

# Waters

## Capillary Ion Analysis Method

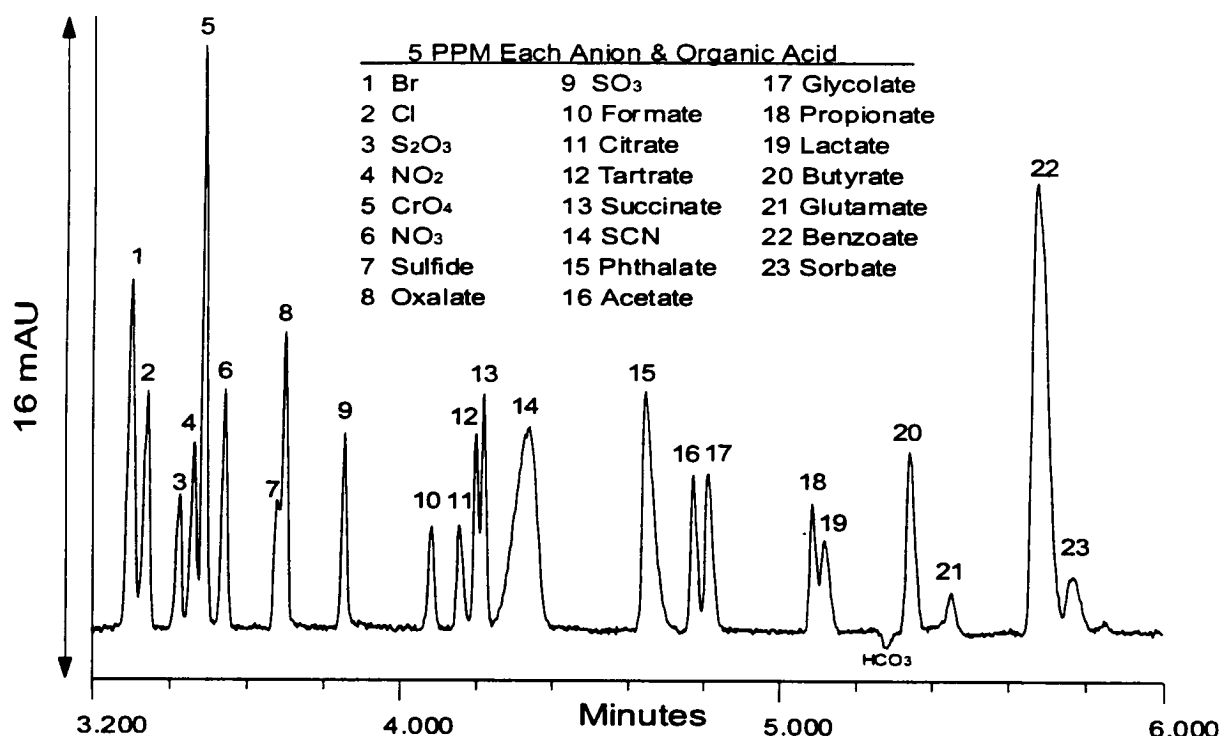
Direct UV Detection at 185 nm of Inorganic Anions and Organic Acids 2000

Required Instrumentation:

Capillary Ion Analyzer  
Bus SAT/IN Module  
Millennium 32

Part / Number

251000  
073645  
Consult Waters



Analysis Conditions:

Electrolyte:	25 mM Sodium Phosphate / 1 mM OFM-OH, pH 7
Capillary:	75 $\mu$ m (id) x 375 $\mu$ m (od) x 60 cm (length)
Temperature:	25°C (5°C Above Ambient)
Power Supply:	Negative
Voltage:	15 kV
Current:	40 $\pm$ 5 $\mu$ A (Use Constant Current for Analysis)
Sampling:	Hydrostatic for 30 Seconds
Detection:	Direct UV at 185 nm, Hg Lamp and 185 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:**

Refer to Reagents Section for Solution Preparation.

