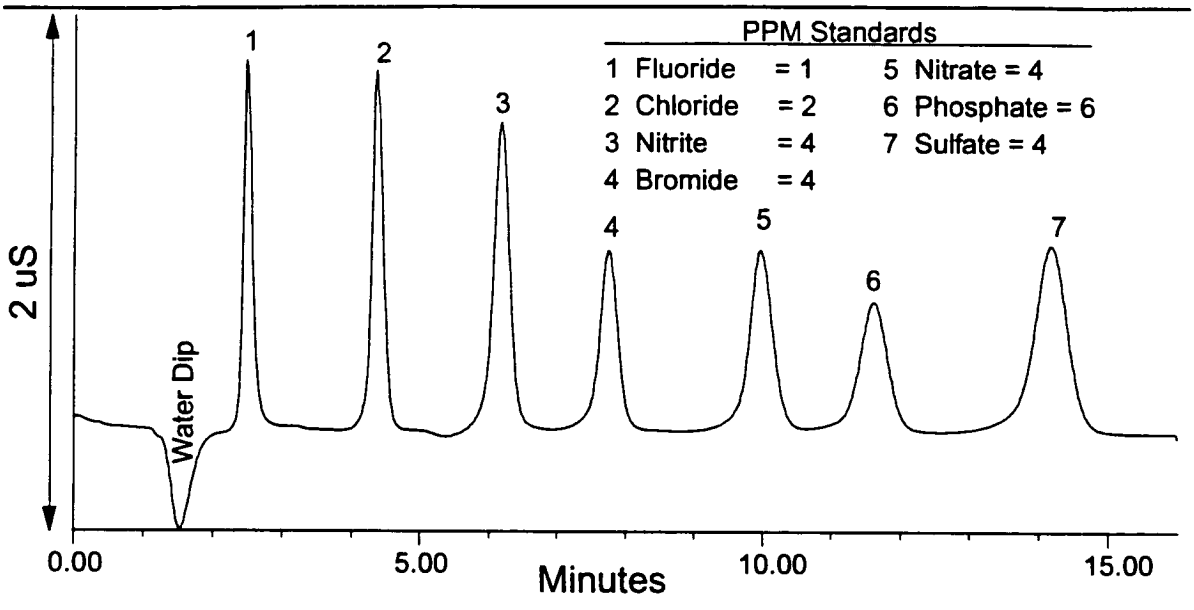
980897

Waters

Ion Chromatography Method

General Anion Analysis Using Chemically Suppressed Conductivity Detection
Equivalent to EPA Method 300.0 2000

Required Instrumentation:	Part / Number
Alliance, 2690 Separations Module (with Column Heater, Seal Wash, Degasser)	271013
432 Conductivity Detector	043061
Bus SAT/IN Module	073645
Millennium 32	Consult Waters
Alltech ERIS™ 1000HP Autosuppressor	Consult Alltech



Analysis Conditions:

Column:	IC-Pak Anion HR
Eluent:	1.6 mM $NaHCO_3$ / 1.4 mM Na_2CO_3
Back Conductivity:	10-15 µS
Regenerant:	DI Water or eluent
Degas:	Continuous
Flow Rate:	1 mL / min
Back Pressure:	1250 ± 200 psi
Temperature:	30°C (Column Heater); 35°C (Detector)
Injection:	25 µL
Needle Wash:	12% AcCN in DI Water
Detection:	Chemically Suppressed Conductivity
Base Range:	20
Attenuation:	100 µS / Volt
Polarity:	Positive

Eluent Preparation:

1) Into a 1 liter volumetric flask add
   - -16 mL of 100 mM $NaHCO_3$
   - -14 mL of 100 mM Na_2CO_3
2) Dilute to volume with DI Water
3) Vacuum degas through a 0.45 µm aqueous compatible membrane
4) Store in a glass or plastic container at ambient temperature. Discard after 1 week.

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 the sample analyte concentration. This method is linear from 0.1 ppm to 100 ppm. After the multi-point calibration curve has been validated, a single point calibration within the expected analyte concentration is appropriate for future calibrations.

Sample Preparation:

Determine the expected range of analyte concentration and other anionic component in the sample matrix. The major analyte should be less than 100 ppm for best results.

If necessary dilute the sample with DI Water.

