Simultaneous determination of trace level bromate and chlorinated haloacetic acids in bottled drinking water by ion chromatography

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Abstract

A convenient and sensitive method for the simultaneous determination of trace level of bromate and chlorinated haloacetic acids in bottled drinking water with ion chromatography is presented. With a high capacity anion-exchange column and 11.5 mmol/l Na_2CO_3 eluent, all the 16 analytes could be separated in one injection within 31 min. By employing a microwave based evaporation technique, the bottled drinking water sample could be concentrated tenfold in 10 min. The recoveries of the compounds ranged from 90.6 to 107.2%. With a 500 μl large volume injection and high performance anion Atlas electrolytic suppressor, the detection limits were 0.06, 0.08, 0.06, 0.14 and 0.85 $\mu\text{g/l}$ for BrO_3^- , ClO_3^- , monoacetic acid, dichloroacetic acid and trichloroacetic acid, respectively.

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1. Introduction

Bottled drinking water has become a healthier choice than tap water for many people because they believe that bottled drinking water contains fewer contaminants, or dislike the taste of chlorinated tap water. For this reason, the annual consumption of bottled drinking water in the world is substantial [1].

It is well known that most of bottled drinking water is disinfected with ozone prior to being bottled. The source water may contain some halides such as Cl^- and Br^- and during the ozonation, disinfection by-products (DBPs) such as bromate and haloacetic acids (HAAs) may be

produced [2,3]. Bromate has been judged by both the World Health Organization (WHO) and the US Environmental Protection Agency (EPA) as a potential carcinogen, even at very low microgram per liter levels. US EPA has estimated a potential cancer risk equivalent to 1 in 10^4 for a lifetime exposure to drinking water containing bromate at 5 $\mu\text{g/l}$ [4]. HAAs, especially dichloroacetic acid (DCAA) and trichloroacetic acid (TCAA) were also found to be animal carcinogenic at low concentration [5]. Therefore, the US EPA has proposed a maximum contaminant level (MCL) of 10 $\mu\text{g/l}$ for bromate and 60 $\mu\text{g/l}$ for five specific HAAs in finished drinking water [6,7]. WHO also specified MCL for bromate (25 $\mu\text{g/l}$), DCAA (50 $\mu\text{g/l}$) and TCAA (100 $\mu\text{g/l}$) in drinking water [8].

Usually, bromate in drinking water can be directly determined with ion chromatography in sup-

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Fig. 1. The influence of pH value of the solution on the recoveries of the three chlorinated HAAs.

pressed conductivity mode [9,10] or determined with UV detector by post-column reaction [11–13]. However, the detection limits were not sufficient, and some of the reagents were possible carcinogens. Alternatively, the determination of trace level of bromate in drinking water could be carried out with MS-based detection techniques [14–17]. Though sub-microgram per liter level detection limits could be achieved with the methods, the cost for the instrumentation is significant and the techniques are considerably sophisticated.

Currently, methods used to determine HAAs involve gas chromatography with electron capture detection [18] or coupled with mass spectrometry [19,20], capillary electrophoresis (CE) [21], electrospray ionization mass spectrometry based technique (ESI-MS) [22–24], and ion chromatography [25,26]. For the GC methods, HAAs must be derivatized and extracted from water samples. However, most of the derivatization reagents were harmful compounds, and the extraction was time-consuming. For CE methods, the extraction was also needed. In addition, the method was not yet adequately rugged or reliable to meet the demands of trace-level quantitation of HAAs. The ESI-MS based technique was a sensitive and selective method. However, the cost of the instrument was too much [27].

In drinking water samples, bromate, MCAA, DCAA and TCAA are the most common DBPs. Until now, there are few methods for the simultaneous determination of the compounds. Therefore,

it is highly desirable to establish a convenient, sensitive and low-cost method for the synchronous detection of trace bromate and chlorinated HAAs in drinking water.

In this article, according to the specification of WHO on the DBPs in drinking water, a sensitive method for the determination of bromate, MCAA, DCAA and TCAA in drinking water was developed. Some anions and organic acids of interest were also detected. The developed method was applied to the detection of the compounds in several bottled drinking water samples.

