Appendix B-1 Sample Collection Parameters for Metals and Classical Chemistry Parameters

Target Analyte/Method	Matrix	Containers	Volume/Mass	Special Note(s)	Preservative	Holding Time ¹
Metals/ICP-AES	Water	Polyethylene or Glass	1 Liter		Acidify to pH < 2 w/HNO ₃ & Cool to 4°C immediately after collection.	1
	Soil/ Sediment	Polyethylene or Glass	100 grams		Cool to 4°C immediately after collection.	1
Metals/ICP-MS	Water	Polyethylene or Glass	1 Liter		Acidify to pH < 2 w/HNO3 & Cool to 4°C immediately after collection. ²	1
	Soil/ Sediment	Polyethylene or Glass	100 grams		Cool to 4°C immediately after collection.	1
Mercury/Cold Vapor AA	Water	Polyethylene or Glass	1 Liter	Sampling devices and sample containers must be free of mercury.	Acidify to pH < 2 w/HNO ₃ immediately after collection.	28 days
	Soil/ Sediment		100 grams	Sampling devices and sample containers must be free of mercury.	Cool to 4°C immediately after collection.	28 days
Cyanide/Spectro- photometric Determination ³	Water	Polyethylene or Glass	2 Liters		Immediately upon collection, add 0.6g ascorbic acid for each liter of sample collected (to neutralize residual chlorine). Add NaOH until pH > 12 and Cool to 4°C immediately after collection.	1

For all metals except for mercury and Cr⁶⁺ the holding time is 6 months. The holding time for mercury is 28 days, and the holding time for Cr⁶⁺ is 24 hours. This holding time is calculated from the time of sample collection. It is recommended that samplers ship samples to the lab the same day that they are collected, or as soon as possible thereafter.

² Note that the sampler may be required to filter the sample through a 0.45-um pore diameter membrane filter at the time of collection. Use a portion of the sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Then preserve as described above. This occurs when the lab will be testing for dissolved elements.

³ Sulfides adversely affect the analytical procedure. The following can be done to test for and neutralize sulfides. Place a drop of the sample on lead acetate test paper to detect the presence of sulfides. If sulfides are present, treat 25 mL more of the sample than that required for the cyanide determination with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide. Repeat this operation until a drop of the treated sample solution does not darken

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	Soil/ Sediment	Polyethylene or Glass	100 grams		Cool to 4°C immediately after collection.	1
Hexavalent Chromium/Atomic Absorption Spectroscopy	Water	Polyethylene or Glass	500 mL		Cool to 4°C immediately after collection.	24 hours
	Soil/ Sediment	Polyethylene or Glass	100 grams		Cool to 4°C immediately after collection.	24 hours
Hexavalent Chromium/lon Chromatography	Water	125 mL Narrow Mouth, High-Density Polypropylene Containers or Equivalent	100 mL		Adjust pH to 9-9.5 by drop- wise addition of buffer solution and Cool to 4°C immediately after collection. ⁴	24 hours
	Soil/ Sediment	Polyethylene or Glass	100 grams		Cool at 4°C immediately after collection	24 hours
TPH/Fluorocarbon- 113 Extraction and IR analysis	Water -do not collect composite samples.	Glass bottle	1 Liter		If more than 4 hours will pass between sampling and analysis, preserve by the addition of 5 mL 1:1 HC1. If more than 48 hours will pass between sampling and analysis, cool to 4°C immediately after collection.	1
	Soil/ Sediment	Glass bottles	100 grams		Cool to 4°C immediately after collection.	1

the lead acetate test paper. Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material.

⁴ Note that the sampler may be required to filter the sample through a 0.45-um pore diameter membrane filter at the time of collection. Use a portion of the sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Then preserve as described above. This occurs when the lab will be testing for dissolved elements.