980901

Waters

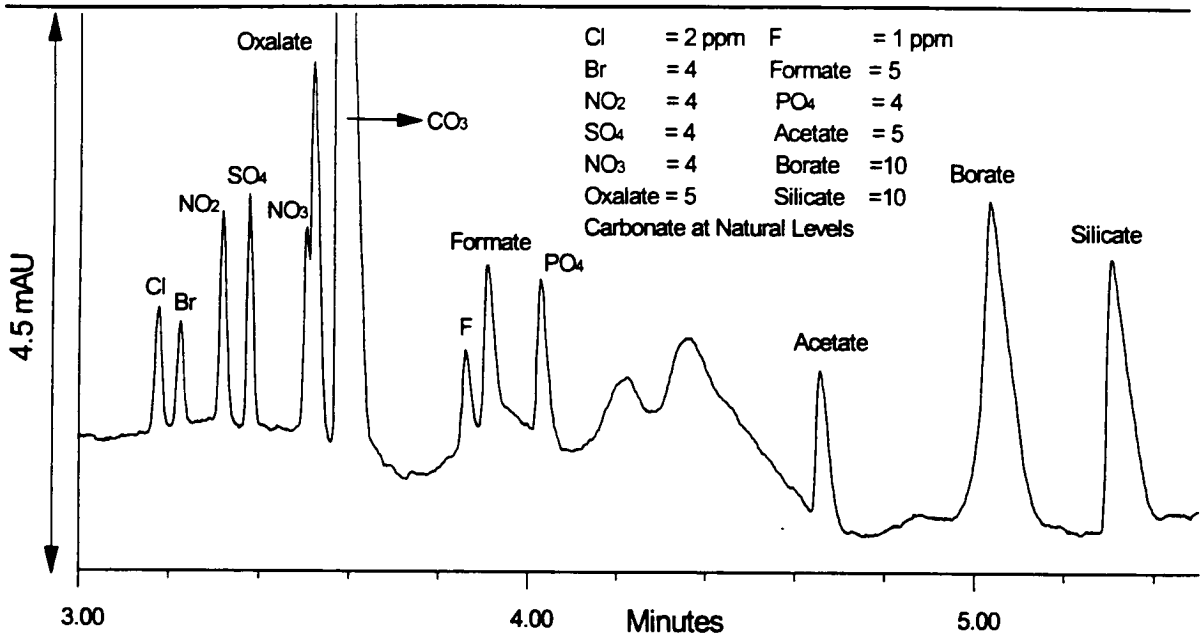
Capillary Ion Analysis Method

Analysis of Borate and Silicate Using a Alkaline Chromate Electrolyte 1997

Experimental Method

Required Instrumentation:

	Part / Number
Capillary Ion Analyzer	251000
Bus SAT/IN Module,	073645
Millennium32	Consult Waters



Analysis Conditions:

Electrolyte:	10 mM Chromate / 3 mM OFM-OH / 2 mM Sodium Carbonate / 1 mM Sodium Hydroxide, pH 11
Capillary:	75 µm (id) x 375 µm (od) x 60 cm (length)
Temperature:	25°C (5°C Above Ambient)
Power Supply:	Negative
Voltage:	15 kV
Current:	40 ± 1 µA (Use Constant Current for Analysis)
Sampling:	Hydrostatic for 30 Seconds
Detection:	Indirect UV at 254 nm, Hg Lamp, 185 or 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:

Prepare this electrolyte as needed as follows:

1) Into a 100 mL plastic volumetric flask add
   - -10 mL of 100 mM Sodium Chromate Solution
   - -3 mL of 100 mM OFM-OH (available from Waters, P/N 49387)
   - -2 mL of 100 mM Sodium Carbonate Solution
   - -1 mL of 100 mM Sodium Hydroxide Solution
   - -Dilute to volume with DI Water.
2) Natural pH is 11 ± 0.2.
3) Vacuum degas through a 0.45 μm aqueous compatible membrane.
4) Allow to thermally equilibrate in the CIA Analyzer for 15 minutes before use.
5) Discard after one day due change in electrolyte pH.

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 High Mobility 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.

Borate, pK_a 9.3, and silicate, pK_a 9.8, exist as anions with sample pH greater than 9, and with full ionization above pH 10.5. It is important that the pH of the sample be at 10.5 or higher for best quantitation. Adjust sample pH with 100 mM NaOH.

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 NaOH 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	1 = Constant Current (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	15 (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	
	Set Peak Width = 6 prior to fluoride migration time	
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 Quick Set) before recalibration.
Do Not use analyte peak height for quantitation due to asymmetrical peak shapes.

Observations and Comments:

-This electrolyte is considered experimental and subject to change.

-Due to the alkaline nature of this electrolyte, it will absorb CO_2 from the air causing a decease in electrolyte pH to 9.5. This pH change effects the ionization of borate and silicate resulting in longer migration times and decreased peak area, and a decrease in overall EOF.

-This electrolyte tends to give non-typical baseline stability.

Stock Reagent Preparation:

100 mM Sodium Chromate Solution: Dissolve 23.41 g of sodium chromate tetrahydrate ($Na_2CrO_4 \bullet 4H_2O$) in a 1 liter 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.

100 mM OFM-OH Solution: This tetradecyltrimethyl ammonium hydroxide (TTAOH) solution can be purchased from Waters Corp., P/N 49387, or can be prepared as needed as follows. Requires an anion exchange cartridge in the Hydroxide form, available from Alltech (as Maxi-Clean IC-OH Plus, P/N 30254; or equivalent) and a 20 mL plastic syringe.

Prepare a 100 mM OFM-Br Solution dissolve 3.365 g of tetradecyltrimethyl ammonium bromide (TTABr) in a 100 mL volumetric flask, and fill to the mark DI water. Convert only the require volume of OFM-Br to prepare the working electrolyte

Wash the anion exchange cartridge with 10 mL of 500 mM NaOH followed by 20 mL of DI water. Discard the washings. (Prepare sufficient volume for immediate use only, and use less than 10 mL per cartridge.)

Slowly pass the desired volume of TTABr through the cartridge into a volumetric flask used to prepare the final electrolyte. Rinse the cartridge with 20 mL of DI water, adding the washings to the volumetric flask.

100 mM Sodium Hydroxide Solution:– Dissolve 4 g of NaOH pellets In a 1 liter plastic volumetric flask, and fill to the mark with DI water. Store this solution in a capped plastic container for up to 1 year.

100 mM Sodium Carbonate Solution: Dissolve 10.6 g of sodium carbonate (Na_2CO_3) in a 1 liter volumetric flask with DI water, and fill to the mark with DI water. Store this solution in a capped plastic container at ambient temperature for up to 1 year.