2. Experimental

2.1. Chemicals

All reagents used were of analytical reagent grade unless specified. MCAA, DCAA, TCAA, bromate and chlorate were all obtained from ChemService (USA). All solutions were prepared with deionized water further purified with EASY-purified LF system (Barnstead) with a specific resistance of 18.3 M Ω cm, filtered through a 0.2 μ m membrane filter. Fluoride, chloride, nitrite, bromide, nitrate, phosphate, sulfate, iodide, formic acid, acetic acid, oxalic acid, bromate, chlorate, MCAA and TCAA stock solutions (1000 mg/l) were prepared by dissolving the corresponding mass of solid compounds in purified water. DCAA stock solution was prepared from 99% DCAA solution. The stock solutions were stored in a refrigerator at 4 °C. Standard solutions were prepared daily by serial dilution of the stock solution prior to use. The eluent was 11.5 mmol/l Na₂CO₃. All the determinations were performed at room temperature, and samples were injected in triplicate. The bottled drinking water samples were the three most popular brands in China, which were bought randomly from a local supermarket. The types of the bottled drinking water were purified water, mineral water and natural water, respectively.

2.2. Instruments

A Dionex Model DX-500 ion chromatography (Sunnyvale, CA) equipped with a GP40 gradient

pump, a Dionex Ionpac AS9HC analytical column ($4 \times 250 \text{ mm}^2$) and an Ionpac AG9HC guard column ($4 \times 50 \text{ mm}^2$) was used throughout. All the detections were performed by a Dionex ED40 electrochemical detector in the conductivity mode. Suppression of the eluent was achieved by a Dionex anion Atlas electrolytic suppressor (4 mm) in the autosuppression recycle mode. The suppressor was controlled with a SC20 suppressor controller. A 500- μl sample loop was used for injection. All tubing in the chromatography path (from the outlet of the pump to the exit of the suppressor) was PEEK (0.005 in (0.125 mm) I.D.). Both instrument control and data collection was performed with a personal computer and a Dionex PeakNet 5.11 chromatography workstation. The microwave oven was a commonly household one with five cooking level (output: 600 W, output frequency: 2450 MHz, Philips 7510T, China). All the samples were filtered with 0.45 μm filters (Autosience, Tianjin, China). One hundred-milliliter polytetrafluoroethylene (PTFE) beakers were used for sample concentration. The beakers were cleaned with a 150 W AS3120A sonicator (Autosience, Tianjin, China).

3. Results and discussion

3.1. Sample pretreatment

In drinking water, the concentrations of chlorinated HAAs were very low. In order to accurately detect HAAs, the sample should be concentrated prior to determination. Sarzanini et al. [26] reported the preconcentration of HAAs in drinking water by employing activated carbon as adsorbent. However, because of the strong retention, HAAs could not be completely eluted, which led to the poor recoveries of all the HAAs. Microextraction procedure was also used for the preconcentration of HAAs in drinking water. Though good recoveries could be obtained, the procedure was time-consuming and laborious. Furthermore, interferences could be easily introduced in the sample [25].

Fluoride, chloride, nitrite, bromide, nitrate, phosphate, sulfate, iodide, bromate and chlorate are all thermally stable anions that will not be decom-

posed in 100 °C. The boiling points of formic acid (100.5 °C), acetic acid (118 °C), MCAA (189 °C), DCAA (194 °C) and TCAA (196 °C) are all above 100 °C and oxalic acid will not be decomposed until 157 °C. Therefore, an evaporative technique could be used for the concentration of the compounds in water solution with microwave radiation.

In this experiment, the container used for sample concentration was 100-ml of PTFE beaker. With the beaker, 20 ml of sample solution could be concentrated to less than 2 ml in 10 min with the microwave oven operated at the middle-high level. The concentrated solution was transferred to a well-calibrated 2.0 ml PTFE vial, and then filled to 2.0 ml with micropipette. The results indicated that all the analytes had good recoveries except DCAA and TCAA, whose recoveries were only 27.7 and 34.2%, respectively.

In heated acidic aqueous solution, DCAA and TCAA could be decomposed. The pH value of the mixed solution of the 16 compounds was approximately 4. Consequently, the two analytes were decomposed during the concentration procedure and recoveries of the 2 compounds were very poor. However, DCAA and TCAA could be converted to dichloracetate and trichloracetate by reaction with alkaline solution. Dichloracetate and trichloracetate would not be decomposed in heated water solution and could be concentrated with the evaporative technique. From Fig. 1, it could be seen that the recoveries of DCAA and TCAA were greatly enhanced along with the increasing of the pH value of the sample solution, which was adjusted with 0.1 mol/l sodium hydroxide. When the pH value of the solution was between 10.5 and 11.0, the recoveries of DCAA and TCAA were all above 95%. Subsequently, the recoveries declined with the increasing of the pH value of the solution. Thereby, the optimum choice was to regulate the pH value of the solution between 10.5 and 11.0. With regards to the pH changing of the solution, the recoveries of the other compounds were not affected. Satisfactory results were always obtained with this technique (Table 1).

