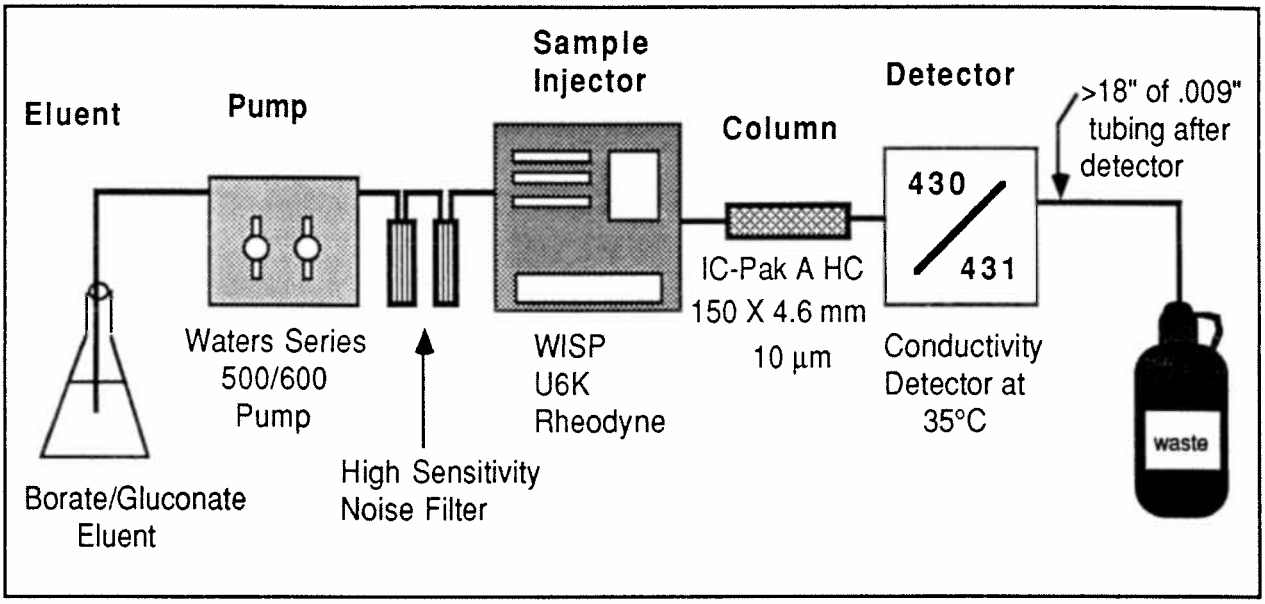
WATERS ION ANALYSIS METHOD

Anion Analysis Using IC-Pak A HC Column Borate/Gluconate Eluent

Method # A-102

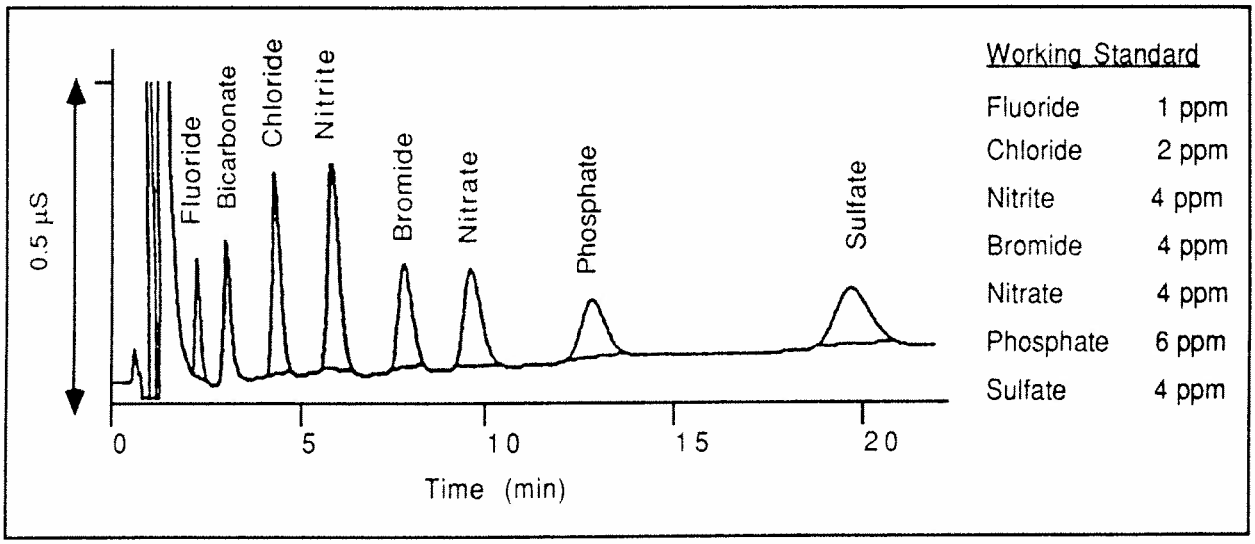
INSTRUMENTATION



CONDITIONS

Eluent: Borate/Gluconate
Pump: 590 Solvent Delivery Module
Injector: 710B WISP
Column: IC-Pak A HC
Data: 840 Data System
Flow rate: 2.0 ml/min
Injection: 100 µl of Standard Anion Mix