If the sample contains high amounts of neutral organics, or is highly colored, then pass the diluted sample through a C_{18} Sep-Pak Cartridge. Anions pass through unretained, but may note a loss of fluoride recovery.

Samples containing suspended solids should be filtered through a 0.45 µm aqueous compatible disk prior to injection. Failure to filter solids results in the risk of increased column backpressure.

Sample pH should be within 3 to 11 for best results.

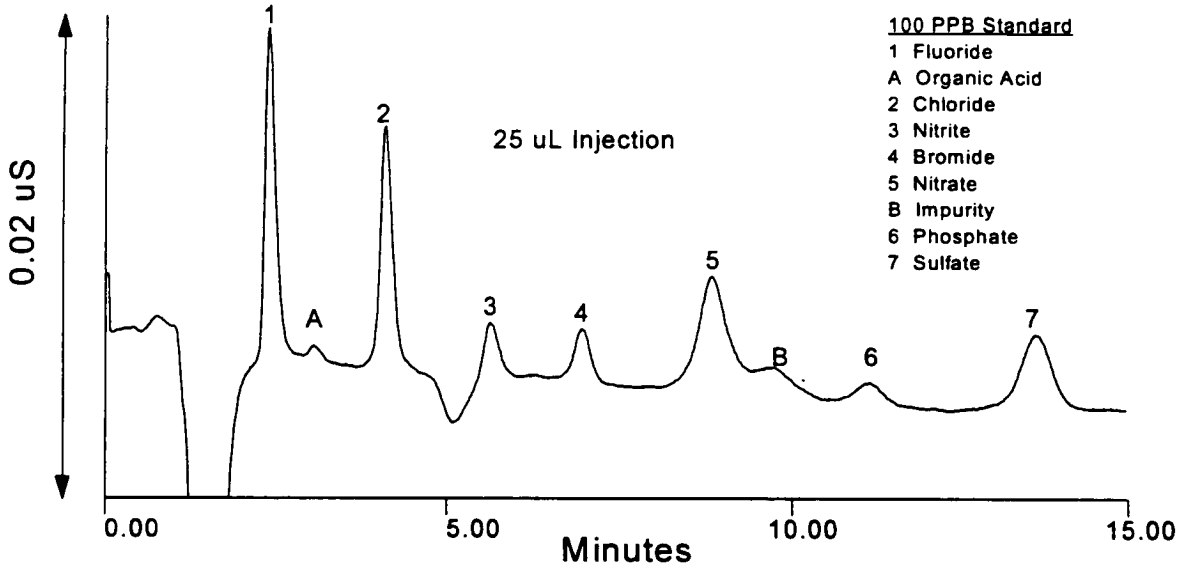
To minimize the water dip and improve quantitation of fluoride, dilute with eluent.

Millennium Data Processing Method:

IC Processing Method using Peal Apex for Retention Time

Integration	Peak Width	= 30.0	Threshold = 4--8
	Min Area	= 1500	Min Height = 75
	Inhibit Intg.	= 0 to 2 min	
Calibration	Averaging	= None	RT Window = 5%
	Update RT	= Never	
	Peak Match	= Closest	
	Quant By	= Peak Area	
	Fit Type	= Linear, for multi-point calibration or Linear Through Zero, for single point	
Report	Analyte Name		
	Retention Time		
	Peak Area		
	Amounts		

Method Detection Limits:



Based upon this representative chromatogram using a 25 µL injection, the estimated detection limits, as ppb, at 3 times signal to noise (S/N) are:

Fluoride = 5	Nitrite = 25	Nitrate = 25	Sulfate = 25
Chloride = 10	Bromide = 25	Phosphate = 75	

Lower detection limits can be obtained using a 250 µL injection. See Examples of Use for representative chromatogram.

Use of Direct UV Detection:

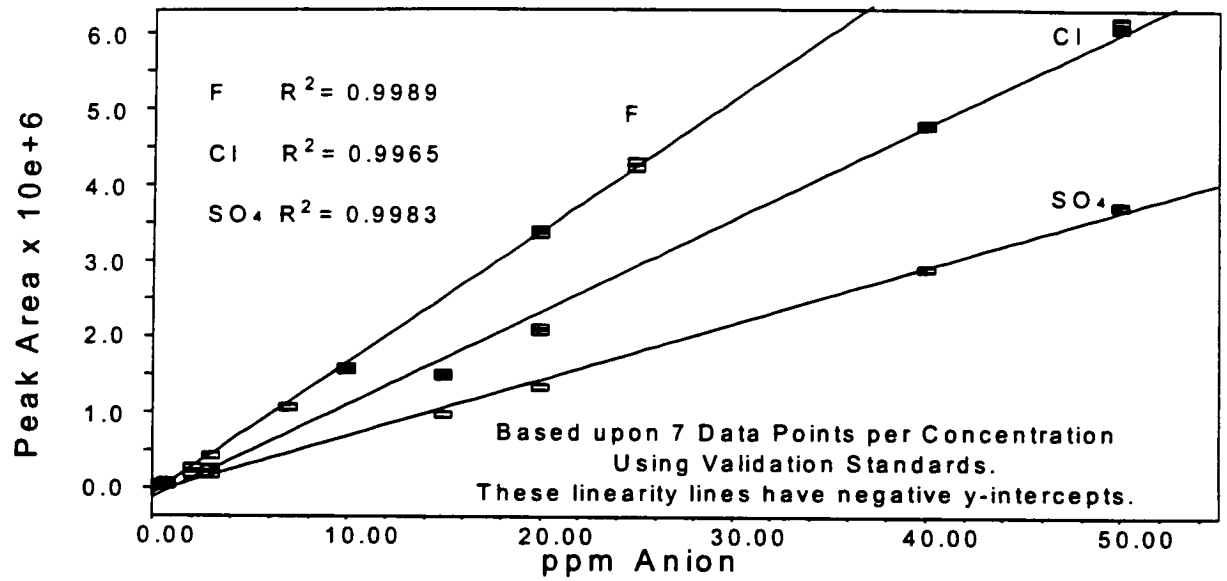
Many anions are UV active in the range of 205 nm to 214 nm, such as NO_2, Br, and NO_3, and the use of direct UV detection provides a greater degree of detector selectivity. Refer to Waters Anion Analysis Method using Borate / Gluconate for details.

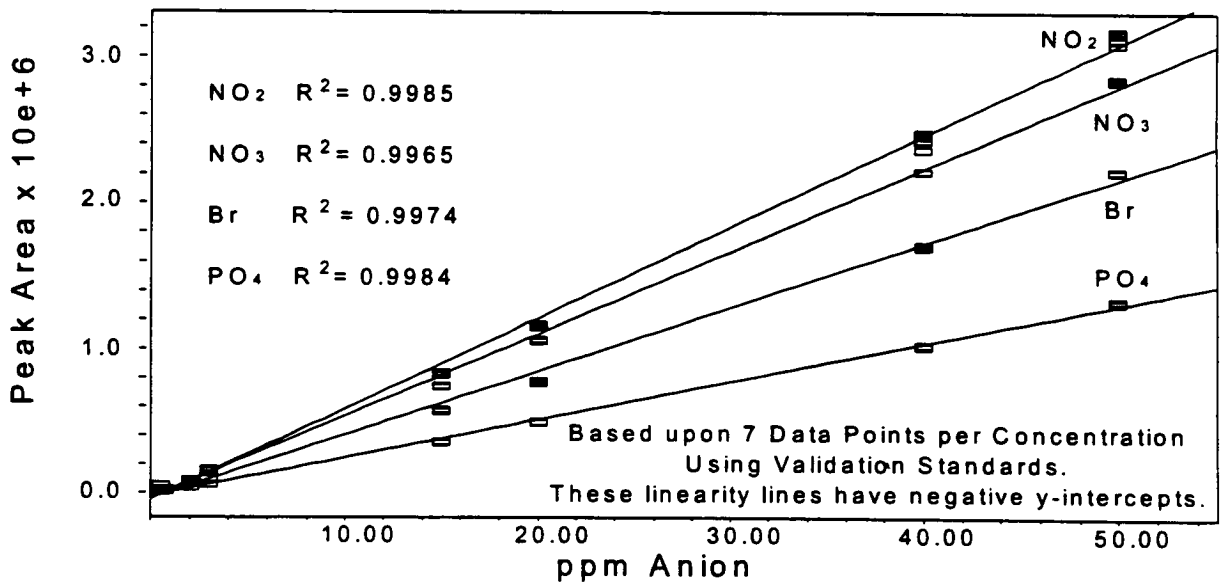
Method Validation: This validation design is abstracted from the ASTM/EPA validation of CIA. It has been used to validated all anion analysis methods. Many of the methods using this validation design are linear above 50 ppm.

