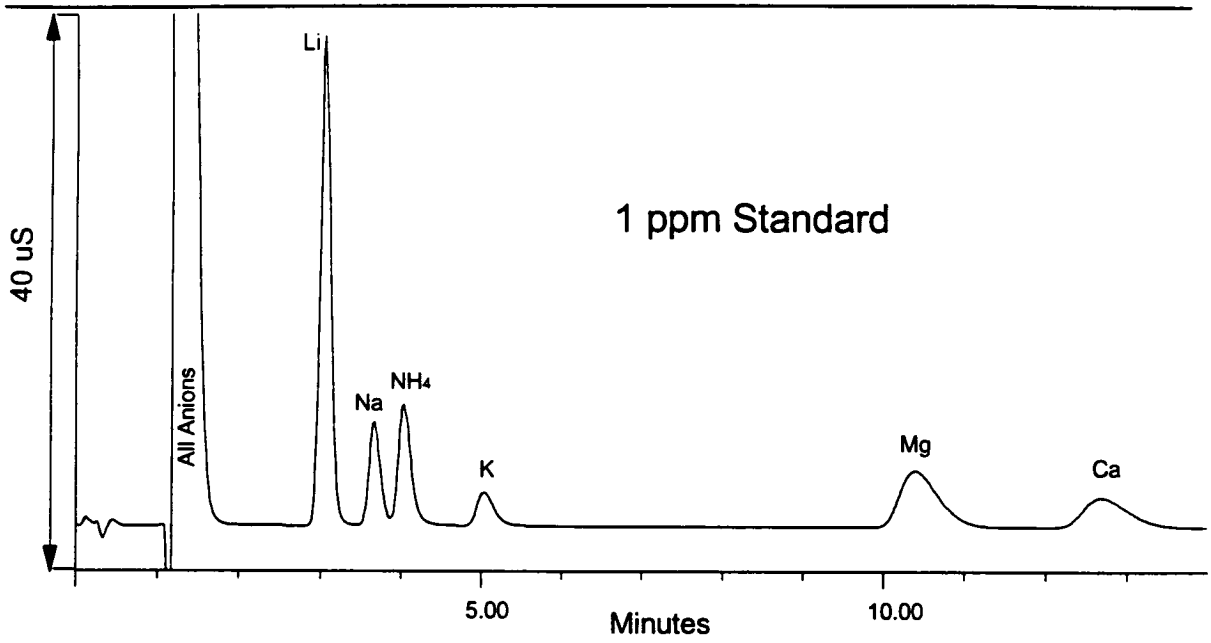
980898

Waters

Ion Chromatography Method

Alkali and Alkaline Earth Cations, Ammonium and Amines 2000

Required Instrumentation:	Part / Number
Alliance, 2690 Separations Module	
(with Column Heater, Seal Wash, Degasser)	271013
432 Conductivity Detector	043061
Bus SAT/IN Module	073645
Millennium 32	Consult Waters



Analysis Conditions:

Column:	IC-Pak Cation M/D
Eluent:	3 mM HNO_3 / 0.1 mM EDTA
Back Conductivity:	1250 ± 50 µS
Degas:	Continuous
Flow Rate:	1 mL / min
Back Pressure:	2100 psi
Temperature:	30°C (Column Heater); 35°C (Detector)
Injection:	100 µL
Needle Wash:	12% AcCN in DI Water
Detection:	Indirect Conductivity
Base Range:	2000
Attenuation:	100 µS / Volt Unattenuated
Polarity:	Negative

Eluent Preparation:

1) Into a 1liter plastic volumetric flask, add
   -0.029 g of EDTA, free acid.
2) Dissolve in 500 mL of DI Water with the aid of a stirring bar.
3) Add 30 mL of 100 mM HNO_3.
4) Dilute to volume with DI Water.
5) Vacuum degas through a 0.45 µm aqueous compatible membrane.
6) Store in a plastic container at ambient temperature. Discard after 1 month.

Standard Preparation:

It is recommended that certified 1000 ppm anion standards be used with this method. If unavailable, see Reagent Section for uncertified standard preparation.

