

Waters

Capillary Ion Analysis Method

Alkali and Alkaline Earth Cations, Ammonium, and Amines

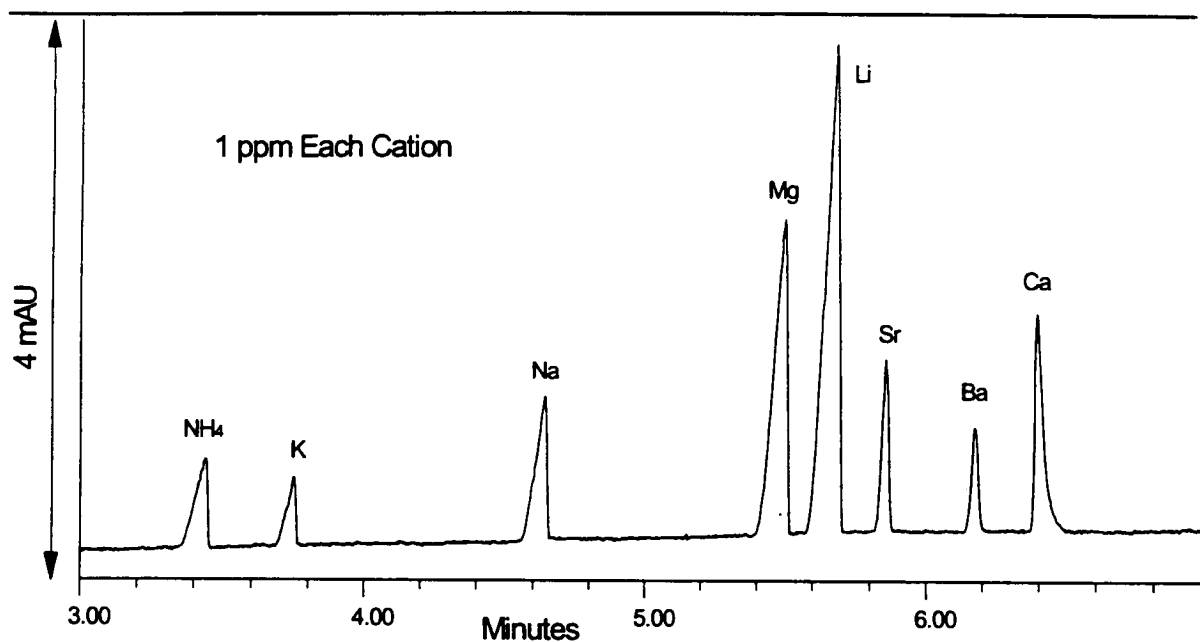
Applicable to High Sodium Low pH Samples (Experimental) 2000

Required Instrumentation:

Capillary Ion Analyzer
Bus SAT/IN Module,
Millennium 32

Part / Number

251000
073645
Consult Waters



Analysis Conditions:

Electrolyte:	6 mM Benzimidazole / 2.5 mM Tartaric Acid / 2 mM 18-Crown-6-Ether, pH 4.5
Capillary:	75 μ m (id) x 375 μ m (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 \pm 1 μ A
Sampling:	Hydrostatic for 30 Seconds
Detection:	Indirect UV at 254 nm, Hg lamp with 254 nm window
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 60 mM Benzimidazole / 25 mM Tartaric Acid Solution
 - 10 mL of 20 mM 18-Crown-6-Ether Solution
 - Dilute to volume with DI water.
- 2) Natural pH is 4.5 ± 0.1 .
- 3) Vacuum degas through 0.45 μ m 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 <u>CIA</u>	1 = CIA (Default)
88 <u>Custom IMT</u>	0, Enter 99 to activate SF# 58-60
89 <u>SPS</u>	0 = Constant Voltage (Enter in Sample Voltage)
90 <u>C2C Mapping</u>	0 = Off (Place electrolyte in position 1 only)
91 <u>t°C Temperature</u>	25 (set 5°C above ambient temperature)
92 <u>r19, Rinse Vial</u>	0 = Off, Enter Rinse Time in Minutes
93 <u>r20, Rinse Vial</u>	0 = Off, Enter Rinse Time in Minutes
94 <u>Adjust Voltage</u>	20 (in kV, use for manual voltage initiation only)
95 <u>Carousel Type</u>	20 = 20 Sample Position Carousel
96 <u>Current Test</u>	0 = Off (Must reset after power down)
97 <u>Conductivity Test</u>	0 = Off
98 <u>Purge Time</u>	1 (Time in Minutes)
99 <u>CIA Version</u>	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.

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. It is especially suited for high sodium samples. Benzimidazole has a lower mobility compared to IonSelect Low Mobility Cation Electrolyte, and gives asymmetrical (fronting) peak shapes for ammonium, potassium, sodium, magnesium, and lithium.
- 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 tartaric acid.
- Amines generally will migrate after Na.
- Transition metals cannot be resolved with this electrolyte.
- 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.

Benzimidazole Electrolyte—Stock Reagent Preparation:

60 mM Benzimidazole / 25 mM Tartaric Acid Solution— Dissolve 0.71 grams of benzimidazole, and 0.38 grams of tartaric acid, in a 100 mL plastic volumetric flask with 50 mL of DI water. With the aid of a stirring bar, mix until dissolved. Dilute to the mark with DI water.

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 at ambient temperature for up to 6 months.