Individual Youden Pair Standard, in ppm

Analyte Anion	1	2	3	4	5	6	7	8
Cl	0.7	2.0	3.0	15.0	40.0	20.0	50.0	0.5
Br	2.0	3.0	15.0	40.0	20.0	50.0	0.7	0.5
NO_2	3.0	40.0	20.0	15.0	50.0	0.5	2.0	0.7
SO_4	40.0	50.0	0.5	0.7	2.0	3.0	15.0	20.0
NO_3	15.0	20.0	40.0	50.0	0.5	0.7	2.0	3.0
F	2.0	0.7	0.5	3.0	10.0	7.0	20.0	25.0
PO_4	50.0	40.0	20.0	0.5	3.0	2.0	0.7	15.0

Method Linearity:





Quantitation Precision: %RSD of analyte peak area at each concentration. Data based upon 7 replicate injections of the validation standards.

Analyte		F	Cl	NO_2	Br	NO_3	PO_4	SO_4
ppm Concentration	0.5	0.62	3.64	6.82	2.57	1.65	8.93	5.58
	0.7	0.71	1.36	4.34	2.39	3.12	3.98	1.28
	2	0.34	3.69	1.95	1.29	1.19	1.58	0.85
	3	0.54	3.91	1.67	1.46	0.74	1.19	0.50
	15	1.36	1.73	0.90	0.84	0.36	0.49	0.34
	20	0.96	1.43	0.80	0.95	0.45	0.64	0.12
	40		0.28	1.64	0.41	0.09	0.48	0.19
	50		0.83	1.05	0.19	0.26	0.46	0.25

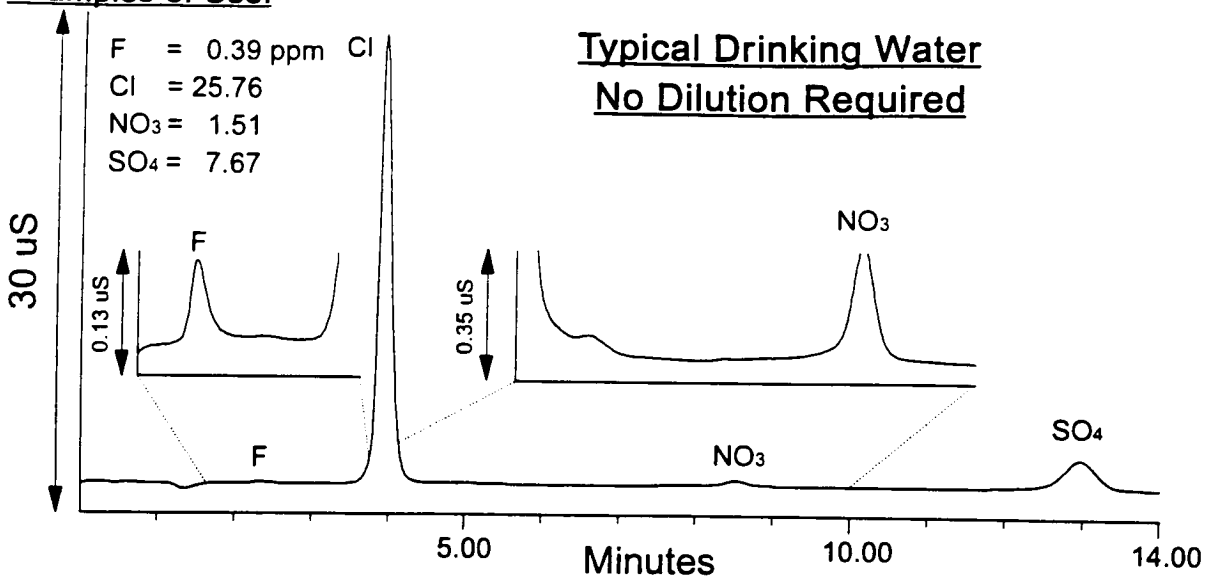
Quantitation Accuracy: Used a Certified Performance Evaluation Standard diluted 1:100 with DI Water. Amounts based upon multi-point calibration curve prepared from certified standards.

