Technical Standards

ELTAC Meeting July 27, 2016

The Method

METHOD 300.1

DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY

Revision 1.0

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1. SCOPE AND APPLICATION

PART A.-- Common Anions

Bromide	Nitrite
Chloride	ortho-Phosphate-P
Fluoride	Sulfate
Nitrate	

PART B.-- Inorganic Disinfection By-products

Bromate	Chlorite
Bromide	Chlorate

1) These are the only analytes that can be reported by the method for regulatory compliance Monitoring
2) Injection Volume must be matched to analyte

2. SUMMARY OF METHOD

2.1 A small volume of sample, 10 uL for Part A and 50 uL for Part B, is introduced into an ion chromatograph. The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

2.2 The ONLY difference between Parts A and B is the volume of sample analyzed by the ion chromatographic system. The separator columns and guard columns as well as eluent conditions are identical.

USEPA Method 300.1 Equipment

6.1.1 Anion guard column: Dionex AG9-HC, 2 mm (P/N 52248), or equivalent. This column functions as a protector of the separator column. If omitted from the system the retention times will be shorter.

6.1.2 Anion separator column: Dionex AS9-HC column, 2 mm (P/N 52244), or equivalent. The microbore (2 mm) was selected in the development of this method as a means to tighten the bromate elution band and thus reduce the detection limit. An optional column (2 mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the requirements of Sect. 9.0 can be met. The AS9-HC, 2 mm column using the conditions outlined in Table 1A and 1B produced the separation shown in Figures 1 through 4.

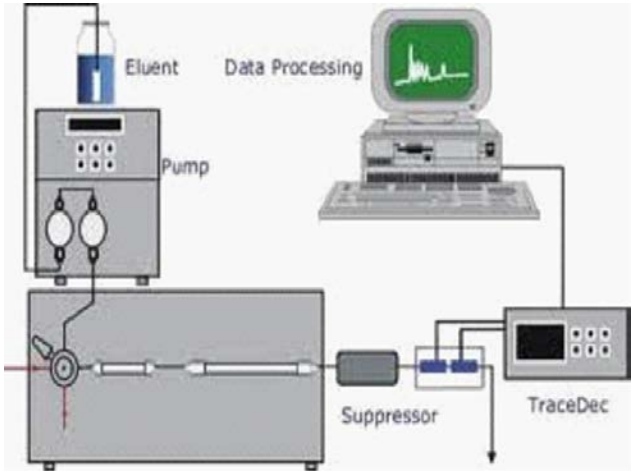
6.1.3 Anion suppressor device: The data presented in this method were generated using a Dionex Anion Self Regenerating Suppressor (ASRS, P/N 43187). An equivalent suppressor device may be utilized provided comparable detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity.

6.1.4 Detector -- Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in Sect. 9.2.

6.2 The Dionex Peaknet Data Chromatography Software was used to generate all th data in the attached tables. Systems using a strip chart recorder and integrator o other computer based data system may achieve approximately the same MDL's but the user should demonstrate this by the procedure outlined in Sect. 9.2.

A laboratory must have:

1) AG9-HC Guard Column
2) AS9-HC Separator Column
3) Anion Suppressor
4) Conductivity Cell
5) Chromatography Software



USEPA Method 300.1 Reagents & Stds

7.1 Reagent water: Distilled or deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

7.2 Eluent solution : Sodium carbonate (CASRN 497-19-8) 9.0 mM. Dissolve 1.91 g sodium carbonate (Na_2CO_3) in reagent water and dilute to 2 L.

7.3 Stock standard solutions, 1000 mg/L (1 mg/mL): Stock standard solutions may be purchased as certified solutions or prepared from ACS reagent grade, potassium or sodium salts as listed below, for most analytes. Chlorite requires careful consideration as outline below in 7.3.5.1.

7.4 Ethylenediamine (EDA) preservation solution, 100 mg/mL: Dilute 2.8 mL of ethylenediamine (99%) (CASRN 107-15-3) to 25 mL with reagent water. Prepare fresh monthly.

7.5 Surrogate Solution: 0.50 mg/mL dichloroacetate (DCA) prepared by dissolving 0.065 g dichloroacetic acid, potassium salt (Cl_2CHCO_2K, CASRN 19559-59-2) in reagent water and dilute to 100 mL in a volumetric flask.

