds						
MBAS	P, G, FP	250	g, c	Cool, $\leq 6^{\circ}\text{C}$	48 h	48 h as per CFR 136
Pesticides*	G(S), PTFE-lined cap	1000	g, c	Cool, $\leq 6^{\circ}\text{C}$, add 1000 mg ascorbic acid/L if residual chlorine present (0.008% sodium thiosulfate in CFR 136)	7 d	7 d until extraction; 40 d after extraction
Phenols	P, G, PTFE-lined cap	500	g, c	Cool, $\leq 6^{\circ}\text{C}$, add H_2SO_4 to $\text{pH} < 2$	*	28 d until extraction, 2 d after extraction
Purgeables* by purge and trap	G, PTFE-lined cap	2×40	g	Cool, $\leq 6^{\circ}\text{C}$; add HCl to $\text{pH} < 2$; add 1000 mg ascorbic acid/L if residual chlorine present (0.008% sodium thiosulfate in CFR 136)	7 d	14 d
Base/ neutrals & acids	G(S) amber	1000	g, c	Cool, $\leq 6^{\circ}\text{C}$, 0.008% sodium thiosulfate in CFR 136 if chlorine is present	7 d	7 d until extraction; 40 d after extraction
Oxygen, dissolved	G, BOD bottle	300	g	Analyze immediately	0.25 h	0.25 h
Electrode				Titration may be delayed after acidification	8 h	8 h
Winkler						
Ozone	G	1000	g	Analyze immediately	0.25 h	N.S.
pH	P, G	50	g	Analyze immediately	0.25 h	0.25 h
Phosphate	G(A)	100	g	For dissolved phosphate filter immediately; Cool, $\leq 6^{\circ}\text{C}$	48 h	48 h as per EPA manual for DW
Phosphorus, total	P, G, FP	100	g, c	Add H_2SO_4 to $\text{pH} < 2$ and cool, $\leq 6^{\circ}\text{C}$	28 d	28 d
Salinity	G, wax seal	240	g	Analyze immediately or use wax seal	6 months	N.S.
Silica	F, P (PTFE) or quartz	200	g, c	Cool, $\leq 6^{\circ}\text{C}$, do not freeze	28 d	28 d
Sludge digester gas	G, gas bottle	—	g	—	N.S.	
Solids ⁹	P, G	200	g, c	Cool, $\leq 6^{\circ}\text{C}$	7 d	2–7 d; see cited reference
Sulfate	P, G, FP	100	g, c	Cool, $\leq 6^{\circ}\text{C}$	28 d	28 d
Sulfide	P, G, FP	100	g, c	Cool, $\leq 6^{\circ}\text{C}$; add 4 drops 2N zinc acetate/100 mL; add NaOH to $\text{pH} > 9$	28 d	7 d
Temperature	P, G, FP	—	g	Analyze immediately	0.25 h	0.25 h
Turbidity	P, G, FP	100	g, c	Analyze same day; store in dark up to 24 h, Cool, $\leq 6^{\circ}\text{C}$	24 h	48 h

* For determinations not listed, use glass or plastic containers; preferably refrigerate during storage and analyze as soon as possible.

† P = plastic (polyethylene or equivalent); G = glass; G(A) or P(A) = rinsed with 1 + 1 HNO_3 ; G(B) = glass, borosilicate; G(S) = glass, rinsed with organic solvents or baked; FP = fluoropolymer [polytetrafluoroethylene (PTFE, Teflon) or other fluoropolymer].

‡ g = grab; c = composite.

§ Cool = storage at, $> 0^{\circ}\text{C}$, $\leq 6^{\circ}\text{C}$ (above freezing point of water); in the dark; analyze immediately = analyze usually within 15 min of sample collection.

|| See citation¹⁰ for possible differences regarding container and preservation requirements. N.S. = not stated in cited reference; stat = no storage allowed; analyze immediately (within 15 min).

Some drinking water (DW) and treated wastewater (WW) matrices may be subject to positive interference as a result of preservation. If such interference is demonstrable, samples should be analyzed as soon as possible without preservation. Do not hold for more than 15 min without demonstrating that cyanide (CN) is stable for longer periods in a specific matrix.

NOTE: This table is intended for guidance only. If there is a discrepancy between this table and the method, the information in the current method takes precedence. If performing the method for compliance purposes, be aware that alternative preservation and holding-time requirements may exist. If so, the regulatory requirements should be used.