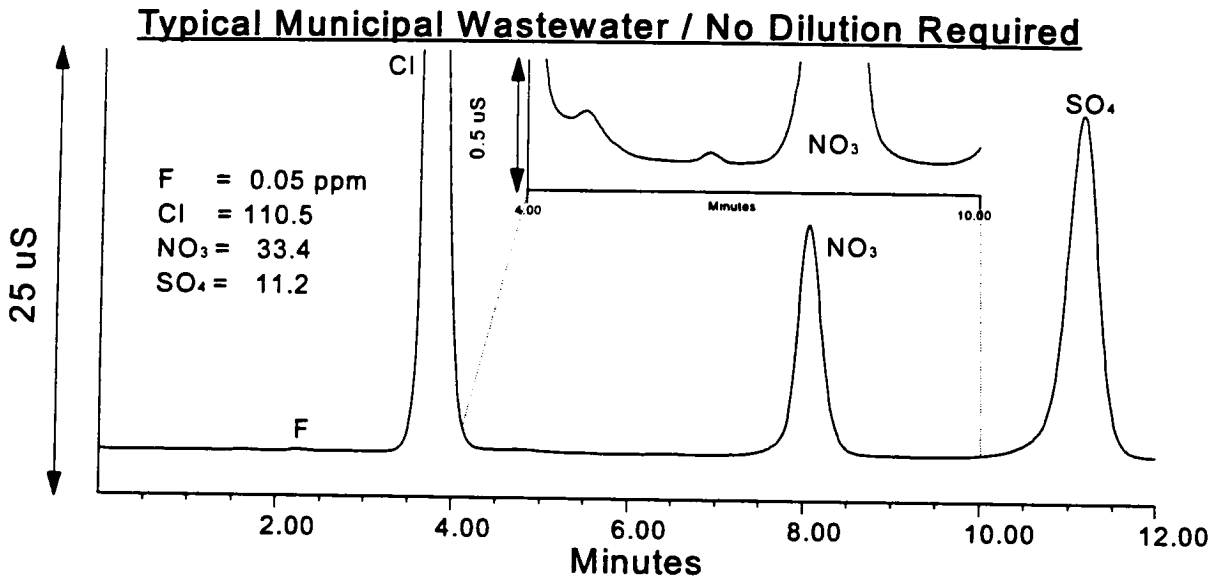
	Analyte	F	Cl	NO_2	NO_3	PO_4	SO_4
Performance Evaluation Standard	True Value in ppm	3.43	104.46	3.68	16.64	4.57	56.70
Official Anion Methods Wet Chem	Measured Mean	3.41	107.01	3.61	16.90	4.69	59.51
	Measured Std Dev	0.16	6.48	0.43	1.15	0.25	6.94
IC Using Alliance & ERIS 1000 IC Pak A/HR HCO_3/CO_3 Eluent	Ave IC n = 3	3.21 ± 0.01	116.54 ± 0.40	3.53± 0.03	15.43 ± 0.04	4.32 ± 0.04	59.32 ± 0.22
	IC / Mean	0.941	1.088	0.978	0.913	0.921	0.997
	IC / True Value	0.936	1.116	0.959	0.927	0.945	1.046

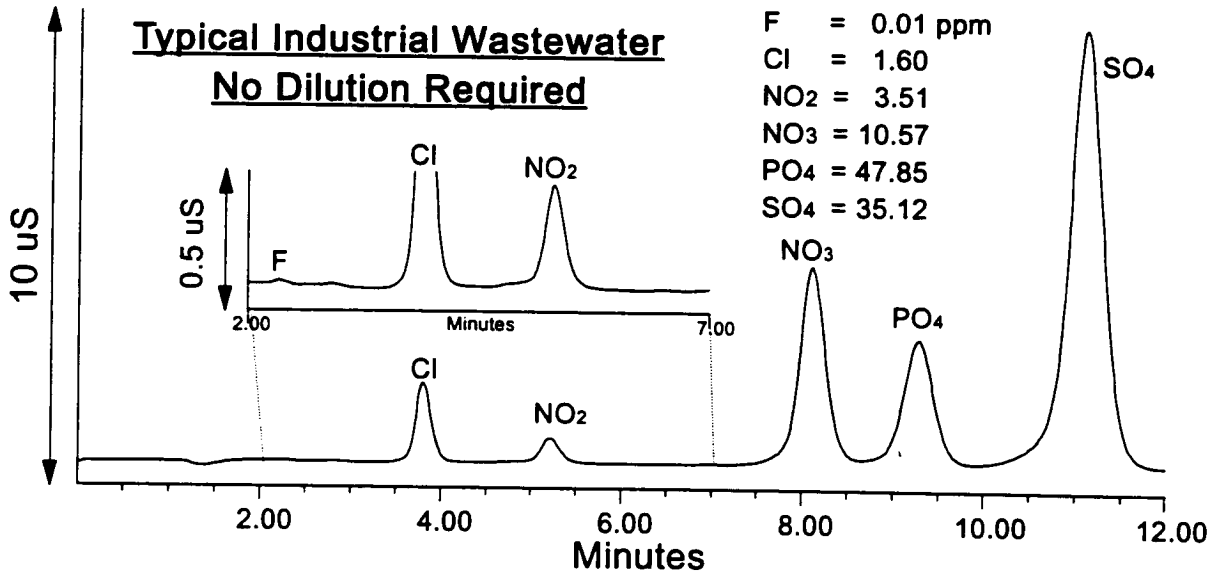
Analyte Recovery: The Certified Performance Evaluation Standard was diluted 1:100 with Typical Drinking Water. Amounts, in ppm, based upon multi-point calibration.