A laboratory must have:

1) Reagent Water
2) Eluent Solution
3) Stock Standard Solutions
4) EDA
5) Surrogate Solution

USEPA Method 300.1 Sampling

8.1 Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

8.3 Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A : Common Anions

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	Cool to 4°C	48 hours
Nitrite-N	Cool to 4°C	48 hours
ortho-Phosphate-P	Cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days

PART B : Inorganic Disinfection By-products

Analyte	Preservation	Holding Time
Bromate	50 mg/L EDA	28 days
Bromide	None required	28 days
Chlorate	50 mg/L EDA	28 days
Chlorite	50 mg/L EDA, Cool to 4°C	14 days

A laboratory must:

1) Use Glass or Plastic Bottles
2) Rinse and Clean
3) Use Preservative
4) Follow Holding Times

Technical Standard

- The above requirements are only found in the individual methods
- None of these requirements are not found in the LCM, TNI 2003, TNI 2009, ISO 17025
- For the last 60 years California has been using requirements like these to accredit laboratories
- These requirements have to be followed with or without a Quality System
- Right now these are the only Technical Standards

What Could be in a Technical Standard

(e) If an analytical method does not have quality control procedures, a laboratory shall use the following:

(1) For UoAs in FoAs 101, 107, and 108, Standard Methods for the Examination of Water and Wastewater, 20th edition (1998), or Standard Methods Online (), April 2004; www.standardmethods.org, Sections 9020, 9030, 9040, and 9050. For UoAs in FoA 101, Section 9060 B is also required;

(2) For UoAs in FoA 102 and 109, Standard Methods for the Examination of Water and Wastewater, 20th edition (1998), or Standard Methods Online (),April 2004;www.standardmethods.org, Sections 2020 for general physical and aggregate property tests, 3020 for non-spectroscopy method for elements, 4020 non-ion chromatography methods for inorganic analytes, and 5020 for non-specific organic substances;

(3) For UoAs in FoA 103 and 110, Standard Methods for the Examination of Water and Wastewater, 20th edition (1998), or Standard Methods Online (), April 2004; www.standardmethods.org, Section 3020 for spectroscopy methods and 4020 for ion chromatography methods.

(4) For UoAs in FoA 104 and 111, Standard Methods for the Examination of Water and Wastewater, 20th edition (1998), or Standard Methods Online (), April 2004; www.standardmethods.org, Section 6020.

(5) For UoAs in FoA 105 and 112, Standard Methods for the Examination of Water and Wastewater20th edition (1998), or Standard Methods Online (), April 2004; www.standardmethods.org, Section 7020.

(6) For UoAs in FoA 113, Standard Methods for the Examination of Water and Wastewater, 20th edition (1998), or Standard Methods Online (), April 2004; www.standardmethods.org, Section 8010 and 8020.

(7) For UoAs in FoAs 115 through 121, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846) U.S.E.P.A., third edition; and

(8) For UoAs in FoAs 122 through 124, the quality control procedures specified by the Department of Food and Agriculture, on the basis of the intended use of the analytical data.

Article 6 Required Test Methods.

Section 64811.

(a) Any laboratory requesting ELAP or NELAP accreditation from the State Board / ELAP for Units of Accreditation in Fields of Accreditation 101 through 106 as identified in 100860.1 or 100862, shall employ those methods identified in H&SC 100852 or as identified by the Division of Drinking Water for regulatory compliance purposes. If a Public Water System has a permit issued by the Division of Drinking Water which requires that Public Water System to use a test method for a specific analyte that had once been listed in the Code of Federal Regulation Title 40 Part 141 but is no longer so listed, a laboratory may seek accreditation for that test method and analyte combination but may only use that combination for samples from that Public Water System. If the permit is updated by the Division of Drinking Water and that requirement to use that method analyte combination is removed, the accreditation for the laboratory shall be revoked.

(b) Any laboratory requesting ELAP or NELAP accreditation from the State Board / ELAP for Units of Accreditation in Fields of Accreditation 107 through 113 as identified in 100860.1 or 100862, shall employ those methods identified in H&SC 100852 or as identified by the State Water Resource Control Board or a Regional Water Quality Control Board for regulatory compliance purposes. If a National Pollutant Discharge Elimination System (NPDES) permittee or a Waste Discharge Requirement (WDR) holder or other permit issued by the SWRCB or RWQCB which requires that permittee to use a test method for a specific analyte that had once been listed in the Code of Federal Regulation Title 40 Part 136 but is no longer so listed, a laboratory may seek accreditation for that test method and analyte combination but may only use that combination for samples from that permittee. If the permit is updated by the SWRCB or RWQCB and that requirement to use that method analyte combination is removed, the accreditation for the laboratory shall be revoked.

(c) Any laboratory requesting ELAP or NELAP accreditation from the State Board / ELAP for Units of Accreditation in Fields of Accreditation 114 through 121 as identified in 100860.1 or 100862, shall employ those methods identified in 22 CCR § 66261.24 or as identified by the Department of Toxic Substance Control for regulatory compliance purposes.

Technical Standard

Are the Methods required in Regulation to used for Compliance Monitoring