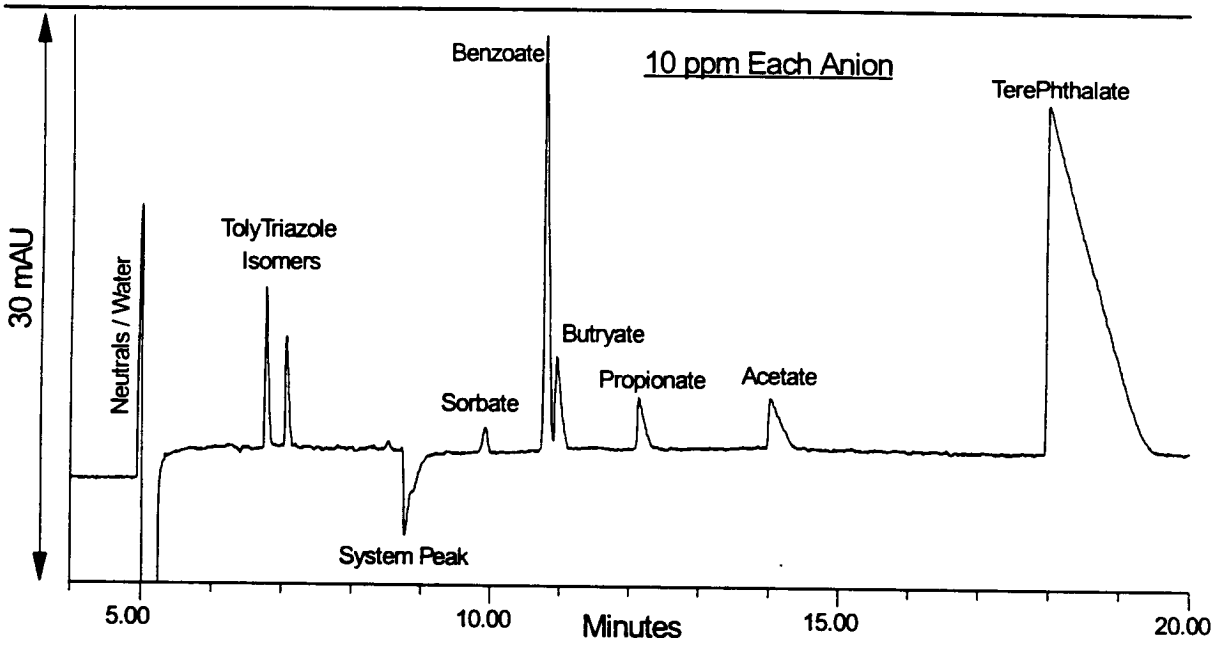
980904

Waters

Capillary Ion Analysis Method

Low Mobility Anion Analysis with Direct UV Detection at 185 nm 2000

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



Analysis Conditions:

Electrolyte:	10 mM Sodium Hexane Sulfonate / 10 mM CHES / 4 mM NaOH / 30% AcCN, pH 9
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
Current:	14 ± 1 µA (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:
This electrolyte can be prepared as follows: See Reagent Section for stock reagent preparation.

1) Into a 100 mL volumetric flask add
   -10 mL of 100 mM Sodium Hexane Sulfonate
   -10 mL of 100 mM CHES Buffer
   -4 mL of 100 mM Sodium Hydroxide
   -30 mL of HPLC grade Acetonitrile (AcCN)
   -Dilute to volume with DI water.
2) Natural pH is 9 ± 0.1.
3) Vacuum degas through 0.45 µm aqueous compatible membrane.
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.

