APPENDIX B

Holding Times and Preservation

Table B-1
Qualification for Holding Times

Parameter	Preservative [4]		Holding Time		Flagging for Holding Time Noncompliance	
	Liquid	Solid	Liquid	Solid	Liquid	Solid
VOLATILE ORGANICS	Cool 4°C [12] No head space HCl to pH <2[8]	Cool 4°C, $NaHSO_4$ (aq) to pH < 2	14 d [9]	14 d [10]	Note 1	Note 1
		Cool 4°C Methanol				Note 2
Purgeable Aromatic Hydrocarbons	Cool 4°C No head space HCl to pH <2[8, 12]	Cool 4°C, $NaHSO_4$ (aq) to pH <2	14 d [9]	14 d [10]	Note 1	Note 1
		Cool 4°C Methanol				Note 2
Purgeable Halocarbons	Cool 4°C No head space[8,12]	Cool 4°C, $NaHSO_4$ (aq) to pH <2	14 d	14 d [10]	Note 2	Note 1
		Cool 4°C Methanol				Note 2
SEMIVOLATILE ORGANICS	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Benzidines	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [9]	Note 1	Note 1
Chlorinated Herbicides	Cool 4°C	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Chlorinated Hydrocarbons	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Chlorinated Pesticides	Cool 4°C	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Polychlorinated Dibenzo Dioxins & Furans	Cool 4°C [12]	Cool 4°C	30 d /45 d [6]	30 d/45 d [6]	Note 2	Note 2
Explosives	Cool 4°C	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Haloethers	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Nitroaromatics & cyclic ketones	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Nitrosamines	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
PCBs	Cool 4°C	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2

Parameter	Preservative [4]		Holding Time		Flagging for Holding Time Noncompliance	
	Liquid	Solid	Liquid	Solid	Liquid	Solid
Phenolics	Cool 4°C, H_2SO_4 to pH < 2	Cool 4°C	28 d	28 d	Note 2	Note 2
Phenols	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Phthalate Esters	Cool 4°C	Cool 4°C	7 d/40 d [3]	14 d/40 d [5]	Note 2	Note 2
Polynuclear Aromatic Hydrocarbons	Cool 4°C [12]	Cool 4°C	7 d/40 d [3]	14 d /40 d [5]	Note 2	Note 2
METALS Metals except Cr(IV) and Hg	Cool 4°C, HNO_3 to pH < 2	Cool 4°C	6 months	6 months	Note 2	Note 2
Chromium (VI)	Cool 4°C	Cool 4°C	24 hours	24 hours	Note 1	Note 1
Mercury	Cool 4°C, HNO_3 to pH < 2	Cool 4°C	28 d	28 d	Note 1	Note 1
MISCELLANEOUS Cyanide (Total & Amenable)	Cool 4°C, NaOH to pH >12 [11]	Cool 4°C	14 d	14 d	Note 1	Note 1
TRPH	Cool 4°C, H_2SO_4 to pH < 2	Cool 4°C	28 d	28 d	Note 2	Note 2
Acidity	Cool 4°C	NA	48 hours	NA	Note 1	NA
Alkalinity	Cool 4°C	NA	48 hours	NA	Note 1	NA
Ammonia	Cool 4°C, H_2SO_4 to pH < 2	NA	28 d	NA	Note 1	NA
Biochemical Oxygen Demand (BOD)	Cool 4°C, H_2SO_4 to pH < 2	NA	48 hours	NA	Note 1	NA
Biochemical Oxygen Demand (Carbonaceous)	Cool 4°C	NA	48 hours	NA	Note 1	NA
Bromide	None	NA	28 d	NA	Note 2	NA
Chemical Oxygen Demand (COD)[1]	Cool 4°C, H_2SO_4 to pH< 2	NA	28 d	NA	Note 1	NA
Chloride	None	None	28 d	28 d	Note 2	Note 2
Chlorine, Total Residual	None	NA	A.S.A.P.	NA	NA	NA
Coliform, Fecal & Total	Cool 4°C [12]	Cool 4°C	6 hours	6 hours	Note 1	Note 1
Color	Cool 4°C	NA	48 hours	NA	Note 1	NA
Common Ions	Cool 4°C	Cool 4°C	28 d	28 d	Note 2	Note 2
Dissolved Oxygen, Probe	None Required	NA	A.S.A.P	NA	Note 1	NA
Dissolved Oxygen, Winkler Method	Fix on Site /Store in dark	NA	8 hours	NA	Note 1	NA
Fecal Streptococci	Cool 4°C [12]	NA	6 hours	NA	Note 1	NA
Fluoride	None Required	NA	28 d	NA	Note 2	NA