Prepare at least 3 mixed analyte standards, using plastic volumetric flasks, within the expected range of the sample analyte concentration. This method is linear from 0.05 ppm to 20 ppm for lithium, sodium, and ammonium, and 0.05 ppm to 50 ppm for potassium, magnesium, and calcium. Above these concentrations the response is off scale. After the multi-point calibration curve has been validated, a single point calibration within the expected analyte concentration is appropriate for future calibrations.

This method can be used for the analysis of Rb, Cs, Sr, and Ba.

Sample Preparation:

Determine the expected range of analyte concentration and other anionic component in the sample matrix. Sodium should be less than 20 ppm for best results.

If necessary dilute the sample with DI Water.

If the sample contains high amounts of neutral organics, or is highly colored, then pass the diluted sample through a C_{18} Sep-Pak Cartridge. Cations pass through unretained. There may be residual Na contamination from the cartridge.

Samples containing suspended solids should be filtered through a 0.45 µm aqueous compatible disk prior to injection. Failure to filter solids results in the risk of increased column backpressure.

Sample pH should be within 2 to 7 for best results, especially the alkaline earth cations. Samples with pH less than 10 is appropriate for the alkali cations, ammonium, and amines.

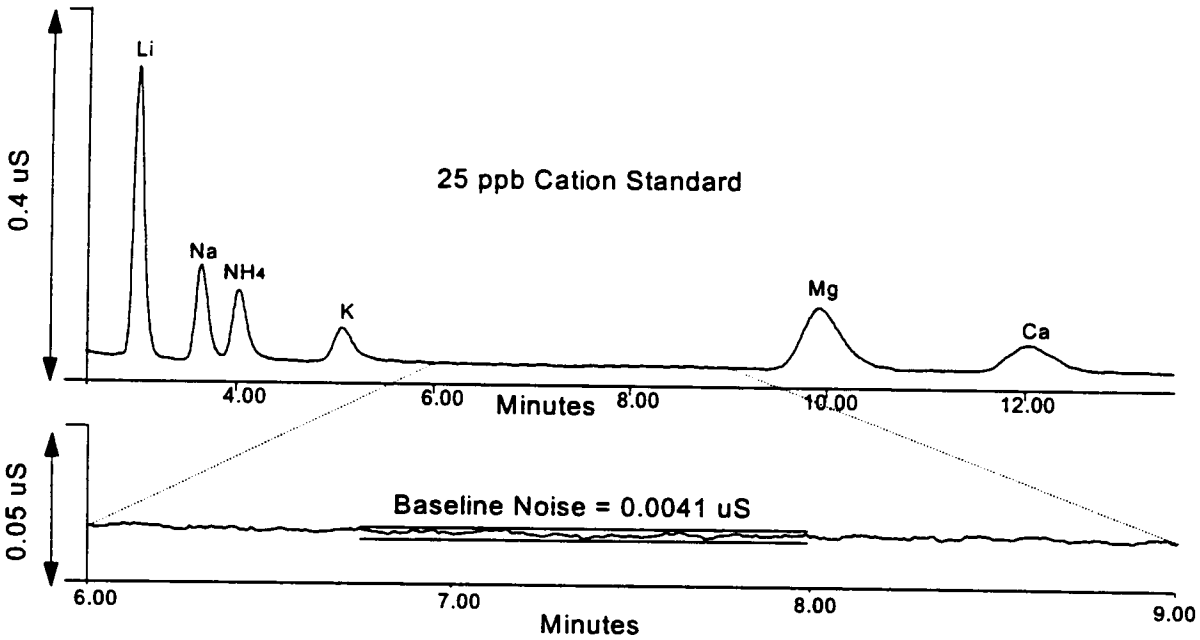
For sample with pH less than 2, dilute the sample 1:10 with DI Water; or treat the sample with an Alltech Maxi-Clean ™IC-OH Cartridge to remove anions and neutralize pH.

