

Aquifer Protection and Underground Storage Tank Preservation and Hold Time Table

Aqueous Samples					
Listed below is information on the collection and preservation of samples. Filtered samples are requested for some parameters as recommended by the USGS manual. If you are submitting filtered (using a 0.45 µm filter) samples, write "DIS" (meaning dissolved) in the block beside applicable parameter(s) on the field sheet. The amount of sample listed is for routine conditions. If you suspect that unusual conditions or interferences exist, please submit double the amount of sample. Excluding purgeable organics and sulfide, a one-half inch air space should be left in all bottles to allow for mixing before analysis.					
Parameter ⁽²⁾	Minimum Required Volume	Container ^{(1) (14)}	F-filtered U-unfiltered	Preservation ⁽¹⁹⁾	Maximum Hold Time ⁽²⁰⁾
A. Alkalinity ⁽¹⁸⁾	200 mL	P (Disposable)	U	Cool, ≤6°C	14 days
A. Bicarbonate	combined w/above	Request on Field Sheet and submit alkalinity sample.			
A. Carbonate	combined w/above	Request on Field Sheet and submit alkalinity sample.			
pH	Inappropriate for laboratory analysis				Immediate - field measurement
Carbon Dioxide	Inappropriate for laboratory analysis				Immediate - field measurement
Chromium, Hexavalent	200 mL	P (Disposable)	U	Cool, ≤6°C	24 hours (notify lab of collection)
Color	200 mL	P (Disposable)	U	Cool, ≤6°C	48 hours ⁽⁶⁾
MBAS	500 mL	P (Disposable)	U	Cool, ≤6°C	48 hours ⁽⁶⁾ (notify lab of collection)
Specific Conductance	200 mL	P (Disposable)	U	Cool, ≤6°C	28 days
D. Chloride	500 mL x 1	P (Disposable)	U	Cool, ≤6°C when combined with SO4 - no thermal preservation required if requesting chloride only	28 days
D. Fluoride	combined w/above	P (Disposable)	U	Cool, ≤6°C when combined with SO4 - no thermal preservation required if requesting fluoride only	28 days
D. Sulfate	combined w/above	P (Disposable)	U	Cool, ≤6°C	28 days
Total Hardness (request by checking Ca and Mg on field sheet - can be part of metals suite) Total Hardness (mg CaCO3/L) = 2.497 [Ca, mg/L] + 4.118 [Mg, mg/L]	500 mL	P (Disposable)	U	1+1 HNO3 to pH<2	6 months
Non-Carbonate Hardness ⁽³⁾ [Non-carbonate Hardness = total hardness - total alkalinity]	Submit samples for Total Hardness and Alkalinity, as specified.				
HEM: Oil and Grease	2 liters (two 1-liter bottles)	G (Wide-mouth quart jar, Teflon-lined cap)	U	Cool, ≤6°C, 6N H2SO4 to pH<2	28 days
Silica	200 mL	P (Disposable)	U	Cool, ≤6°C	28 days
Sulfide	40 mL x 3 ⁽²¹⁾	G (40 mL VOA vials with Teflon-lined septum)	U	Cool, ≤6°C, add 0.1 mL 2N zinc acetate plus 6N NaOH to pH>9, leave NO headspace in the bottle	7 days
Cyanide, Total	2 liters (two 1-liter bottles)	P	U	Cool, ≤6°C, 0.6 g ascorbic acid ⁽⁴⁾ , 6N NaOH to pH>12	14 days ⁽¹²⁾
Phenols, Total Recoverable	2 liters (two 1-liter bottles)	G (Phenol bottle) only ⁽⁵⁾	U	Cool, ≤6°C, 1:1 H2SO4 to pH<2 (1 mL ferrous ammonium sulfate if sample contains oxidizer)	28 days