Prepare as follows:

- 1) Into a 100 mL volumetric flask add
  - 10 mL of 250 mM Sodium Phosphate Solution
  - 1 mL of 100 mM OFM-OH Solution (available from Waters , P/N 49387)
- 2) Dilute to volume with DI Water.
- 3) Natural pH should be  $7 \pm 0.1$ , adjust with 100 mM NaOH if necessary.
- 4) Vacuum degas through 0.45  $\mu$ m aqueous compatible membrane.
- 5) Store any unused electrolyte in a plastic container at ambient temperature.
- 6) Allow to thermally equilibrate in the CIA Analyzer for 15 minutes before use.
- 7) 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 High Mobility Electrolyte Kit should be used to evaluate electrolyte selectivity only. Only the UV active anions Br, NO<sub>2</sub>, and NO<sub>3</sub> will be detected; all other anions in the mix are not detected. Do not use for accurate quantitation.

Prepare at least 3 mixed analyte standards within the expected range of sample analyte concentration. This method is linear from 0.5 ppm to 100 ppm. After the multi-point calibration curve has been validated a single point calibration within the expected analyte concentration range is appropriate for recalibration.

**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.

The best quantitation is achieved when analytes are baseline resolved.

If necessary, dilute the sample with DI Water only. Analyte migration time reproducibility 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 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.

<u>SF# and Description</u>	<u>Value</u>
55 <u>CIA</u>	1 = CIA (Default)
88 <u>Custom IMT</u>	0, Enter 99 to activate SF# 58-60
89 <u>SPS</u>	1 = Constant Current (Enter in Sample Voltage)
90 <u>C2C Mapping</u>	0 = Off (Place electrolyte in position 1 only)
91 <u>t°C Temperature</u>	25 (5°C above ambient)
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>	15 (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

