Bromate Analysis in Mineral Water Using LC-MS/MS

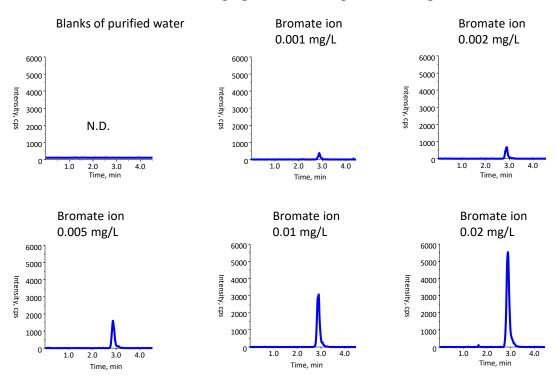
LT167
GL Sciences Inc.

The standard values for bromate content in mineral water are stipulated by the "Standards for Standardization of Foods, Additives, etc. (Not. 370, 1959)" in Article 1 of Food D (Standards for Composition of Soft Drinks). For mineral water to be disinfected or sterilized, the standard values are specified to be 0.01 mg bromate/L or less. The test method used is an ion chromatography post-column absorbance method.

In this study, bromate analysis in commercial mineral water was performed using LC-MS/MS. Good results were obtained using a SYPRON AX 1 ion-exchange column with LC-MS.

(R.Takahara, A.Tamura)

Confirmation of dissolution using a Bromate reference standard solution was added to ultrapure water to confirm concentrations ranging from 0.001 mg/L to 0.02 mg/L.



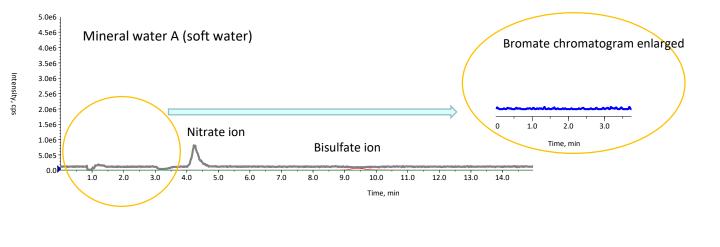
Assay conditions

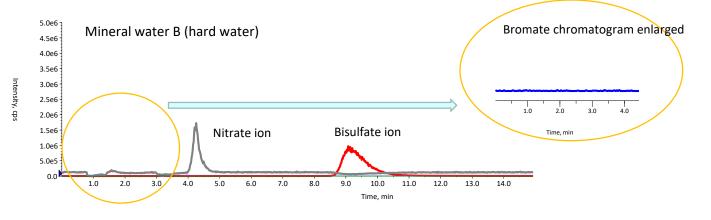
			Q1/Q3		
Column	:SYPRON AX-1 (5 μm, 100 x 2.1 mm I.D.)	Bromate ion	127.0/111.0		
Eluent	:A) 25 mM CH ₃ COONH ₄ in H ₃ O	Chloride ions	35.0/35.0		
	:B) CH ₃ CN	Nitrate ion	62.0/62.0		
	:A/B=70/30, v/v	Bisulfate ion	96.9/79.9		
Flow rate	:0.2 mL/min				
Inj. Vol	:10 μL				
Col. Temp.	:40 °C				
Detection	:MS/MS (4000 QTRAP: ESI, Negative, MRM)				

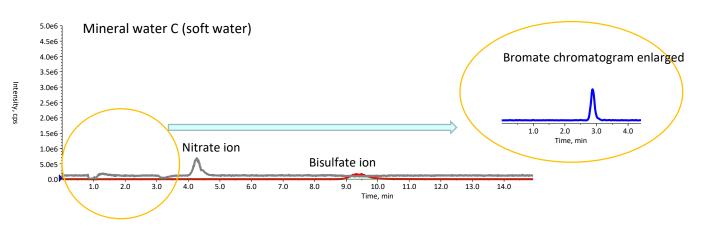


Chromatograms of mineral water

The World Health Organization (WHO) references state that the hardness of soft water is less than or equal to 120 mg/L, and hard water is greater than or equal to 120 mg/L. The chromatograms for analysis of commercial mineral water A (soft water), B (hard water), and C (soft water) are shown below. The concentration of bromate in mineral water C calculated using an added recovery calibration curve was about 0.0035 mg/L, and all mineral water levels were less than the standard values.



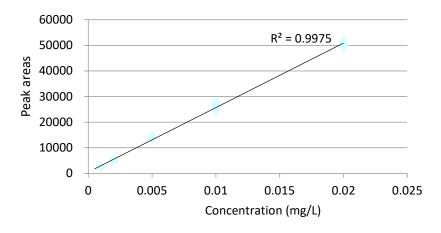




Creation of a standard calibration curve

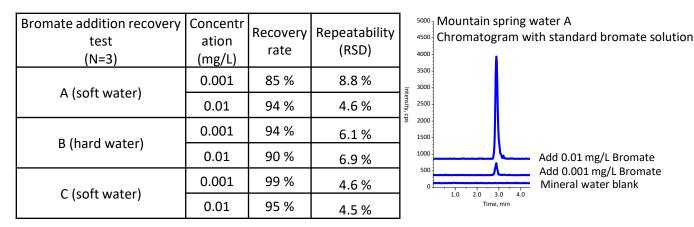
A bromate standard solution was added to ultrapure water to a prepare a calibration curve at concentrations ranging from 0.001 mg/L to 0.02 mg/L, good linearity was obtained with $R^2 = 0.9975$.

Concentration (mg/L)	Repeatability (RSD)
0.001	3.9 %
0.002	3.0 %
0.005	4.1 %
0.01	5.1 %
0.02	2.1 %



Spiked recovery tests

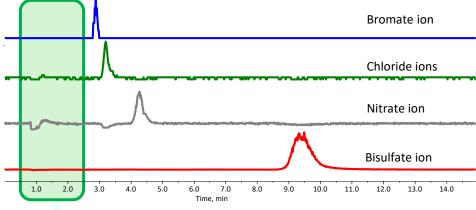
A standard bromate solution was added to commercially available mineral waters A, B, and C, and an additional recovery calibration curve was prepared at concentrations of 0.001 mg/L and 0.01 mg/L in a blank test. Good results were obtained for both recovery rate and repeatability.



For mineral water C, in which bromate was detected as present, the recovery rate was calculated by subtracting the results of the blank test.

For elution of minerals

Mineral water contains cations such as sodium, magnesium and calcium ions. Because the anion exchange column is unable to retain these cations, all components are eluted before the bromate peak.



The chromatograms have been modified in height to facilitate visualization of the elution positions of each ion

Analytical column

Description	Particle size (µm)	Inside diameter (mm)	Length (mm)	Cat. No.
SYPRON AX-1	5	2.1	100	5020-11002



NOTE) Columns are 12 MPa (120 bar) in pressure resistance.

Guard column

Description	Inside diameter (mm)	Length (mm)	Cat. No.
Replacement cartridge SYPRON AX 1 (two sets)	2.1	10	5020-08641
SYPRON Guard holders (with PEEK male nut)			5020-08640



SYPRON Guard holders



PEEK nut

NOTE) Columns are 12 MPa (120 bar) in pressure resistance.

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