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METHOD 300.0

I. SCOPE AND APPLICATION:

This sampling procedure is applicable for the determination of inorganic anions in drinking water, surface water, groundwater, reagent water, as well as to mixed domestic and industrial wastewater.

<u>Analyte</u>	<u>Chemical Abstract Services Registry Numbers (CASRN)</u>
Bromide	refer to elemental analyte
Chloride	refer to elemental analyte
Fluoride	refer to elemental analyte
Nitrate	not applicable
Nitrite	not applicable
<i>Ortho</i> -phosphate-P	not applicable
Sulfate	not applicable

II. REAGENTS:

- Sulfuric acid solution: (ultra high purity grade) 1:1 acid dilution. Add 500 ml of concentrated ultra pure nitric acid to 400 ml of ASTM type I water (reagent water-- free of analytes) and dilute to a volume of 1 liter.
- Ethylenediamine preservation solution: Dilute 10 ml of ethylenediamine (99%) (CASRN 107-15-3) to 200 ml of reagent water. Add 1 ml of this dilution to each 1-liter sample bottle.

III. MATERIALS:

- 1 liter (32 oz) high density polyethylene bottles with poly-foamed lined screw on caps.
- Collect duplicate samples if 500 ml sample bottles are used.
- Latex gloves
- Paper towels
- Kim wipes
- pH indicator paper (low end)
- Plastic container for disposal of used pipette tips
- Disposable glass pipette and rubber bulb.
- Protective eyewear

IV. PRESERVATION & HOLDING TIMES:

<u>Analyte</u>	<u>Preservation</u>	<u>Holding Time</u>
Bromate	None Required	28 days
Bromide	None Required	28 days
Chlorate	None Required	28 days
Chloride	None Required	28 days
Chlorite	Chill to 4°C	immediately
Fluoride	None Required	28 days
Nitrate-N	Chill to 4°C	48 hours
Combined nitrate/nitrite	H ₂ SO ₄ to pH _≤ 2	28 days
Nitrite-N	Chill to 4°C	48 hours
o-Phosphate-P	Chill to 4°C	48 hours
Sulfate	Chill to 4°C	28 days

The method of preservation and the holding time for samples analyzed by this method are determined by the inorganic anions of interest. In a given sample, the anion that requires the most preservation treatment as well as the shortest holding time will determine the preservation treatment.

If the sample cannot be analyzed for chlorite within 10 minutes, residual chlorine dioxide should be removed from the sample prior to the addition of ethylenediamine (EDA) by purging the sample with an inert gas for approximately 5 minutes. The sample may now be preserved by adding 1 ml of the ethylenediamine (EDA) preservation solution to 1 liter of sample. This will preserve the concentration of the chlorite for up to 14 days. The addition of EDA has no effect on bromate or chlorate, so they can be determined in a sample preserved with EDA.

V. PROCEDURE:

1. Remove any attachments such as hoses, screens or aeration devices on the faucet. Inspect the faucet for anything that may fall into the sample container.
2. Open the tap and allow the system to flush for approximately 10 minutes. This should be sufficiently long enough to get a representative sample.
3. *If sampling for inorganic anions, which do not require the addition of preservative,:* remove the cap from the polyethylene container. Because there is no dechlorinating agent or acid used to preserve the sample, it is preferred to rinse the bottle using a small volume of sample and discard the rinsate before collecting the actual sample.
4. To fill, tip the bottle at about a 45° angle into the stream of water. Slow the stream sufficiently so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to the fill line or within ½ inch of the top.
5. Remove the bottle from the flow and recap.
6. After obtaining the water samples (in duplicate if necessary), attach the preprinted sample label onto the bottle as well as the completed chain of custody seal around the plastic cap of each sample bottle.
7. *If sampling for inorganic anions that require the addition of preservative:* To fill, tip the bottle at about a 45° angle into the stream of water. Slow the stream sufficiently so as to be able to anticipate when the bottle is nearly full and thus avoid over flowing. Fill the bottle to the fill line or within ½ inch of the top. This will allow enough space for mixing and the addition of any additional acid if required.
8. Remove the bottle from the flow and recap. Invert the container five times.
9. Place a pH indicator test strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the pH indicator test strip with the aliquot from the glass pipette and immediately flick the pH indicator test strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference pH range. A determination must be made within 30 seconds.
10. If the pH is ≤ 2 , recap the bottle firmly, dry the sample bottle, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in field notebook and place the sample bottle in the ice chest.

V. PROCEDURE (continued):

11. If the pH is not ≤ 2 , add 3 ml of 1:1 sulfuric acid (H_2SO_4), recap the bottle firmly and invert the bottle 5 times.
12. Place a pH indicator test strip on a dry opened paper towel. Remove the screw-on cap and obtain an aliquot of the sample using a glass pipette. Moisten the pH indicator test strip with the aliquot from the glass pipette and immediately flick the pH indicator test strip once using a sharp wrist motion to shake off the excess water. Compare the strip with the reference pH range. A determination must be made within 30 seconds.
13. If the pH is ≤ 2 , recap the sample bottle firmly, dry, attach the sample/laboratory label to the bottle and secure the chain of custody seal around the cap. Record the results in field notebook and place the sample bottle in the ice chest to cool to 4°C .
14. Continue the process of adding acid to the sample, recapping, mixing, and testing until the pH of the sample reaches a pH of ≤ 2 . Remember to note the amount of acid added to the water sample in the field notebook.

VI. SAMPLE TRANSPORT:

Place the sample bottle(s) into the ice chest for transport. The samples must be chilled and preserved at a temperature of 4°C and maintained at that temperature until analysis. Always use chopped, grated, or dry ice when chilling the samples for transportation. Never use “blue ice” as the samples may not chill adequately.

Field samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4°C upon arrival at the laboratory. Samples that cannot be acid preserved at the time of collection because of sampling limitations or transport restrictions should be acidified with sulfuric acid (H_2SO_4) to a pH ≤ 2 upon receipt in the laboratory and allowed to sit of sixteen hours. This waiting period is to allow any inorganic metals to return back into solution in the event they may have precipitated or “plated out” along the inside of the sample container as a result of inadequate acidification at the time of collection.

VII. SAMPLE STORAGE:

Store samples at 4°C until analysis.

VIII. DEFINITIONS:

- A. *Ion chromatography (IC):* Ion chromatography is a technique for sequential determination of anions or cations using ion exchange and conductivity, amperometric, or colorimetric detectors.

IX. SAFETY:

The use of protective eyewear and laboratory quality latex gloves is highly recommended when collecting and preserving samples.

X. SUMMARY OF METHOD:

METHOD 300.0-- A water sample is injected into a stream of carbonate-bicarbonate eluent and passed through a series of ion exchangers. The anions of interest are separated on the basis of their relative affinities for a low capacity, strongly basic anion exchanger (guard and separator columns). The separated anions are directed through a hollow fiber cation exchanger membrane (fiber suppressor) or micromembrane suppressor bathed in a continuously flowing strongly acidic solution (regenerant solution). In the suppressor, the separated anions are converted to their highly conductive acid forms and the carbonate-bicarbonate eluent is converted to a weakly conductive carbonic acid. The separated anions in their acid forms are measured by conductivity. They are identified on the basis of retention time as compared to standards. Quantitation is by measurement of peak area or peak height.