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Parameter ⁽²⁾	Minimum Required Volume	Container ⁽¹⁾⁽¹⁴⁾	F-filtered U-unfiltered	Preservation ⁽¹⁹⁾	Maximum Hold Time ⁽²⁰⁾
C. Metals: Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr (Total), Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Sb, Sn, Se, Tl, V, Zn and Hg)	500 mL x 1	P (Disposable)	U	1+1 HNO ₃ to pH<2	72 hours - should be delivered to lab within 48 hours to allow for sample preparation time (28 days for Hg)
Boron ⁽²²⁾	500 mL x 1	P (Disposable)	U	1+1 HNO ₃ to pH<2	72 hours - should be delivered to lab within 48 hours to allow for sample preparation time (28 days for Hg)
BOD, 5-day	1 liter	P	U	Cool, ≤6°C	48 hours ⁽⁶⁾
CBOD, 5-day	1 liter	P	U	Cool, ≤6°C	48 hours ⁽⁶⁾
COD	200 mL	P (Disposable)	U	Cool, ≤6°C, 25% H ₂ SO ₄ to pH<2	28 days
Coliform (Total or Fecal)	250 mL each	P (Sterile) ⁽⁷⁾	U	Cool, <10°C, 0.008% Na ₂ S ₂ O ₃ (0.1 mL 10% Na ₂ S ₂ O ₃ per 125 mL) and 15% EDTA ⁽⁷⁾	6 hours ⁽⁸⁾
TOC	200 mL	P (Disposable)	U	Cool, ≤6°C, H ₃ PO ₄ to pH<2	28 days
DOC	200 mL - A Field Blank must accompany all DOC samples	P (Disposable)	F	Field filter using 0.45 µm filter, Cool, ≤6°C, H ₃ PO ₄ to pH<2	28 days
Turbidity	200 mL	P (Disposable)	U	Cool, ≤6°C	48 hours ⁽⁶⁾
C. NH₃ as N	500 mL x 1	P (Disposable)	U	Cool, ≤6°C, 25% H ₂ SO ₄ to pH<2 ⁽⁹⁾ (0.008% Na ₂ S ₂ O ₃ if chlorine present) ⁽¹¹⁾	28 days
C. TKN as N	combined w/above	P (Disposable)	U	Cool, ≤6°C, 25% H ₂ SO ₄ to pH<2 ⁽⁹⁾ (0.008% Na ₂ S ₂ O ₃ if chlorine present) ⁽¹¹⁾	28 days
C. NO₃+NO₂ as N	combined w/above (except when NH ₃ and TKN require dechlorination)	P (Disposable)	U	Cool, ≤6°C, 25% H ₂ SO ₄ to pH<2 ⁽⁹⁾	28 days
C. TP, total as P	combined w/above (except when NH ₃ and TKN require dechlorination)	P (Disposable)	U	Cool, ≤6°C, 25% H ₂ SO ₄ to pH<2 ⁽⁹⁾	28 days
TP, dissolved as P	200 mL	P (Disposable)	F	Filter immediately, Cool, ≤6°C, 25% H ₂ SO ₄ to pH<2 ⁽⁹⁾	28 days
PO ₄ as P	200 mL	P (Disposable)	F	Filter immediately, Cool, ≤6°C	48 hours
NO ₂ as N	200 mL	P (Disposable)	U	Cool, ≤6°C	48 hours (notify lab of collection)

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Parameter ⁽²⁾	Minimum Required Volume	Container ^{(1) (14)}	F-filtered U-unfiltered	Preservation ⁽¹⁹⁾	Maximum Hold Time ⁽²⁰⁾
NO3 as N	NO3 as N is calculated from the analytical results obtained for NO3+NO2 as N and NO2 as N. Submit one bottle for NO3+NO2 and one bottle for NO2 as instructed above.				
Residue (TS, TSS) ⁽¹⁸⁾	500 mL each	P (Disposable)	U	Cool, ≤6°C	7 days
Total Dissolved Solids (TDS)	500 mL	P (Disposable)	U	Cool, ≤6°C	7 days
Semivolatile Organics - Base/Neutral Acid Extractables	1 gal ⁽¹⁰⁾	G (Amber with Teflon-lined cap)	U	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) ⁽¹¹⁾	7 days until extraction 40 days after extraction
Pesticides/PCBs (OP Pest/OC Pest/ON Pest)	1 gal ⁽¹⁰⁾	G (Amber with Teflon-lined cap)	U	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) ⁽¹¹⁾	7 days until extraction ⁽¹⁶⁾ 40 days after extraction
Acid Herbicides	1 gal ⁽¹⁰⁾	G (Amber with Teflon-lined cap)	U	Cool, ≤6°C, 0.008% Na2S2O3 if chlorine present) ⁽¹¹⁾	7 days until extraction ⁽¹⁶⁾ 40 days after extraction
Purgeable (Volatile) Organics (VOA)	40 mL x 4 - A Trip Blank (3 vials) must accompany all VOA samples	G (Teflon-lined septum)	U	Cool, ≤6°C, 0.6 g ascorbic acid ⁽¹³⁾ if chlorine present, Sodium Bisulfate (NaHSO4) ⁽¹³⁾ to pH<2 ^{(15) (17)} , Leave no headspace in the bottle	14 days (7 days for aromatics only when unpreserved)
TPH Gasoline Range Organics and BTEX (aqueous)	40 mL x 4 - A Trip Blank (3 vials) must accompany all VOA samples	G (Teflon-lined septum)	U	Cool, ≤6°C, 0.6 g ascorbic acid ⁽¹³⁾ if chlorine present, Sodium Bisulfate (NaHSO4) ⁽¹³⁾ to pH<2 ^{(15) (17)} , Leave no headspace in the bottle	14 days
TPH Diesel Range Organics (aqueous)	1 gal	G (Teflon-lined cap)	U	Cool, ≤6°C	14 days until extraction 40 days after extraction

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Soil Samples

When submitting soil and sludge samples for analysis, a separate sample container must be collected for each of the analytical groups listed below:

Parameter/Analytical Group	Minimum Required Volume	Container ⁽¹⁾ ⁽¹⁴⁾	F-filtered U-unfiltered	Preservation ⁽¹⁹⁾	Maximum Hold Time ⁽²⁰⁾
Oil and Grease	8 oz. jar	G, Teflon-lined cap	N/A	Cool, ≤6°C	refer to aqueous
Metals: Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr (Total), Cu, Fe, K, Li, Mg, Mn, Na, Ni, Pb, Sb, Sn, Se, Tl, V, Zn and Hg	8 oz. jar	G, Teflon-lined cap	N/A	Cool, ≤6°C	6 months (28 days for Hg)
Pesticides/PCBs (OP Pest/OC Pest/ON Pest)	8 oz. jar	G, Teflon-lined cap	N/A	Cool, ≤6°C	14 days to extract; analyze within 40 days
Acid Herbicides	8 oz. jar	G, Teflon-lined cap	N/A	Cool, ≤6°C	14 days to extract; analyze within 40 days
Semivolatile Organics - Base/Neutral Acid Extractables	8 oz. jar	G, Teflon-lined cap	N/A	Cool, ≤6°C	14 days to extract; analyze within 40 days
Purgeable (Volatile) Organics (VOA)	4 oz. jar + trip blank	G, Teflon-lined cap or septum	N/A	Cool, ≤6°C	14 days
TPH Gasoline Range Organics and BTEX (soil)	4 oz. jar + trip blank	G, Teflon-lined cap or septum	N/A	Cool, ≤6°C	14 days
TPH Diesel Range Organics (soil)	8 oz. jar	G, Teflon-lined cap	N/A	Cool, ≤6°C	14 days to extract; analyze within 40 days

Footnotes

- (1) The container types listed are those commonly throughout the Department. Other container types may be acceptable. Please consult the laboratory about use of proper containers before deviating from those listed. P-plastic, G-glass, P (Disposable)-plastic disposable "juice"
- (2) Determinations preceded by the same letter (i.e., A, B, C) may be submitted in the same bottle if the bottle contains enough sample. If not letter precedes a parameter, it must be submitted in a separate bottle .
- (3) When non-carbonate hardness is requested, samples for both metals (Ca and Mg should be requested) and alkalinity must be submitted.
- (4) Add 0.6 g ascorbic acid only if the sample contains total residual chlorine.
- (5) Use two 1-liter round glass bottles.
- (6) 48 hours is the maximum holding time, however, samples should be submitted to the lab as soon as possible.
- (7) Use the 250 mL wide-mouth sterile plastic bottles for all samples. All bottles contain sodium thiosulfate and EDTA reagents.
- (8) Litigation samples must be delivered to the laboratory within 6 hours of sample collection.
- (9) Caution: Addition of excessive amounts of acid will interfere with the test procedures. The 2.0 mL of 25% H₂SO₄ per 500 mL sample should be added using a graduated or precise volume dispensing device. If no dispenser is available you may add exactly 40 drops of the 25% H₂SO₄. In most cases, the addition of 2.0 ml (~40 drops) of 25% H₂SO₄ to 500 mL of surface water will reduce the pH to <2, however, if the pH remains above 2, add acid dropwise with stirring until the pH is lowered to <2. For nutrient samples, the pH range of 1.5-2.0 is ideal to insure best possible recovery of analytes.
- (10) In a glass container, submit a small quantity of the pure compound of any suspected material.
- (11) Should only be used in the presence of residual chlorine. Add sodium thiosulfate or ascorbic acid (as appropriate) to the container first; fill at least half way before adding acid (if used). Adding 0.1 mL of a 10% solution of sodium thiosulfate (Na₂S₂O₃) per each 125 mL of sample is equivalent to 0.008% Na₂S₂O₃.
- (12) Maximum hold time is 24 hours when sulfide is present. Optionally, all samples may be tested on-site with lead acetate paper before pH adjustment in order to determine if sulfide is present. If it is, it can be removed by the addition of CdNO₃ powder until a negative spot test is obtained.
- (13) Used by the DWQ Chemistry Lab only at this time.
- (14) The container types listed are those commonly throughout the Department. Other container types may be acceptable. Please consult the laboratory about use of proper containers before deviating from those listed. P-plastic, G-glass, P (Disposable)-plastic disposable "juice" bottle.
- (15) Samples submitted for purgeable halocarbons only should not be acid-preserved.

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(16) Samples submitted for pesticide and acid herbicide analyses must be extracted within 72 hours of collection if the pH is not adjusted in the lab to a pH range of 5-9.

(17) Samples submitted for purgeable aromatics receiving no pH adjustment must be analyzed within 7 days of collection.

(18) Total Residue and Total Suspended Residue samples are to be shipped directly to the Central Laboratory for repacking and shipment to the Washington Regional Laboratory for analysis. Samples for these parameters collected in the Washington Region are sent directly to the WARO Lab.

(19) Sample preservation should be performed immediately upon collection. For composite samples, each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then the samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

(20) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Collection times must allow for sample preparation and analytical set-up. Some samples may not be stable for the maximum time period given in the table. Collectors are obligated to hold the sample for as short a time as possible especially if knowledge exists showing that this is necessary to maintain sample stability.

(21) Fill the bottle to overflowing and cap, leaving no air space (i.e., headspace).

(22) You must write in "Boron" in one of the blank cells on the field sheet to request Boron analysis and submit a separate metals sample.

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