The inner wall of the PTFE beaker was extremely smooth, hydrophobic, and the water solution was not retained on it. In order to test the reliability

Table 1

The recoveries of the compounds concentrated with evaporative technique

Analyte	F ⁻	CH ₃ COOH	HCOOH	MCAA	BrO ₃ ⁻	Cl ⁻	NO ₂ ⁻	DCAA
Original concentration (μg/l)	5	20	10	10	10	5	10	10
Found after tenfold concentration (μg/l)	53.6	188.2	95.2	92.7	93.9	52.1	90.7	92.4
Average recovery (% , n = 7)	107.2	94.1	95.2	92.7	93.9	104.2	90.7	92.4
R.S.D. (% , n = 7)	4.21	6.98	5.47	4.26	4.88	3.09	4.58	4.96
Analyte	Br ⁻	ClO ₃ ⁻	NO ₃ ⁻	PO ₄ ³⁻	SO ₄ ²⁻	COOHCOOH	TCAA	I ⁻
Original concentration (μg/l)	10	10	10	10	5	15	15	15
Found after tenfold concentration (μg/l)	103.1	96.2	105.8	103.4	45.9	135.9	136.7	136.4
Average recovery (% , n = 7)	103.1	96.2	105.8	103.4	91.8	90.6	91.1	90.9
R.S.D. (% , n = 7)	5.51	4.22	4.37	5.69	4.11	5.45	4.56	6.86

of the method, after concentration procedure of a standard solution, 20 ml of deionized water was instantly concentrated tenfold with the beaker. The concentrated deionized water was analyzed with ion chromatography. The results indicated that all the 16 analytes could be detected in the concentrated solution, but the amount of each compound

Fig. 2. The chromatogram of standard solution of the 16 analytes. Dionex IonPac AG9HC and AS9HC columns; detection: suppressed conductivity with anion Atlas electrolytic suppressor-4 mm operated at 80 mA in autosuppression recycle mode; injection volume: 500 μl; eluent: 11.5 mmol/l Na₂CO₃; flow rate: 1.0 ml/min 1. Fluoride (0.1 mg/l), 2. CH₃COOH (2 mg/l), 3. HCOOH (1 mg/l), 4. MCAA (1 mg/l), 5. Bromate (0.5 mg/l), 6. Chloride (0.1 mg/l), 7. Nitrite (0.4 mg/l), 8. DCAA (1 mg/l), 9. Bromide (0.7 mg/l), 10. Chlorate (0.6 mg/l), 11. Nitrate (0.6 mg/l), 12. Phosphate (1 mg/l), 13. Sulfate (0.8 mg/l), 14. COOHCOOH (1.5 mg/l), 15. TCAA (5 mg/l), 16. Iodide (3 mg/l).

was very little. Each analyte accounted for only 1–2% of the original amount of the corresponding compound in the standard solution. When the deionized water was concentrated for the second time in the same manner, none of the compounds could be detected. Therefore, the adsorption of the compounds on the surface of the beaker could be ignored. The method was available for the concentration of the 16 analytes in bottled drinking water samples.

It was reported that MCAA could be hydrolyzed in alkali solution to form glycolate in some circumstances [28], which may result in serious interference to the accuracy determination of MCAA. Additional experiments were performed to investigate whether the hydrolysis would occur during the concentration process and the final results indicated that during the concentration process, no MCAA was hydrolyzed under the presented condition.

The pH value of the bottled mineral water, natural water and purified water were 7.60, 8.30 and 9.25, respectively. All the samples were corrected with 0.1 mol/l sodium hydroxide prior to being concentrated.

