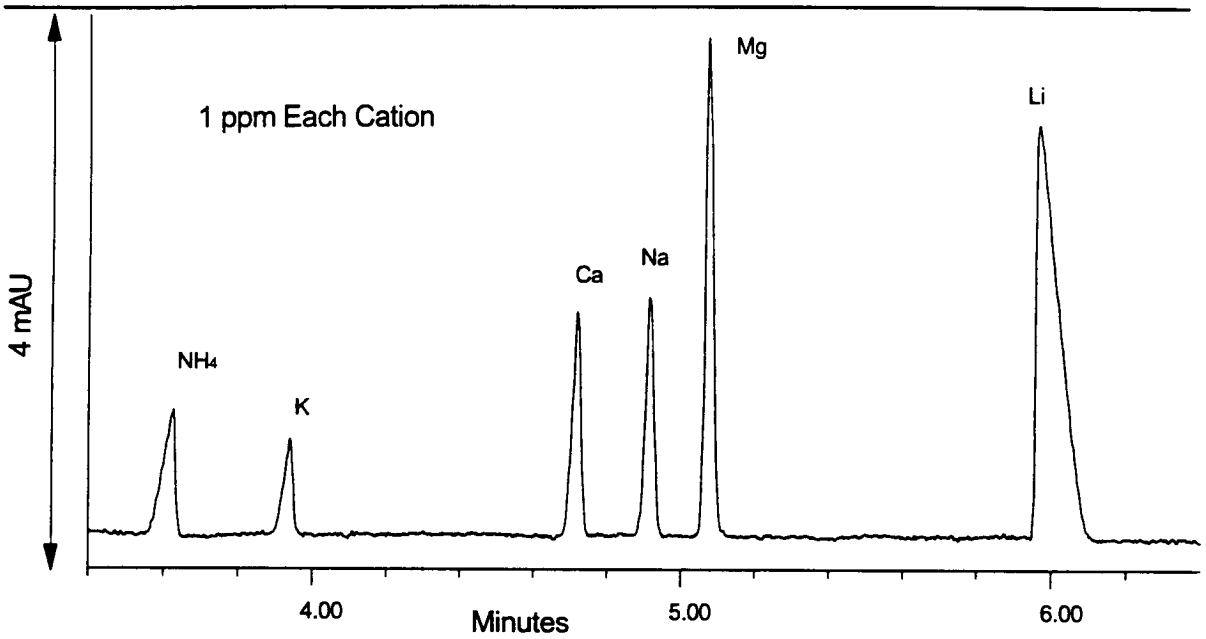
9809906

Waters

Capillary Ion Analysis Method

Alkali and Alkaline Earth Cations, Ammonium, and Amines 2000

Required Instrumentation:	Part / Number
Capillary Ion Analyzer	251000
Bus SAT/IN Module,	073645
Millennium 32	Consult Waters



Analysis Conditions:

Electrolyte:	5 mM Imidazole / 6.5 mM HIBA / 2 mM 18-C-6-E, pH 4.4
Capillary:	75 µm (id) x 375 µm (od) x 60 cm (length)
Temperature:	25°C (5°C Above Ambient)
Power Supply:	Positive
Voltage:	20 kV (Use Constant Voltage for Analysis)
Current:	6 ± 1 µA
Sampling:	Hydrostatic for 30 Seconds
Detection:	Indirect UV at 214 nm & Zn lamp; or 185 nm & Hg lamp
Time Constant:	0.3 Seconds, or less
Sampling Rate:	20 Data Point per Second
Analyte MT:	Mid-Point of Analyte Peak Width at Baseline
Quantitation:	Time Corrected Peak Area (Peak Area / MT)

Electrolyte Preparation:
This electrolyte can be prepared as follows: See Reagents Section for stock reagent preparation.

1) Into a plastic 100 mL volumetric flask add
   -10 mL of 50 mM Imidazole Solution
   -10 mL of 65 mM HIBA Solution
   -10 mL of 20 mM 18-Crown-6-Ether Solution
   -Dilute to volume with DI water.
2) Natural pH is 4.4 ± 0.1.
3) Vacuum degas through 0.45 µm aqueous compatible membrane. Disregard any remain brown residue on the filter.
4) Store any unused electrolyte in a plastic container at ambient temperature.
5) Allow to thermally equilibrate in the CIA Analyzer for 15 minutes before use.
6) Use fresh electrolyte daily; recalibrate with every change in electrolyte.