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 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	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

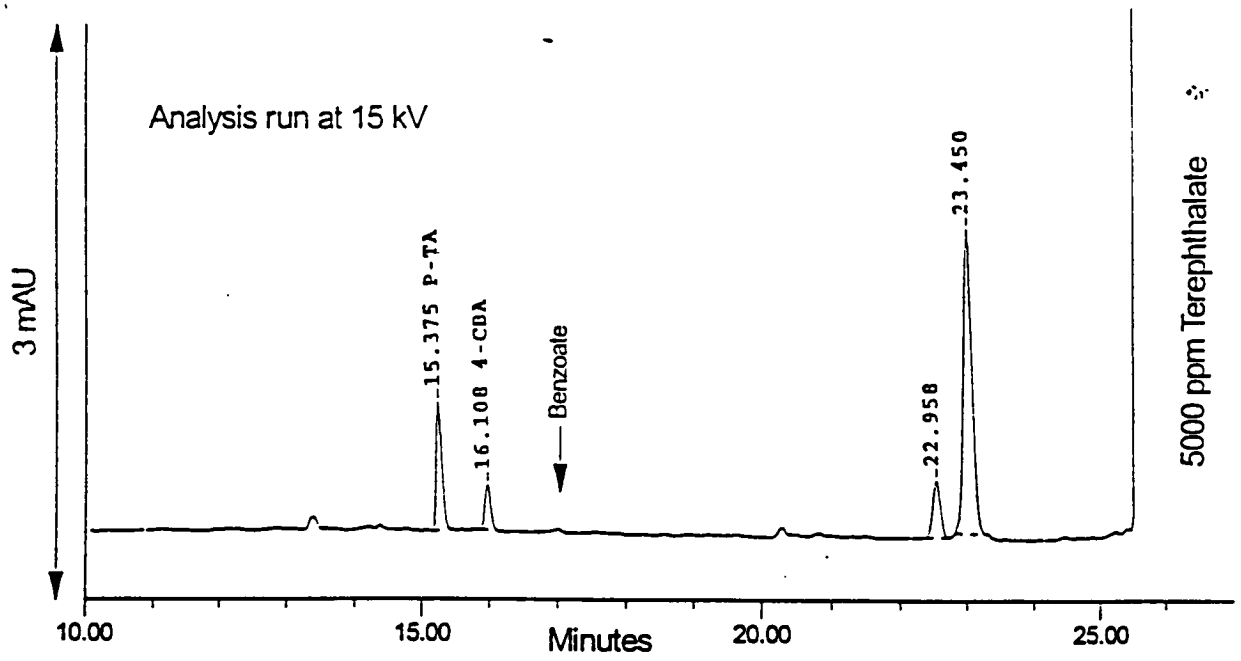
Integration
Peak Width = 2.25 - 3.00 Threshold = 100 ± 25
Min Area = 100 Min Height = 50
Inhibit Intg. = 0 to 4 min
Set Peak Width = 6 at 12 min

Calibration
Averaging = None MT Window = 2%
Update MT = Average Standards
Peak Match = Closest
(Use the neutrals peak as the reference peak)
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 Page) before recalibration.
Do Not use analyte peak height for quantitation due to asymmetrical peak shapes.

Examples of Use:
The QC analysis of impurities terephthalic acid; Benzoate, p-Tolulate, 4-Carboxybenzaldehyde, Hydroxymethylbenzoate. In a 100 mL volumetric flask dissolve 0.5 g of terephthalic acid in 400 mM NaOH (or NH_4OH); this gives a 5000 ppm terephthalic acid solution. Other unidentified impurities will be detected.



Observations / Comments:

-Low mobility through intermediate mobility, UV active anions are separated and detected in a "reversed" order, i.e. the low mobility anions are detected before the intermediate mobility anions. This is the reverse of the migration order noted with the IonSelect High Mobility Anion Electrolyte.

-This electrolyte does not use any OFM-OH to reverse the EOF. For the natural EOF to flow towards the detector a positive power supply is used. Do Not use capillaries that have been exposed to OFM.

-All cations pass through the detector first but are not detected because cations are transparent at 185 nm.

-A "neutrals Peak" will always be the first peak detected in the pherogram and contains water and all neutral organic in the sample matrix. This response is not quantitative. It can be used as a migration time reference peak.

-Improved peak shape for the intermediate mobility anions can be obtained by increasing the concentration of hexane sulfonate to 25 mM.

-Acetonitrile is added only to ensure solubility of the marginally soluble organic anions. If not needed for solubility, remove from the electrolyte; faster migration times will be observed.

-This electrolyte is optimized for the analysis of impurities in terephthalic acid manufactured using the "Amoco" process.