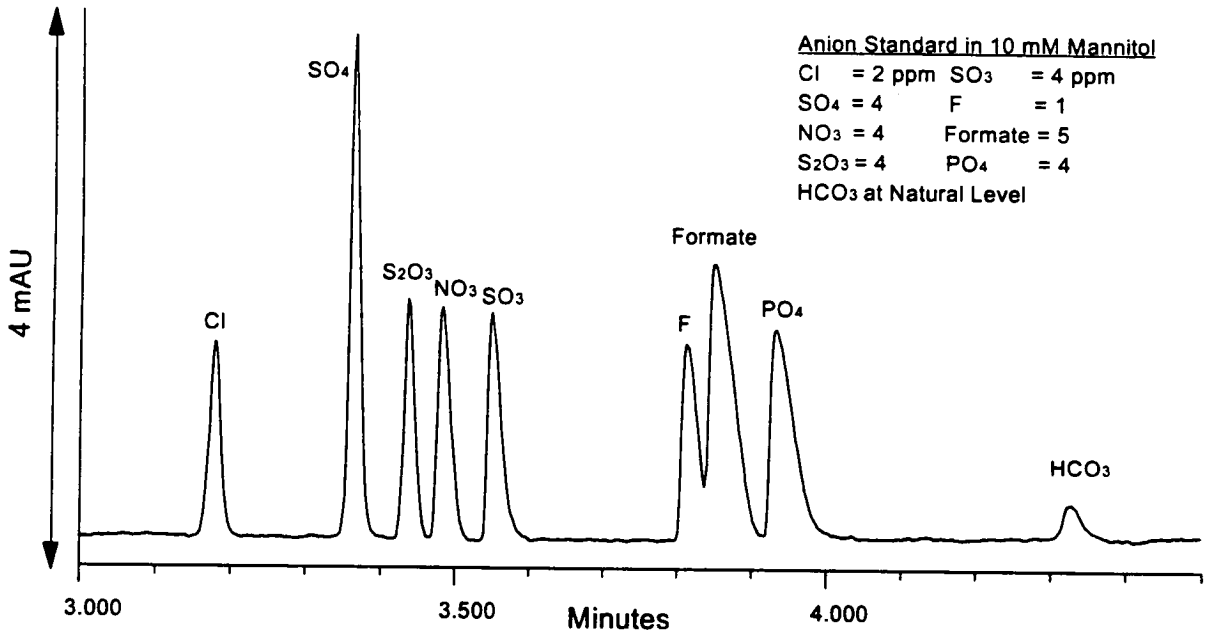
980900

Waters

Capillary Ion Analysis Method

Analysis of Sulfur Species and Common Anions 2000

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



Analysis Conditions:

Electrolyte:	5 mM Chromate / 3 mM OFM-OH / 10 mM CHES, pH 9
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:	14 ± 1 µA (Use Constant Current for Analysis)
Sampling:	Hydrostatic for 30 Seconds
Detection:	Indirect UV at 254 nm, Hg lamp and 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:

1) Into a 100 mL volumetric flask add
   - -5 mL of 100 mM Sodium Chromate Solution
   - -3 mL of 100 mM OFM-OH (available from Waters, P/N 49387)
   - -10 mL of 100 mM CHES Buffer Solution
   - -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.

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 in 10 mM Mannitol. 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. Add mannitol to aid in the preservation of sulfite (SO_3)

If necessary, dilute the sample with 10 mM Mannitol 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	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		
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/Comments:

-Phosphate quantitation in the sample may give low recovery due to complexation with dissolved transition metals in the sample matrix.

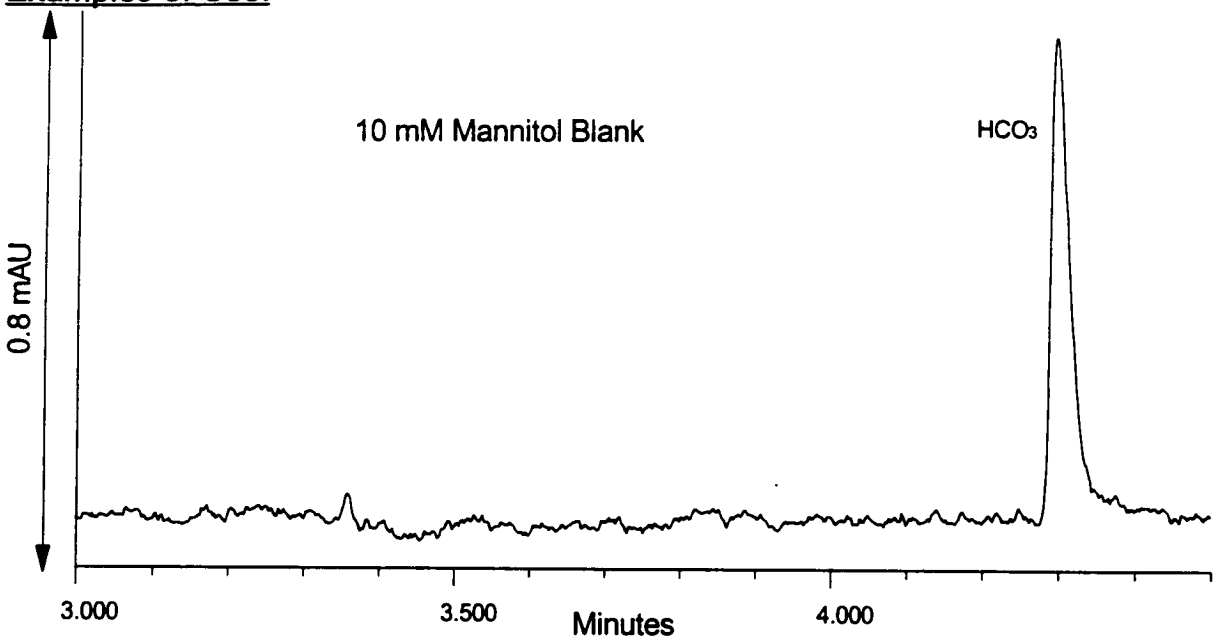
-Selectivity of this method can be changed by changing the concentration of the OFM-OH. At 4 mM OFM-OH, S_2O_3 and SO_3 comigrate; at 2 mM OFM-OH S_2O_3 and NO_3 comigrate.

