

Occurrence of Haloacetic Acids in Drinking Water in Certain Cities of China

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Objective Since haloacetic acids (HAAs), which are nonvolatile and of high carcinogenic risk, are common species of chlorinated disinfection by-products (DBPs) in drinking water, and little has been known in China, it is necessary to make a survey about the kinds and levels of HAAs in drinking water of the nation. **Method** HAAs were analyzed using gas chromatography with electron capture detector (GC/ECD) and relatively complex pretreatment process of sample was applied. Five main cities in different areas of China were chosen in the survey. **Results** Studies showed that the main species of HAAs in drinking water in China were DCAA and TCAA, ranging from 0.4 µg/L to 12.85 µg/L and from 0.56 µg/L to 10.98 µg/L, respectively. MBAA and DBAA were also detected in one city, ranging from 2.20 µg/L to 4.95 µg/L and 1.10 µg/L to 2.81 µg/L, respectively. Therefore, the contents of HAAs varied, usually no more than 25 µg/L. Based on the acquired data to date, it is known that the concentrations of HAAs in drinking water in China were surely under the limits of Sanitary Standard for Drinking Water Quality (China, 2001). **Conclusion** A wider survey of HAAs in drinking water should be conducted throughout the nation to get adequate data and information, the ultimate aim of which is to control HAAs pollution and keep the balance between microbiological safety insurance and chemical risk control, minimize the formation of DBPs and ensure the safety of water supply at the same time.

Key words: Haloacetic acids (HAAs); Disinfection; Drinking water; Waterworks; Disinfection by-products (DBPs)

INTRODUCTION

Disinfection by chlorine or other chlorine-related reagents is common in water treatment process, though it has been found that haloacetic acids (HAAs), which were firstly found as a nonvolatile and high carcinogenic risk chemical in the middle of 1980s, are a main kind of chlorinated disinfection by-products besides trihalomethane (THMs)^[1-5]. Among them, monochloroacetic acid (MCAA), dichloroacetic acid (DCAA), trichloroacetic acid (TCAA), monobromoacetic acid (MBAA) and dibromoacetic acid (DBAA) are the most common ones detected in drinking water. Usually, they are not biosynthetic and come from reactions between chlorine and natural organic materials (NOM), such as fulvic acid and humic acid, or some species of fatty organics^[6-8].

Abbreviations: HAAs, haloacetic acids; MCAA, monochloroacetic acid; DCAA, dichloroacetic acid; TCAA, trichloroacetic acid; MBAA, monobromoacetic acid; DBAA, dibromoacetic acid; NOM, natural organic materials; DBPs, disinfection by-products.

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On the other hand, DCAA is a metabolic product of many chlorides, such as chloromycetin and hydrochloroaldehyde. There are toxicological tests showing that DCAA may increase occurrence rates of liver cancer in mice or rats. DCAA is also clinically useful in curing some kinds of metabolic diseases or diseases of heart and blood vessel, whose pharmacological effect has been carefully studied^[9].

Several kinds of HAAs are genotoxic and have been identified as rodent hepatocarcinogens. It has been testified that MCAA, DCAA, TCAA, MBAA and DBAA were toxic to rat cecal microbiota, especially to enterococci^[10]. As to their possible toxicity in the body of human beings, a lot should be done to make decisions.

In the newly printed Sanitary Standard for Drinking Water Quality^[11] (issued by Ministry of Public Health, China, 2001), for the first time, the upper limits of DCAA and TCAA are suggested to be 50 $\mu\text{g/L}$ and 100 $\mu\text{g/L}$, respectively. Though they are not routine items required at present, such problem is being paid increasing attention. Because Ministry of Public Health in China is responsible for monitoring and supervising drinking water quality, this standard is truly applicable and practical in drinking water quality management.

Researches concerning DBPs in China are mostly about THMs^[12-14] and not much has been known about HAAs in drinking water except some studies in 1998. DCAA and TCAA were detected and concentration of HAAs in finished water and distribution water were mostly below or around 10 $\mu\text{g/L}$ ^[15].

Other countries have done much more work about HAAs and the first step of research usually is undertaking surveys. For example, in Canadian finished water and distribution water, the concentrations of MCAA were 0.3-9.7 $\mu\text{g/L}$, DCAA 0.2-163.3 $\mu\text{g/L}$, TCAA 0.04-473.1 $\mu\text{g/L}$, MBAA <0.01-9.2 $\mu\text{g/L}$ and DBAA <0.01-1.9 $\mu\text{g/L}$ ^[11]. MCAA, DCAA and TCAA were detected in all samples and the occurrence rate of MBAA and DBAA were 31% and 62%. Surveys in Australia showed that MCAA, DCAA and TCAA were found in both finished water and distribution water and the concentration of MCAA varied from 10 $\mu\text{g/L}$ to 244 $\mu\text{g/L}$, DCAA 1-46 $\mu\text{g/L}$ and TCAA <0.02-14 $\mu\text{g/L}$ ^[16]. It seemed that MCAA in Australia was much higher than DCAA and TCAA.

