Preparing samples and standards for anion analysis

Analytical information

The Dionex AS19 column has a stationary phase containing positively charged alkanol quaternary ammonium functional groups and uses a dilute KOH eluant. The eluant is made by the eluant generator, usually using a concentration gradient program that gradually increases the eluant concentration as the run progresses. This system can separate a wide variety of anions, including some low molecular weight organic anions such as acetate, formate, and oxylate. The procedure here is for nine inorganic anions in natural and tap waters, plus some organic acids. Other ions such as iodide and a wide variety oxyanions can also be analyzed with modified procedures. Read the literature and be sure to run tests.

Sample Preparation

All samples should be kept refrigerated and in the dark until they are analyzed, which, in general, should be done as quickly as possible. Samples must be free of high molecular weight organics (tannins, humic acids, hydrocarbons, etc.), high concentrations of transition metals, and particulates. Samples must be between pH 3 and 11.

All natural samples need to be filtered for particulates, preferably through a 0.2 µm filter. Some samples also have to be filtered to remove high-molecular weight organics or transition metals (e.g., iron). High molecular weight organics and transition metals will damage the column, and particulate damage is irreversible.

Sample filtration requirements.

Sample content	0.2 µm particle filter $2.00 each*	OnGuard P filter $4.50 each**	OnGuard H filter $4.50 each***
Normal, colorless, dilute samples.	Yes	No	No
Brown samples having dissolved tannins or other high molecular weight organic compounds.	Yes	Yes	No
Samples rich in transition metals (e.g., iron).	Yes	No	Yes
Brown samples rich in organics *and* rich in transition metals.	Yes	Yes	Yes
* Unless already filtered, in the field, for example.			
** Removes high molecular weight organics.			
*** Removes transition metals and calcium. Solutions become acidic and may need to have CO_2 degassed.			

Follow the instructions with the filters. Filters can be stacked on the end of the syringe and filtered in one step, with the particulate filter being the last. The filters are expensive so don't waste them.

Standard preparation

In general, standards should be similar in composition to the samples being analyzed. Because samples vary enormously, you may want to design your own. For many natural waters the following set of standards is a good place to start. It has 4 standards of inorganic components and 1 organic acid standard.

Standard 1, other standards are diluted from this.

Component	Stock concentrate, ppm	ml added to a 250 ml volumetric flask	Final concentration in 250 ml volumetric flask	Units
NO_2^-	1000	0.25	1	ppm
Br^-	1000	0.25	1	ppm
ClO_3^-	1000	0.25	1	ppm
ClO_2^-	1000	0.25	1	ppm
F^-	1000	0.5	2	ppm
PO_4^{3-}	1000	0.5	2	ppm
NO_3^-	1000	5	20	ppm
Cl^-	1000	25	100	ppm
SO_4^{2-}	1000	25	100	ppm

Prepare the organic acid standard separately. The diluted organic standard doesn't last very long. I think bacteria start working on it.

Organics standard, not diluted any more.

Component	Stock concentrate, ppm	ml added to a 100 ml volumetric flask	Final concentration in 100 ml volumetric flask	Units
Acetate	1000	0.1	1	ppm
Formate	1000	0.1	1	ppm
Oxalate	1000	0.1	1	ppm

Transfer **Standard 1** to a clean 250 ml plastic bottle. Transfer appropriate numbers of 13.9 ml aliquots of DI water and **Standard 1** to three other 250 ml bottles, as follows (we have an adjustable 20 ml pipette):

Diluting to make the other standards.

	Standard 1	Standard 2	Standard 3	Standard 4
DI water, aliquots	-	5	8	9
Standard 1, aliquots	-	5	2	1
NO_2^-	1	0.5	0.2	0.1
Br^-	1	0.5	0.2	0.1
ClO_3^-	1	0.5	0.2	0.1
ClO_2^-	1	0.5	0.2	0.1
F^-	2	1	0.4	0.2
PO_4^{3-}	2	1	0.4	0.2
NO_3^-	20	10	4	2
Cl^-	100	50	20	10
SO_4^{2-}	100	50	20	10
Alliquots are 13.9 ml. 139 ml of each are left at the end.				

Pour ~5 ml of each standard into Dionex autosampler tubes, and press a black filter cap down into the top of each using the filter cap tool. You should run standards at least at the beginning of the run and perhaps also within the run. Alternatively, you can periodically run a check sample and do corrections off line.