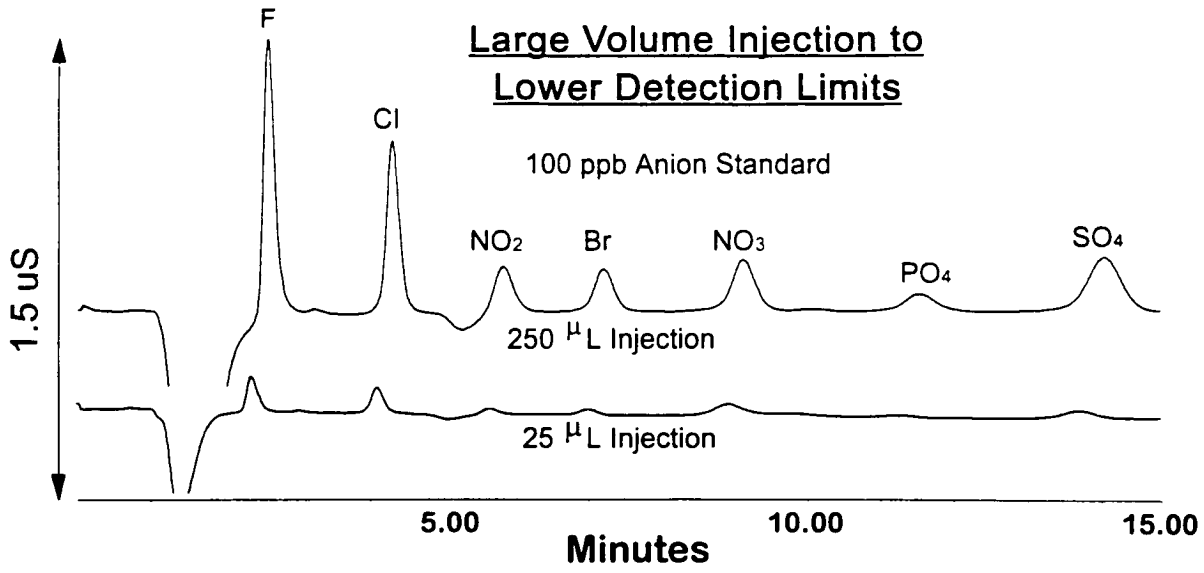
Analyte	F	Cl	NO_2	NO_3	PO_4	SO_4
Milford Drinking Water n=3, as ppm	0.39 ± 0.002	25.76 ± 0.21	Not Detected	1.51 ± 0.04	Not Detected	7.67 ± 0.01
%RSD	0.40	0.80		2.46		0.16
Performance Evaluation Std	3.43	104.46	3.68	16.64	4.57	56.70
MDW + PES n=3, as ppm	3.28 ± 0.02	138.24 ± 0.91	3.37± 0.02	17.23± 0.03	3.99± 0.05	69.40± 0.27
%RSD	0.49	0.66	0.43	0.163	1.215	0.39
%Recovery	84.3%	107.7%	91.4%	94.4%	87.3%	108.8%

Examples of Use:









Stock Reagent Preparation:

100 mM Sodium Bicarbonate Solution: Dissolve 8.4 g of sodium bicarbonate ($NaHCO_3$) 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.

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.

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