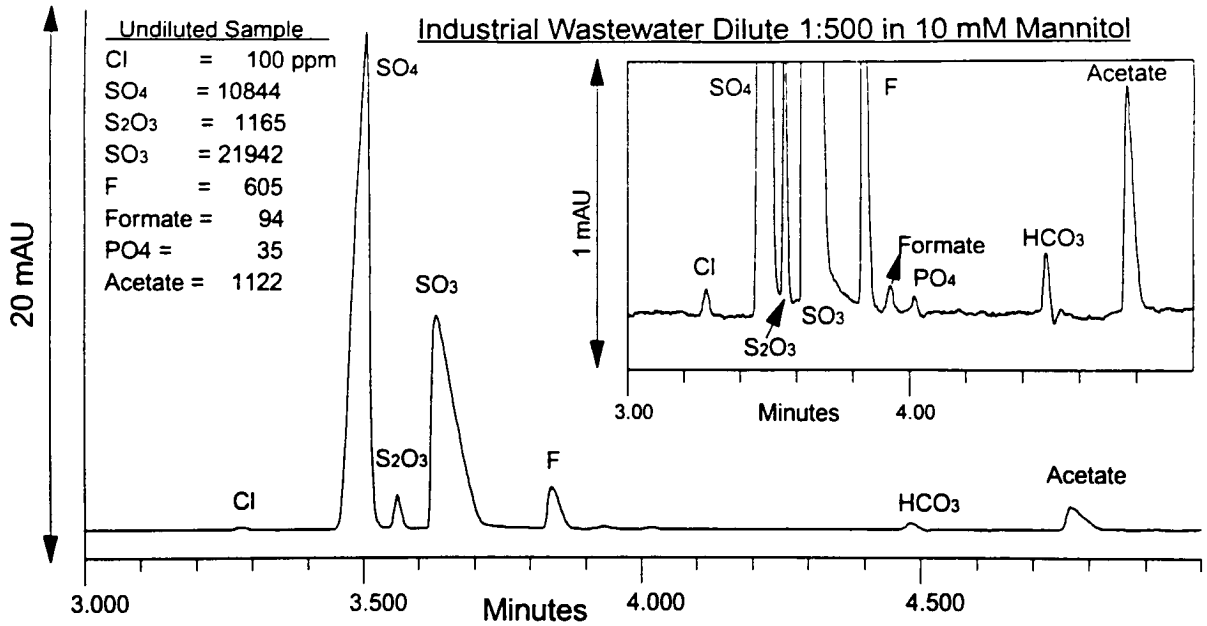
-Mannitol is added to stabilize sulfite (SO_3) from oxidizing to SO_4, per AOAC Sulfite analysis method.

-Sulfide as HS^- migrates in the SO_4 / NO_3 region of the pherogram and observed as broad "smeared" peak. It cannot be quantitated due to peak shape, its reactivity, and its complexation with sample matrix transition metals. Peak shape can be improved by increasing electrolyte pH to 11 without CHES buffer.

-Thiocyanate (SCN) is strongly effected by OFM-OH concentration, and it is not observed in this electrolyte. Best quantitated at 0.5 mM OFM-OH concentration.

Examples of Use:





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 CHES Buffer Solution:-- In a 1 liter volumetric flask dissolve 20.73 g of CHES (2-[N-CycloHexylamino]-Ethane Sulfonic Acid), and fill to the mark with DI water. Store this solution in a capped plastic container for up to 1 year.

10 mM Mannitol Solution: -- In a 1 liter volumetric flask dissolve 1.822 g of d-mannitol in 500 mL of DI water, and fill to mark with DI water.

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