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.

The test mix available in the IonSelect Low Mobility Cation Electrolyte Kit should be used to evaluate electrolyte selectivity only. Do not use for accurate quantitation.

Prepare at least 3 mixed analyte standards within the expected range of sample analyte concentration. After the multi-point calibration curve has been validated, a single point calibration within the expected analyte concentration range is appropriate for future calibration.

Analyze in triplicate and evaluate resulting pherograms for analyte migration time reproducibility.

Sample Preparation:
Determine the expected range of analyte concentration and other anionic components in the sample matrix. The major analyte should be less than 100 ppm for best results.

If necessary, dilute the sample with DI water only. Analyte migration time reproducibility and peak shape improves with increased sample dilution.

Initially analyze in triplicate and evaluate resulting pherograms for analyte migration time reproducibility. Significant migration time change amongst the replicates indicates a change in EOF due to sample matrix effects at the capillary wall. Consider a 500 mM HCl capillary rinse between samplings. If migration time reproducibility is less than 1% then no rinse is needed and the sample can be run in duplicate to ensure reproducibility.

CIA Analyzer Special Function Programming: Use to program the CIA Analyzer as a stand alone system, without Millennium control. Use the same programming for the Millennium Instrument Method.

SF#	and Description	Value
55	CIA	1 = CIA (Default)
88	Custom IMT	0, Enter 99 to activate SF# 58-60
89	SPS	0 = Constant Voltage (Enter in Sample Voltage)
90	C2C Mapping	0 = Off (Place electrolyte in position 1 only)
91	t°C Temperature	25 (set 5°C above ambient temperature)
92	r19, Rinse Vial	0 = Off, Enter Rinse Time in Minutes
93	r20, Rinse Vial	0 = Off, Enter Rinse Time in Minutes
94	Adjust Voltage	20 (in kV, use for manual voltage initiation only)
95	Carousel Type	20 = 20 Sample Position Carousel
96	Current Test	0 = Off (Must reset after power down)
97	Conductivity Test	0 = Off
98	Purge Time	1 (Time in Minutes)
99	CIA Version	3.0

Millennium Data Processing Method:

CIA Processing Method using Mid-Point of Peak Width for Migration Time

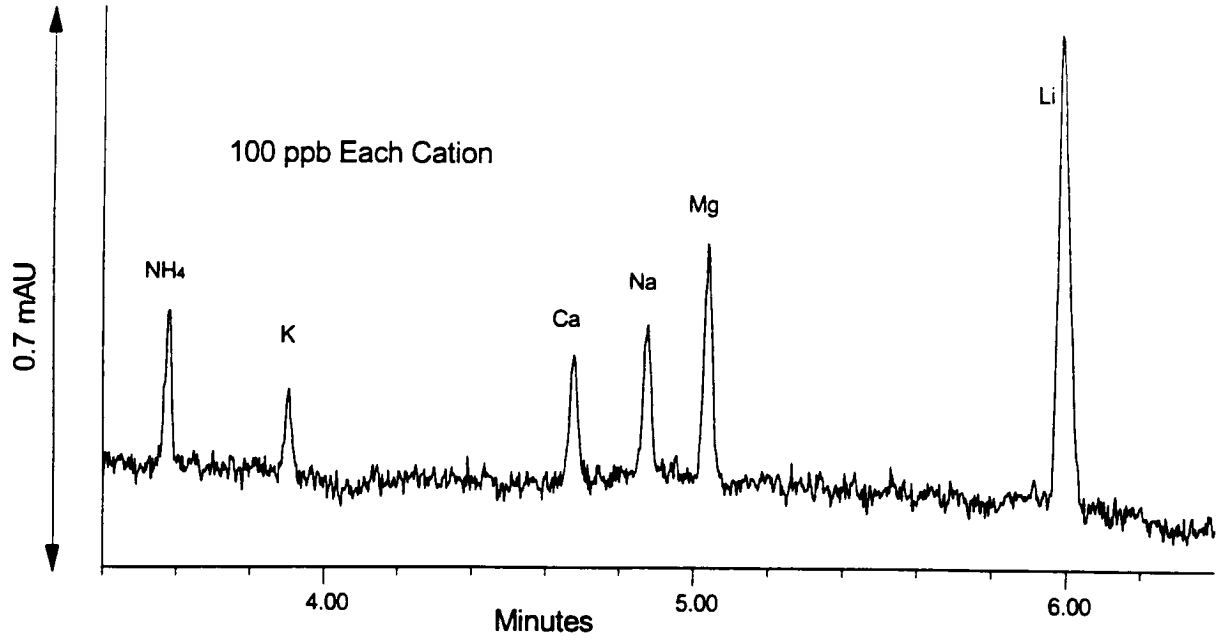
Integration
Peak Width = 2.25 - 3.00 Threshold = 100 ± 25
Min Area = 100 Min Height = 50
Inhibit Intg. = 0 to 3 min