Parameter	Preservative [4]		Holding Time		Flagging for Holding Time Noncompliance	
	Liquid	Solid	Liquid	Solid	Liquid	Solid
Hardness	HNO_3 or H_2SO_4 to pH < 2	NA	6 months	NA	Note 2	NA
Hydrogen Ion (pH)	None	Cool 4°C	A.S.A.P.	A.S.A.P.	NA	
Kjeldahl & Organic Nitrogen	Cool 4°C, H_2SO_4 to pH < 2	NA	28 d	NA	Note 2	NA
Nitrate	Cool 4°C	Cool 4°C	48 hours	48 hours	Note 1	Note 1
Nitrate-Nitrite	Cool 4°C, H_2SO_4 to pH < 2	Cool 4°C	28 d	28 d	Note 1	Note 1
Nitrite	Cool 4°C	NA	48 hours	NA	Note 1	NA
Oil & Grease	Cool 4°C, H_2SO_4 to pH < 2	Cool 4°C	28 d	28 d	Note 2	Note 2
Organic Carbon	Cool 4°C, HCl or H_2SO_4 to pH < 2	Cool 4°C	28 d	28 d	Note 2	Note 2
Orthophosphate	Filter Immediately Cool 4°C	NA	48 hours	NA	Note 1	NA
Phosphorus (Elemental)	Cool 4°C	NA	48 hours	NA	Note 1	NA
Phosphorus (Total)	Cool 4°C, H_2SO_4 to pH < 2	NA	28 d	NA	Note 1	NA
Gross Alpha	HNO_3 to pH < 2	Cool 4°C	6 months	6 months	Note 2	Note 2
Gross Beta	HNO_3 to pH < 2	Cool 4°C	6 months	6 months	Note 2	Note 2
Radium (Total)	HNO_3 to pH < 2	Cool 4°C	6 months	6 months	Note 2	Note 2
Residue, Filterable	Cool 4°C	NA	7 d	NA	Note 1	NA
Residue, Non-Filterable	Cool 4°C	NA	7 d	NA	Note 1	NA
Residue, Settleable	Cool 4°C	NA	48 hours	NA	Note 1	NA
Residue, Total	Cool 4°C	NA	7 d	NA	Note 1	NA
Residue, Volatile	Cool 4°C	NA	7 d	NA	Note 1	NA
Silica	Cool 4°C	NA	28 d	NA	Note 2	NA
Specific Conductance	Cool 4°C	NA	28 d	NA	Note 2	NA
Sulfate	Cool 4°C	Cool 4°C	28 d	28 d	Note 2	Note 2
Sulfide	Cool 4°C, 4 mL ZnAc plus NaOH to pH > 9	Cool 4°C	7 d	7 d	Note 1	Note 1
Sulfite	Cool 4°C	NA	A.S.A.P.	NA	NA	NA
Surfactant	Cool 4°C	NA	48 hours	NA	Note 1	NA
TCLP Volatile Organics	Cool 4°C	Cool 4°C	14 d/NA/ 14 d [7]	14 d/NA/ 14 d [7]	Note 1	Note 1

Parameter	Preservative [4]		Holding Time		Flagging for Holding Time Noncompliance	
	Liquid	Solid	Liquid	Solid	Liquid	Solid
TCLP Extractable Organics	Cool 4°C No headspace	Cool 4°C, No Headspace	14 d/7d/ 40 d [7]	14 d/7d/ 40 d [7]	Note 1	Note 1
TCLP Inorganics, Hg	Cool 4°C	Cool 4°C	28 d/NA/ 28 d [7]	28 d/NA /28 d [7]	Note 1	Note 1
TCLP Inorganics, all other metals	Cool 4°C	Cool 4°C	180 d/NA/ 180 d [7]	180 d/NA/ 180 d [7]		
Temperature	None Required	NA	A.S.A.P.	NA	NA	NA
Total Organic Halogens (TOX)	Cool 4°C, H_2SO_4 to pH < 2	28 d	28 d	28 d	Note 2	Note 2
Turbidity	Cool 4°C	NA	48 hours	NA	Note 1	NA

Notes:

1. Qualify all nondetections with the R flag and all detections with the J- flag or X flag when the holding time limit is exceeded.
2. When the holding time limit is exceeded but a gross holding violation does not occur, qualify all nondetections with the UN flag and all detections with the J- flag. When the holding time is grossly exceeded, qualify all nondetections with the R flag and all detections with the J- flag or X flag.
3. 7 days until extraction; analyzed within 40 days of extraction.
4. High concentration samples only require cooling to 4°C.
5. 14 days until extraction; analyzed within 40 days of extraction.
6. 30 days from collection to extraction; 45 days from collection to analysis.
7. Holding times shown are as follows: From collection to TCLP extraction; from TCLP extraction to preparative procedure; from preparative procedure to analysis.
8. All aqueous VOC samples must be protected from light. Project-specific preservatives (e.g., mercuric chloride, copper sulfate, and sodium azide) may be used when matrix interference (e.g., high carbonates) precludes the use of acid preservation.
9. For aqueous aromatic VOCs, the holding time is 7 days if the samples have not been preserved with HCl or a project-specific preservative (but have otherwise been properly preserved).
10. If the samples are stored in an air-tight vessel (e.g., the EnCore sampler) at 4°C without taking additional preservation measures (e.g., methanol immersion and acidification), then the holding time is only 48 hours. Sample results should be qualified for holding time exceedences as described in Note 2.
11. If oxidizing agents are present (e.g.,residual chlorine), add 0.06 g of ascorbic acid or 5 mL of 0.1 N $NaAsO_2$ per liter of sample.
12. Free chlorine must be removed by the addition of 0.008% $Na_2S_2O_3$.

    d = day
    NA = Not Applicable
    A.S.A.P = As soon as possible, immediately