3.2. Optimization of chromatographic parameters

The IonPac AS9HC column has very high capacity that requires the use of a highly ionic strength eluent to achieve satisfactory elution times. In order to achieve a satisfactory separation

Table 2

The detection limits and linear ranges of the 16 analytes in original solution

Analyte	F ⁻	CH ₃ COOH	HCOOH	MCAA	BrO ₃ ⁻	Cl ⁻	NO ₂ ⁻	DCAA
Concentration (μg/l)	50	50	50	50	50	50	50	50
R.S.D. (% , n = 7)	2.88	2.67	2.08	2.71	3.01	1.45	1.39	1.09
Detection limit ^a (μg/l)	0.009	0.18	0.03	0.06	0.06	0.01	0.04	0.14
Concentration range (μg/l)	0.01–60	0.2–80	0.1–80	0.1–80	0.06–80	0.05–200	0.5–100	0.5–80
Correlation coefficient (r ²)	0.9912	0.9757	0.9964	0.9976	0.9812	0.9783	0.9980	0.9982
Analyte	Br ⁻	ClO ₃ ⁻	NO ₃ ⁻	PO ₄ ³⁻	SO ₄ ²⁻	COOHCOOH	TCAA	I ⁻
Concentration (μg/l)	30	50	50	50	50	50	50	50
R.S.D. (% , n = 7)	1.82	1.66	1.59	1.43	1.75	1.46	1.83	1.49
Detection limit ^a (μg/l)	0.07	0.08	0.06	0.22	0.11	0.15	0.85	0.32
Concentration range (μg/l)	0.5–50	0.2–80	0.5–15 000	0.5–100	5–500	1–500	2–500	1–100
Correlation coefficient (r ²)	0.9982	0.9984	0.9712	0.9934	0.9641	0.9976	0.9972	0.9962

^a Calculated as three times of baseline noise.

Fig. 3. The chromatogram of the tenfold concentrated natural water sample. Dionex IonPac AG9HC and AS9HC columns: detection: suppressed conductivity with anion Atlas electrolytic suppressor-4 mm operated at 80 mA in autosuppression recycle mode; injection volume: 500 μL; eluent: 11.5 mmol/l Na₂CO₃; flow rate: 1.0 mL/min. 1. Fluoride (382.1 μg/l), 2. HCOOH (534.1 μg/l), 3. Bromate (1.3 μg/l), 4. Chloride (211.2 μg/l), 5. Nitrite (73.3 μg/l), 6. DCAA (6.5 μg/l), 7. Bromide (30.4 μg/l), 8. Chlorate (4.2 μg/l), 9. Nitrate (12890.1 μg/l), 10. Phosphate (52.4 μg/l), 11. Sulfate (48510.2 μg/l), 12. Unknown peak, 13. COOHCOOH (440.2 μg/l). a: sample chromatogram, b: magnified bromate chromatogram, c: magnified DCAA chromatogram.

of the weakly and strongly retained components, 11.5 mmol/l Na₂CO₃ was chosen as the eluent throughout. With this eluent, all the 16 analytes could be well separated and determined within 31

min in one injection while the flow rate was kept at 1.0 ml/min (Fig. 2).

Direct large sample loop injection has been proved as an effective method to determine the

Table 3

The concentrations of the analytes in original bottled drinking water samples ($\mu\text{g/l}$)

Analyte	F^-	CH_3COOH	HCOOH	MCAA	BrO_3^-	Cl^-	NO_2^-	DCAA
Original mineral water	18.0	–	58.1	–	–	153	9.1	0.6
Original purified water	6.1	–	33.2	–	–	44.3	11.4	–
Original natural water	38.2	–	53.4	–	0.1	21.1	7.3	0.6
Spiked in tenfold concentrated natural water	500	200	500	200	1	300	100	10
Recovery (% , $n=7$)	103.9	92.3	90.1	92.6	95.5	109.1	100.9	94.8
R.S.D. (% , $n=7$)	3.58	3.27	3.31	2.89	2.68	2.17	1.99	2.18
Analyte	Br^-	ClO_3^-	NO_3^-	PO_4^{3-}	SO_4^{2-}	COOHCOOH	TCAA	I^-
Original mineral water	0.9	0.7	1398	17	6325	42	–	–
Original purified water	–	–	301	19	266	23	–	–
Original natural water	3	0.4	1289	5	4851	44	–	–
Spiked in tenfold concentrated natural water	50	5	15 000	50	25 000	500	200	200
Recovery (%)	103.5	95.3	92.1	103.7	90.6	98.7	93.9	97.1
R.S.D. (% , $n=7$)	1.67	1.98	2.04	1.86	2.09	1.88	2.21	1.91

–, Not detected.

trace level anions in drinking water samples [9]. The high capacity of the AS9HC column allows the introduction of relatively large sample volumes without resolution or overloading problems. Consequently, a 500- μl sample loop was used for the injection.