Calibration
Averaging = None MT Window = 2%
Update MT = Average Standards
Peak Match = Closest
Quantitate By = Time Corrected Peak Area
Fit Type = Linear, for multi-point calibration, or Linear Through Zero, for single point.

Report
Analyte Name
Analyte Migration Time
Analyte Migration Time Ratio
Peak Area
Time Corrected Peak Area
Amounts

Use fresh electrolyte daily; recalibrate with every change in electrolyte.
Clear previous calibration (in QuickSet Page) before recalibration.
Do Not use analyte peak height for quantitation due to asymmetrical peak shapes.

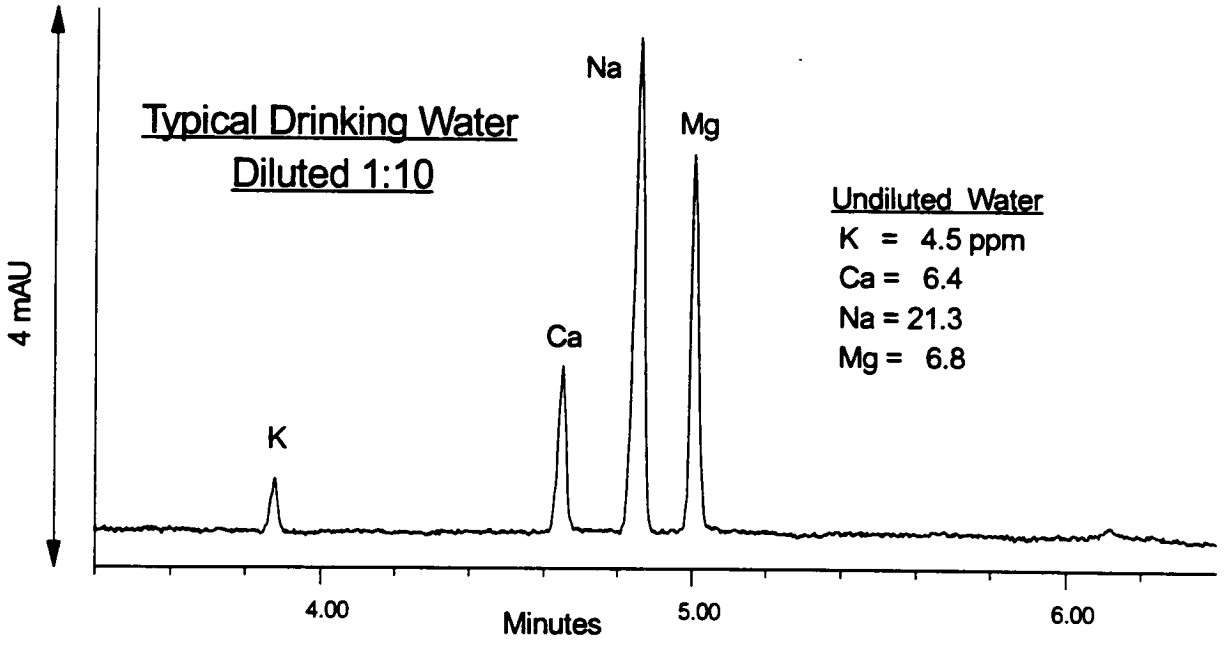
Method Detection Limits:



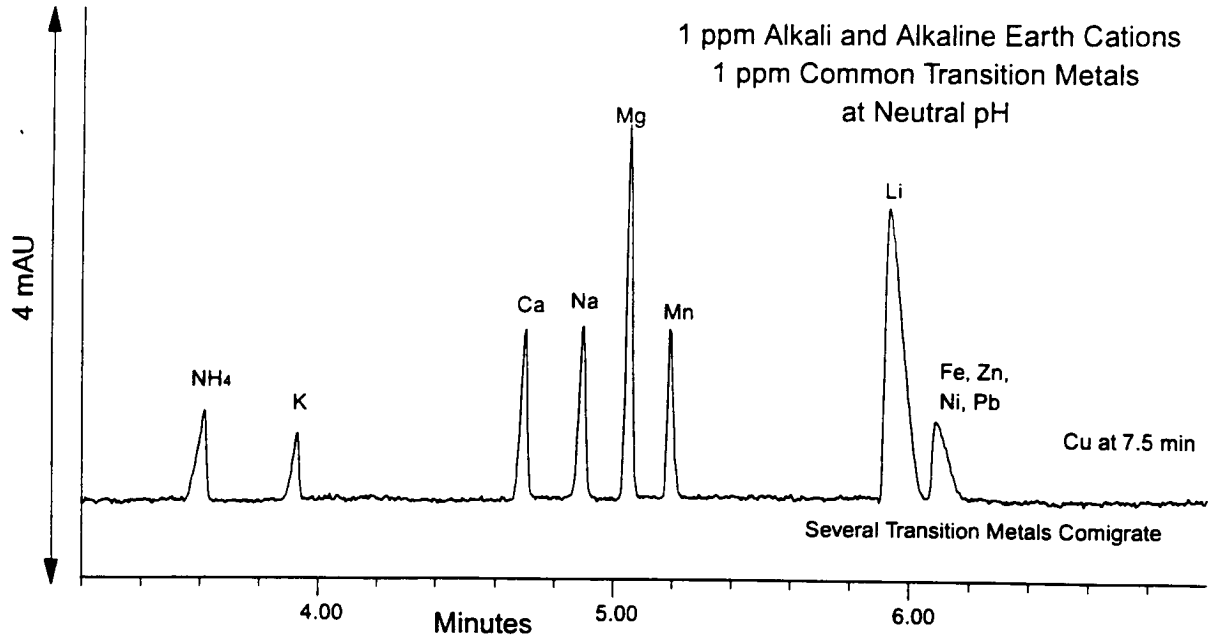
Based upon the above 100 ppb pherogram, the estimated detection limits at 3 times signal to noise (3X S/N) are:

NH_4 = 50 ppb K = 60 ppb Ca = 50 ppb Na = 45 ppb Mg = 30 ppb Li = 15 ppb.

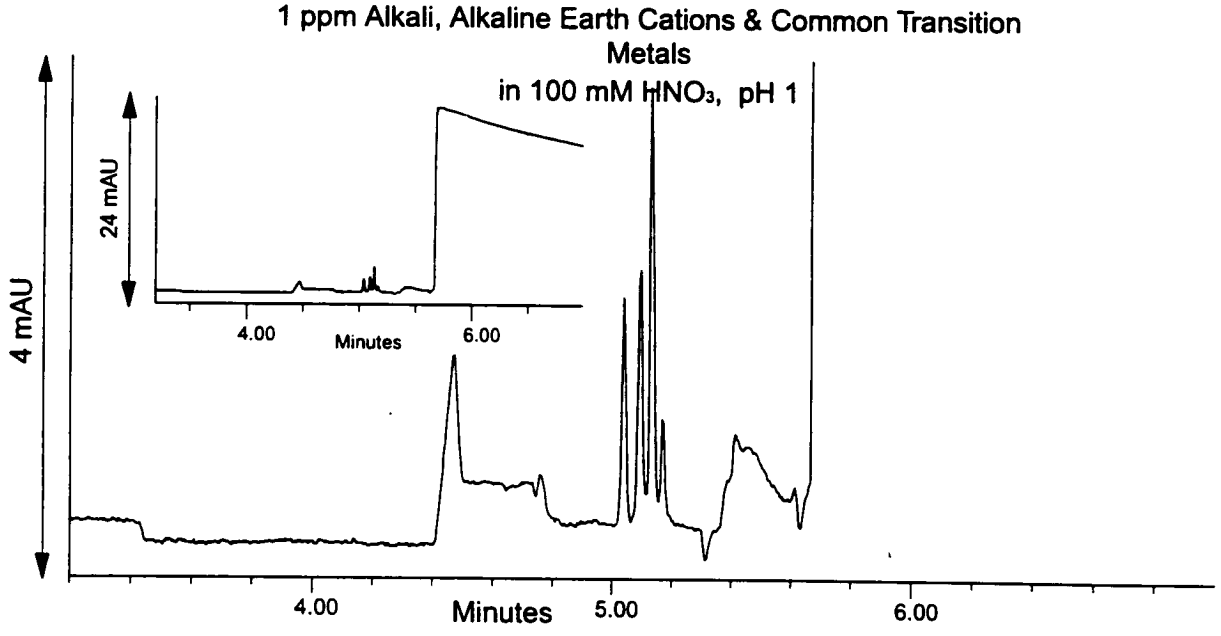
Examples of Use:



Transition Metals:



CIA requires that cations be in the free, uncomplexed cationic state for separation. In a sample matrix, transitions metals are complexed with various inorganic and/or organic acids present in the matrix. Some of these complexes are stable and are separated as the complex; it is possible to have a single transition metals in several different complex forms. This problem is eliminated when the sample is digested in nitric acid similar to the typical atomic absorption digestion. This digestion puts transition metals into their free cationic form but in high acid. The high acid effects the complexation of the transition metals with HIBA and significantly alters selectivity, as shown below.



Observation and Comments:

-This electrolyte is an alternative to Waters IonSelect Low Mobility Cation Electrolyte for the analysis of alkali, alkaline earth, ammonium, and amine cations. Imidazole has a higher mobility than IonSelect Low Mobility Cation Electrolyte, and gives more symmetrical peak shapes and increased resolution of Ca, Na, and Mg.

-This electrolyte can be used with 185 nm indirect UV detection with reduced sensitivity. It is transparent at 254 nm.

-Strontium and Barium can be analyzed with this electrolyte; Sr migrates after Mg (5.2 min) and Ba just before Li (5.8 min).

-Always use plasticware for all electrolyte, standard, and sample preparation. Glassware leaches sodium into the solution which will effect sodium quantitation.

-Ammonium is a common contaminant in DI water or from absorption from the air.

-The quality of DI water, mainly the TOC, will effect selectivity of the alkaline earth cations by competing with the HydroxyIsoButyric acid.

-Amines generally will migrate after Mg and will exhibit asymmetrical (tailing) peak shapes. Improved amine peak shapes and resolution can be obtained with the Waters Low Mobility Cation Electrolyte.

-Transition metals cannot be resolved with this electrolyte, and except for Mn transition metals migrate as a group shortly after Li. Mn migrates after Mg.

-Samples with pH less than 2 may show changes in alkaline earth selectivity.

-Samples with pH less than 2 should not be used with this method without prior sample pretreatment. Suggest pass low pH samples through an Alltech anion exchange cartridge in the hydroxide form to remove anions and replace with OH^- which then neutralizes pH.

Imidazole Electrolyte—Stock Reagent Preparation:

50 mM Imidazole Solution: Dissolve 0.340 g of Imidazole in a 100 mL plastic volumetric flask with DI water, and fill to the mark with DI water. Store this solution in a capped plastic container for up to 1 year.

65 mM Hydroxyisobutyric Acid Solution: Dissolve 0.677 g of Hydroxyisobutyric Acid (HIBA) in a 100 mL plastic volumetric flask using 75 mL of DI water, and fill to the mark with DI water. Store this solution in a capped plastic container for up to 1 year.

20 mM 18-Crown-6-Ether Solution: (Refer to reagent label for precautions)-- Dissolve 0.528 grams of 18-Crown-6-Ether in a 100 mL plastic volumetric flask, with DI water and dilute to the mark with DI water. Store in a plastic container in the refrigerator for up to 6 months. (This solution is toxic, prepare with caution using non-talc gloves.)