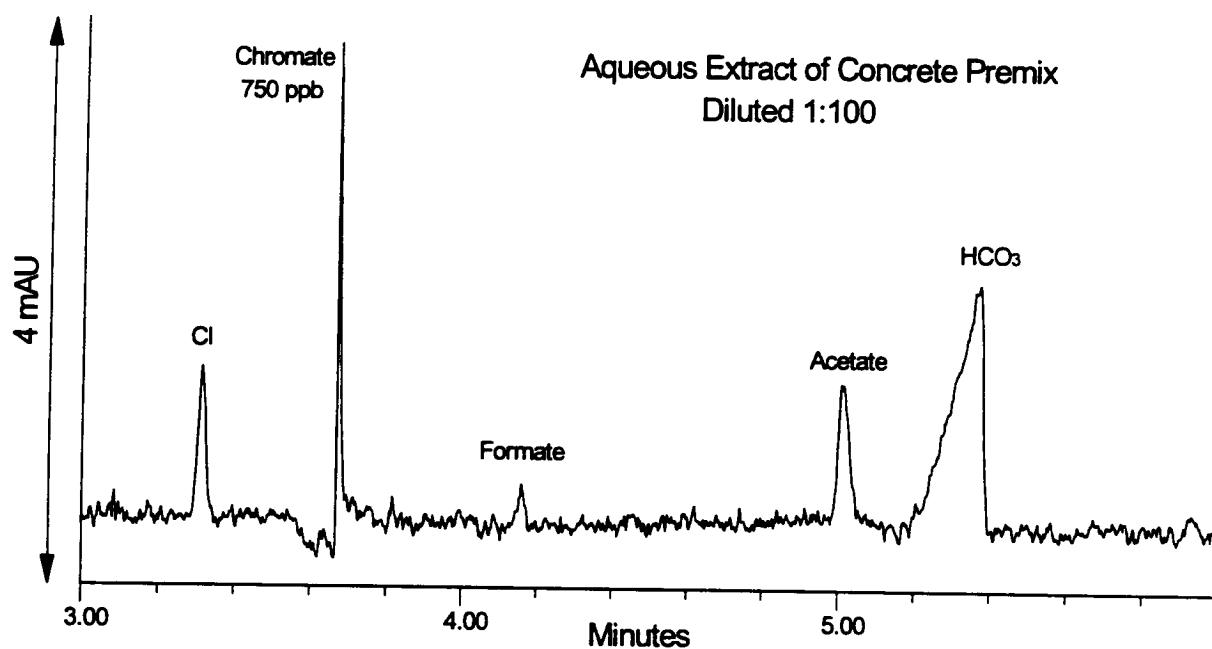
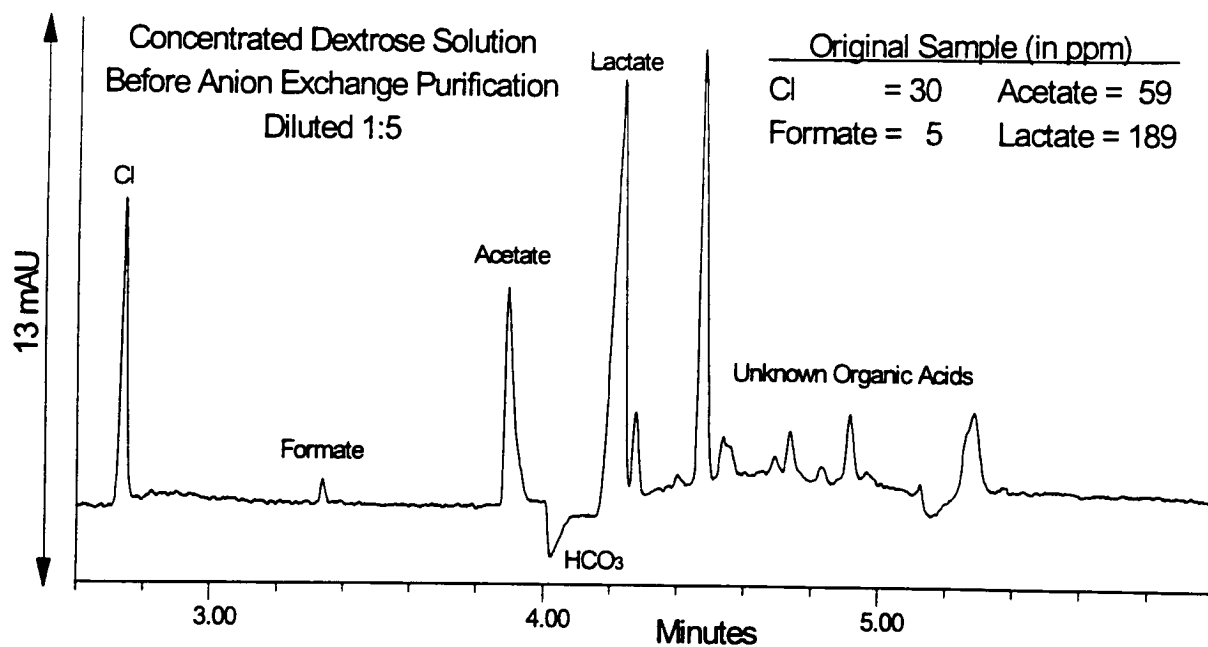
<u>Integration</u>	Peak Width = 2.25-3.00 Threshold = 100 ± 25
	Min Area = 100 Min Height = 50
	Inhibit Intg. = 0 to 3 min
<u>Calibration</u>	Averaging = None MT Window = 2%
	Update MT = Average Standards
	Peak Match = First (CI always first); MT Window = 10%
	= Closest for remaining analytes
	Quantitate By = Time Corrected Peak Area
	Fit Type = Linear, for multi-point calibration
	Linear Through Zero, for single point
<u>Report</u>	Analyte Name
	Analyte Migration Time
	Migration Time Ratio
	Peak Area
	Time Corrected Peak Area

Use fresh electrolyte daily; recalibrate with every change in electrolyte.

Clear previous calibration (in Quick Set Page) before recalibration.

Do Not use analyte peak height for quantitation due to asymmetrical peaks shapes.

### Examples of Use:



### Observations and Comments:

-Selectivity of this method can be changed by changing the concentration of the OFM-OH, and/or adjusting electrolyte pH.

-Presence of negative peaks suggests an anion present in the electrolyte that is not present in the sample. Its identity corresponds to its migration time.

**Stock Reagent Preparation:**

**250 mM Sodium Phosphate Solution:** Dissolve 1.725g of monobasic sodium phosphate monohydrate and 1.775 g anhydrous dibasic sodium phosphate in a 100 mL 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 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.

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