



IC APPLICATION NOTE

Simultaneous Determination of Anions and Disinfection By-Products (EPA 300.1) using Hydroxide as the Eluent

948 CEP Module to generate high purity hydroxide eluent

In this work, a single IC method was developed to simultaneously determine both Part A and Part B analytes in one run. Using a 100 μ L injection volume and conductivity detection, the method achieves detection limits down to low single digit ppb for these oxyhalides. Notably, chlorite and bromate are effectively detected and resolved even in the

presence of 100 ppm chloride. The surrogate, DCAA, is also included and is well separated even from a 100-ppm sulfate peak. The method utilizes a Supp 21-150/4.0 mm column in combination with the 948 CEP module for the generation of high purity potassium hydroxide eluent.

SAMPLE AND STANDARD PREPARATION

No sample preparation steps were needed for this application. However, if suspended particles are present, the sample needs to be filtered before injected into the IC. The mix standard solution was prepared using certified 1000 ppm individual stock solutions.

EXPERIMENTAL

This method utilizes A Supp 21-150/4.0 mm column for the separation of anions and disinfection by-products (DBPs) using potassium hydroxide as the eluent. A single conductivity detector was used to detect all of the analytes with a 100 µL sample loop at a flow rate of 1 mL/min. The column was maintained at a constant temperature of 40 °C.

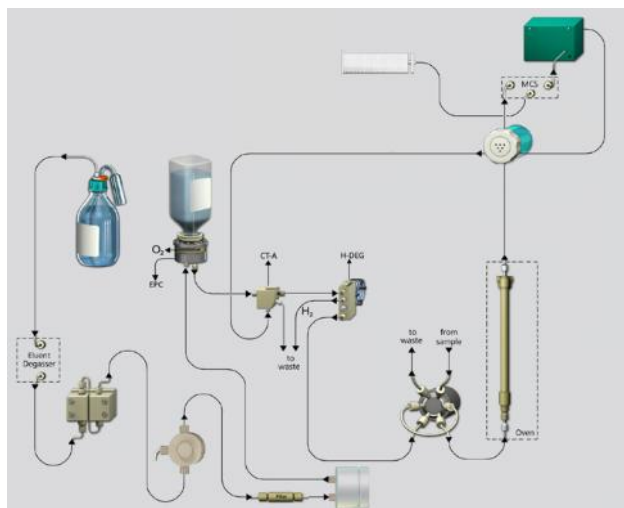


Figure 1. Flow path for the determination of anions and oxyhalides by suppressed ion chromatography.

RESULTS

Quadratic calibration curves were generated for all the analytes at various concentration ranges to cover the concentrations of these analytes present in the samples analyzed. The % RSDs for triplicate analysis showed excellent repeatability and the spike recoveries were all within the acceptable limits. The DCAA spike recovery (90 to 115 %) and the PGF values (0.80 to 1.15) were within the range described in EPA 300.1.

Table 1. Results (Sample R)

Analytes	Average conc., ppm (n=3)	RSD, % (n=3)
Fluoride	1.18	0.4
Chlorite	-	-
Bromate	-	-
Chloride	95.4	0.0
Nitrite	-	-
Bromide	0.09	1.9
Chlorate	-	-
Nitrate	4.49	0.1
DCAA	-	-
Sulfate	52.97	0.1
Phosphate	0.04	4.8

Table 2. Spike Recovery Results

Analytes	Sample R	Sample T	Sample O
Fluoride	101 %	102 %	99 %
Chlorite	106 %	117 %	110 %
Bromate	90 %	100 %	117 %
Chloride	101 %	100 %	102 %
Nitrite	114 %	97 %	112 %
Bromide	113 %	111 %	112 %
Chlorate	104 %	95 %	101 %
Nitrate	99 %	101 %	100 %
DCAA	96 %	94 %	96 %
Sulfate	100 %	100 %	101 %
Phosphate	105 %	99 %	102 %