Detection: 430 Conductivity
Range: 500 µS
Temperature: On
Polarity: +
Background: 274 µS



ELUENT PREPARATION

I. Borate/Gluconate Concentrate

To a one liter volumetric flask add:

16 g Sodium Gluconate
18 g Boric Acid
25 g Sodium Tetraborate Decahydrate

Add approximately 500 ml of Milli-Q water and mix thoroughly until dissolved, then add:

250 ml Glycerin

Fill the flask to the mark with Milli-Q water and mix thoroughly. Concentrate may be stored refrigerated for up to six months before replacement.

II. Borate/Gluconate Eluent (pH 8.5)

Place approximately 500 ml of Milli-Q water into a one liter volumetric flask and add:

20 ml Borate/Gluconate Concentrate
20 ml n-Butanol
120 ml Acetonitrile

Fill the flask to the mark with Milli-Q water and mix thoroughly. Filter through a 0.22 µm Durapore membrane (GVWP).

STANDARD PREPARATION

I. Concentrated Standard

1000 ppm F^-	0.221 g NaF (ACS) +/- 0.001 g
2000 ppm Cl^-	0.329 g NaCl (ACS) +/- 0.001 g
4000 ppm NO_2^-	0.600 g $NaNO_2$ (ACS) +/- 0.001 g
4000 ppm Br^-	0.596 g KBr (ACS) +/- 0.001 g
4000 ppm NO_3^-	0.548 g $NaNO_3$ (ACS) +/- 0.001 g
6000 ppm HPO_4^{2-}	0.854 g KH_2PO_4 (ACS) +/- 0.001 g
4000 ppm SO_4^{2-}	0.592 g Na_2SO_4 (ACS) +/- 0.001 g

Prepare separate standard concentrates by diluting each of the above to 100 ml with Milli-Q water

II. Working Standard

Pipet 100 µl of each standard concentrate into a 100-ml volumetric flask and dilute with Milli-Q water to result in the working standard (see front page). Prepare fresh working standard weekly.

DETECTION LIMITS:
3 x Baseline Noise (500 µl)

Analyte	PPB
F^-	20
Cl^-	10
NO_2^-	20
Br^-	30
NO_3^-	20
HPO_4^{2-}	75
SO_4^{2-}	50

COMMENTS:

1. For samples with low levels of analytes, injection volumes of 500 µl may be used.

2. For specific sample preparation methods, see Section 1.

3. When analyzing for anions in drinking water and other environmental samples, the presence of high levels of carbonate, interfering metals (such as magnesium and calcium), and excess base are often encountered. In an untreated sample, these may cause disturbances in the chromatographic baseline. The presence of high levels of carbonate will result in a large carbonate peak which may interfere with the quantitation of fluoride and chloride. In extreme cases carbonate may overload the column and cause loss of early eluting analytes. Alkaline earth metals , such as magnesium and calcium, may cause severe interferences in the early part of the chromatogram.

When using a borate/gluconate eluent, high pH samples may result in a pH system peak in the middle portion of the chromatogram which can interfere with nitrate and bromide analysis. In many of these samples, the analyte levels are too low for dilution of the sample to be practical. Dilution would cause analyte levels to drop below the detection limits for direct injection.

The best method for analyzing samples of this type is to pretreat the sample with a MilliTrap™ H^+ Membrane Cartridge. The MilliTrap™ H^+ cartridge is a hand held, multiple use, disposable device that removes interferences from many matrices such as drinking, surface or waste water samples. The diagram on pages 7 & 8 of Section 1 illustrates and explains how the MilliTrap™ H+ cartridge functions to remove matrix interferences.

When using the MilliTrap™ H+ Membrane Cartridge, the eluent described below is recommended. Using the MilliTrap™ cartridge with this eluent will give superior results for both high pH and low pH samples.

Concentrate

To a 1-liter volumetric flask add:
500 ml Milli-Q® water
7.2 g Lithium Hydroxide Monohydrate
Mix until dissolved, then add:
25.5 g Boric Acid
Mix until dissolved, then add:
13.2 ml Gluconic Acid (50% wt solution)
94 ml Glycerin (95%)
Finally, add Milli-Q® water to the mark and mix thoroughly.

Eluent

To a 1-liter volumetric flask add:
20 ml of above concentrate
120 ml Acetonitrile
Fill to the mark with Milli-Q® water, mix thoroughly, and filter through a 0.22 μ Millipore membrane (GVWP). Background conductivity = 220 μS.