Millennium Data Processing Method:

IC Processing Method using Peal Apex for Retention Time

Integration	Peak Width	= 30.0	Threshold = 25--40
	Min Area	= 3000	Min Height =500
	Inhibit Intg.	= 0 to 2 min	
Calibration	Averaging	= None	RT Window = 5%
	Update RT	= Never	
	Peak Match	= Closest	
	Quant By	= Peak Area	
	Fit Type	= Linear, for multi-point calibration or Linear Through Zero, for single point	
Report	Analyte Name		
	Analyte Retention Time		
	Peak Area		
	Amounts		

Method Detection Limits:

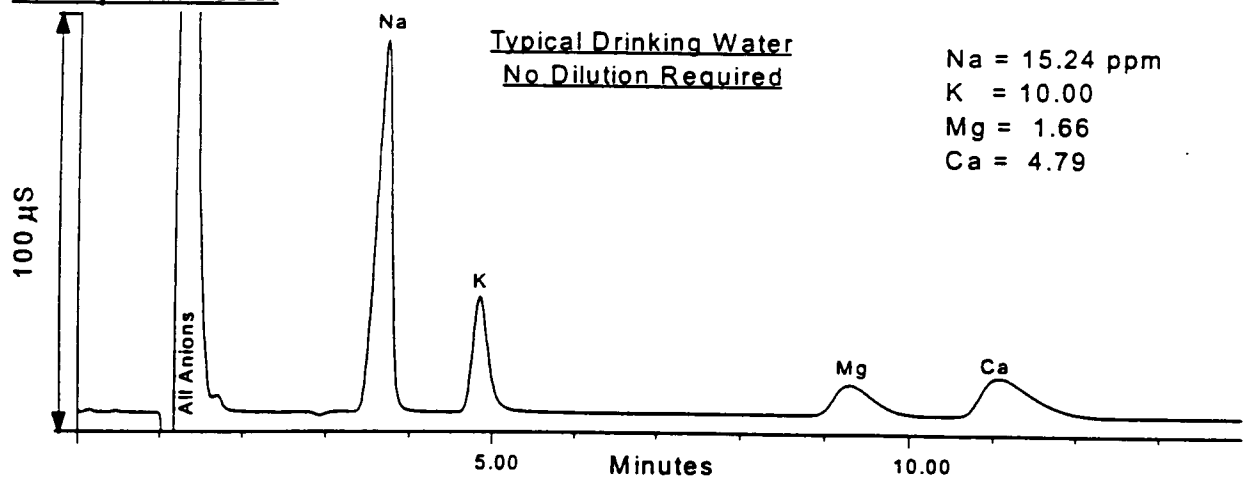


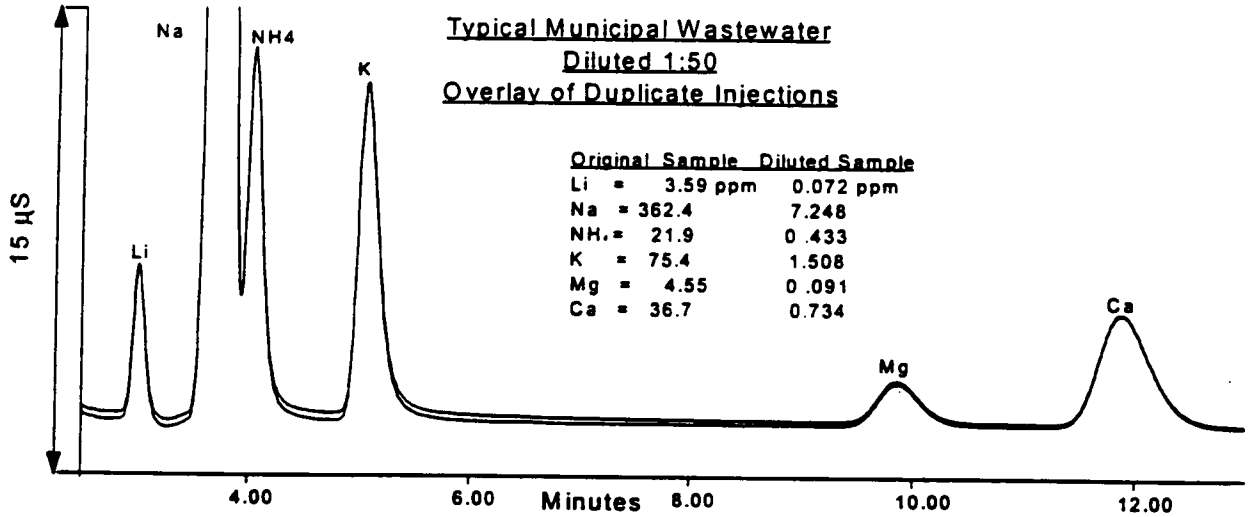
Based upon this representative chromatogram using a 100 µL injection, the estimated detection limits, as ppb, at 3 times signal to noise (S/N) are:

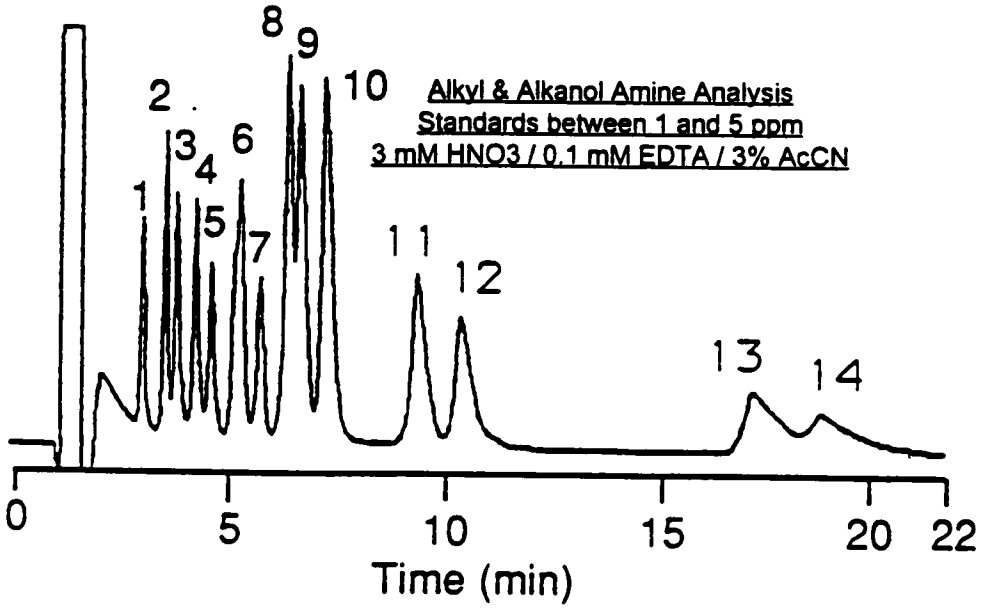
Lithium = 1	Sodium = 5	Ammonium = 5
Potassium = 15	Magnesium = 10	Calcium = 15

Lower detection limits can be achieved by using a 250 µL injection.

Examples of Use:







1 Lithium
2 Sodium
3 Ammonium
4 Methylamine
5 Ethylamine
6 Dimethylamine/ n-propylamine
7 Morpholine
8 n-Butylamine
9 Diethylamine
10 Trimethylamine
11 Cyclohexylamine
12 Dipropylamine
13 Triethylamine
14 Dibutylamine

Stock Reagent Preparation:

It is difficult to prepare a stock eluent for this column; it is best to prepare fresh working eluent.

In a 1 liter plastic volumetric flask, add 0.029 g of EDTA (as the free acid, not its salts) in 800 mL of DI water, and a stirring bar. Place on a magnetic stir plate and stir for 10 minutes.

While stirring add 30 mL of 100 mM Nitric Acid and mix for 5 minutes.

Remove the stir bar and fill to the mark with DI water.

Filter through a 0.45 μm aqueous compatible membrane filter before use. There may be some remaining white crystals on the filter(EDTA); this does not effect the performance of the eluent. Discard the filter.

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Capillary Ion Analysis Methods