Anion Atlas electrolytic suppressor could provide excellent performance by significantly decreasing noise when the carbonate was used as eluent. From Fig. 2, it could be seen that the negative peak of water was much diminished. The weakly retained fluoride was far apart from the water dip, and could be exactly detected. With the preconcentration technique, large volume injection and improved baseline, excellent detection limits and linear ranges of the 16 analyte could be obtained (Table 2). The detection limits for BrO_3^- , ClO_3^- , MCAA, DCAA and TCAA were 0.06, 0.08, 0.06, 0.14 and 0.85 $\mu\text{g/l}$, respectively. In addition, excellent reliability could be obtained with the suppressor.

3.3. Application to bottled drinking water samples

The described method was applied to the detection of the 16 compounds in the bottled natural water, purified water and mineral water samples. Firstly, 20 ml drinking water sample was taken

and the pH value of the sample was adjusted to between 10.5 and 11.0 with 0.1 mol/l sodium hydroxide. Then the sample was enriched with the microwave oven for 10 min. After the calibration of the final sample volume, the concentrated solution could be analyzed with IC directly.

From Fig. 3a, it could be seen that 12 compounds including fluoride, formic acid, bromate, chloride, nitrite, DCAA, bromide, chlorate, nitrate, phosphate, sulfate, oxalic acid could be detected in the concentrated natural water samples. From the magnified chromatogram of the concentrated natural water sample (Fig. 3b), it could be seen that there had a significant peak of BrO_3^- in the chromatogram. The concentration of bromate in the concentrated solution was 1.3 $\mu\text{g/l}$. As for chlorinated HAAs, only a little of DCAA could be found (Fig. 3c). Its concentration was 6.5 $\mu\text{g/l}$. No MCAA and TCAA were detected in the solution. The concentrations of the analytes in the three kinds of bottled drinking water were listed in Table 3. In order to confirm the reliability of the developed method in real sample, simulated experiments were conducted by spiking, pretreating and analyzing the tenfold concentrated natural water samples. From Table 3, it can be seen that all the recoveries of the analytes were between

90.1 and 109.1%, which proved that the developed method was available in real sample analysis.

The reason for why there were DBPs in natural water and mineral water but not purified water is because the source water of the bottled natural water and mineral water was from natural lake and underground water, respectively. They had many ions and organic compounds in them. During the manufacturing process, the two kinds of original water were simply treated, neither of them was deionized. However, for the purified water, the source water was deionized and treated with reverse osmosis technique etc. Therefore, the concentrations of organic matters and anions in bottled natural water and mineral water were much higher than those in purified water. During ozonation, more DBPs such as BrO_3^- , DCAA and ClO_3^- were generated. The results were also in accordance with Dojlido's report [3].

It also could be seen from Fig. 3 that the small peak of bromate was much influenced by the big peak of chloride. In the investigated samples, the concentration of chloride was low, chloride could be quickly eluted out from the column. Though the concentration of chloride in the concentrated sample was approximately 200 times higher than that of BrO_3^- , it did not interfere the quantification of BrO_3^- . However, for some bottled drinking water samples, the content of chloride would be very high. If the samples were directly concentrated and determined with the developed technique, the high response of chloride would completely cover the peak of bromate. Dionex OnGuard Ag II cartridge is used to remove chloride by precipitation. With one 1.0 cc Ag cartridge, more than 99.9% of chloride could be removed when the concentration of chloride was less than 400 mg/l [29]. A simulated solution that contained 100 mg/l chloride, 1 $\mu\text{g/l}$ bromate and 5 $\mu\text{g/l}$ of chlorate, MCAA, DCAA, TCAA was treated with the cartridge. The results indicated that 99.99% of chloride could be removed. Bromate was free from the interference of chloride and could be accurately determined. The recoveries for bromate, chlorate, MCAA, DCAA and TCAA were 96.5, 95.8, 97.2, 98.1 and 94.3%, respectively. Consequently, with the pretreatment of the cartridge, the developed technique became more rugged and could be

extended to the determination of trace level of bromate in the drinking water samples with high content of chloride.

4. Conclusions

By employing the microwave based evaporative concentration technique, the developed method was very sensitive for the simultaneous determination of trace level bromate, chlorate, MCAA, DCAA, TCAA, formic acid, acetic acid, oxalic acid, iodide and seven common anions in bottled drinking water. With the treatment of OnGuard Ag cartridge, the method became much rugged and was successfully applied to the detection of the 16 compounds in some bottled drinking water samples.

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