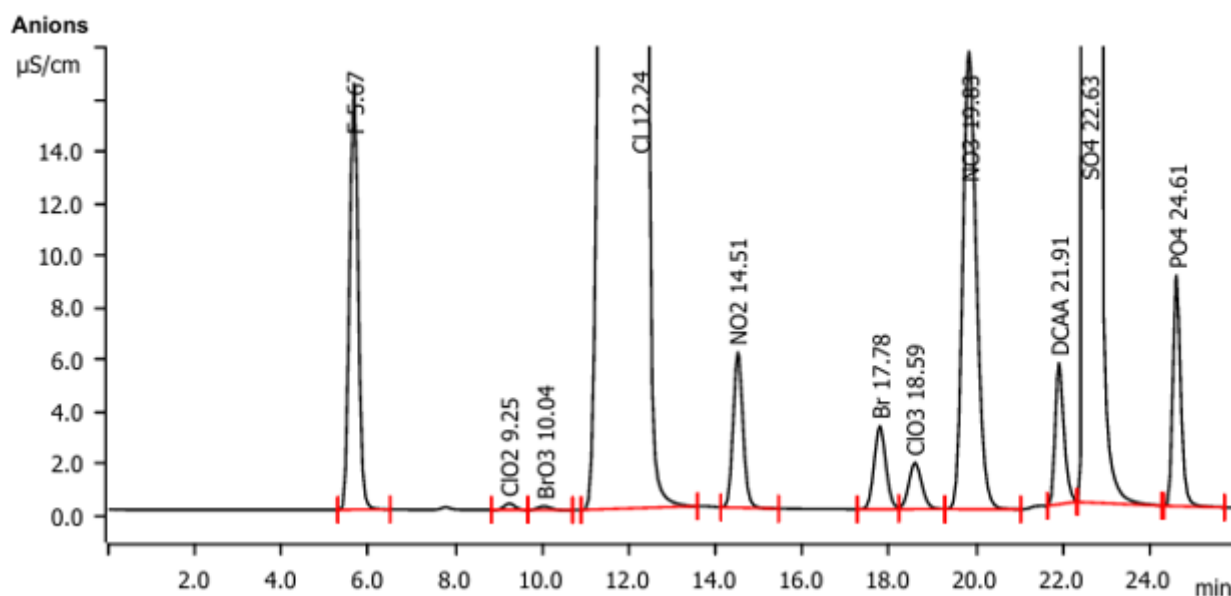


Figure 2. Standard 6

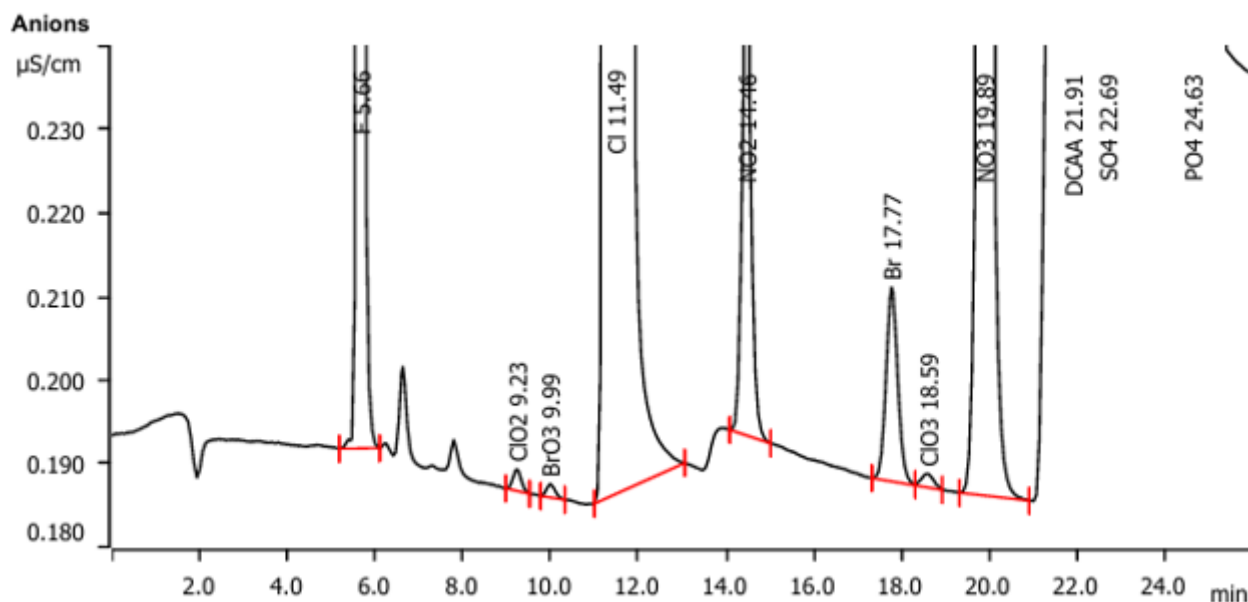


Figure 3. 2 ppb each of chlorite, bromate & chlorate in presence of other anions

CONCLUSION

This new method uses hydroxide as the eluent to perform EPA 300.1, both part A & B in various water matrices in a single run. Moreover, the method provides sufficient resolution between chlorite, bromate and chloride peak (especially chloride at 100 ppm). The automated eluent generation, the inbuilt gradient capability and short analysis time are key features of this method.

Analyte:	Anions and Disinfection By-Products
Matrix:	Water
Method:	Ion chromatography
Industry:	S02 Env. Testing
Standards:	USEPA 300.1