Content ranges of HAAs in distribution system were reported to be 0.1-222 $\mu\text{g/L}$ in 1991 and 0.2-223 $\mu\text{g/L}$ in 1992 in USA^[17]. The latest survey of disinfection and DBPs in water supply system of different sizes (large, medium and small) in USA provided the concentration ranges of five main kinds of HAAs: MCAA 1.8-4.0 $\mu\text{g/L}$, DCAA 11-51 $\mu\text{g/L}$, TCAA 8-53 $\mu\text{g/L}$, MBAA 0-1.2 $\mu\text{g/L}$ and DBAA 0.5-1.4 $\mu\text{g/L}$, respectively^[18,19]. There were more HAAs in distribution system than in finished water in general (with some exceptions). It was clear that DCAA and TCAA were dominant species in drinking water of USA.

Thus, initial investigations were carried out from 1999 to 2003 in China. Five main cities were chosen as sampling sites, and the kinds and distribution of HAAs in drinking water in different areas of China were presented here.

METHODS

HAAs were analyzed using gas chromatography with electron capture detector (GC/ECD) and the pretreatment was relatively complex. For the limits of equipments and pretreatment process, only a few cities were selected in the survey. With reference to USEPA Standard Method 522, a method with minor changes was used. Oven temperature raising procedure was altered according to actual tests and acidic methanol was used as esterifying agent that was safe and effective. The details were as follows.

Standards and Chemicals

Five single HAAs standards were used to make mixed standard solution of 100 µg/L. Thereinto, chloroacetic acid, dichloroacetic acid and trichloroacetic acid were from Sigma Aldrich, USA. Bromoacetic acid and dibromoacetic acid were produced by Supelco USA. Their purity was all above 97.3%. Reagent-grade H₂SO₄ and Na₂SO₄, tert-butyl-methylaether (METB) (for HPLC, product of Fluka, US), methanol (for GC, product of Tianjin Si You) and 1,2-dibromopropane (for GC, product of Fluka, US) were also used.

Samples Collections

Water samples were collected in glass bottles and bottles must be filled with water. If there was chlorine residual in the sample, certain amount of Na₂S₂O₃ solution should be added in the bottle to consume it before sampling in order to stop the reaction between chlorine and the organic substances in water. As experiments were carried out in different cities for about three years, specific sampling time and places would be pointed out separately when results and discussions were given.

Samples Preparations

Since HAAs were of high boiling points (187°C-210°C), pretreatment was needed before analysis by GC/MS. Extraction reagent was tert-butyl-methylaether (METB) and internal standard was 1,2-dibromopropane. After HAAs turned into methyl acetate (whose boiling points were lower, between 129°C-185°C), they could be analyzed. Fig.1 showed the procedure of water sample preparations clearly.

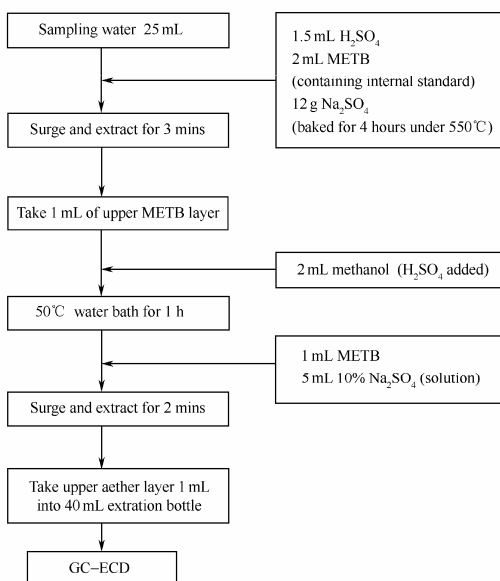
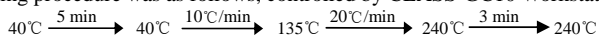


FIG. 1. Procedure of water samples preparations.

Gas Chromatography

GC-ECD analysis was done with a Shimadzu GC-17A with the CLASS-GC10 workstation. J&W DB-1 or HP-5 capillary column (25 m×0.2 mm×0.33 μm) was used in different labs. Maximum volume of sample injector was 5 μL and it was airtight to ensure accuracy. Injector temperature was at 220°C, detector temperature was at 300°C. Oven temperature raising procedure was as follows, controlled by CLASS-GC10 Workstation.



High pure nitrogen (≥ 99.99) was used as carrying gas and its chapter pressure was 65.0 kPa (controlled by electronic pressure-controlling system). Velocity of N₂ was 16.8 cm/s. Injection volume was 1 μL and sampling time was 0.5 min. Flow of tailing gas was 40 mL/min.

Qualitative and Quantitative Analysis

Quality of five HAAs was determined by matching the retention time with standards and quantitative analysis was realized by standard curves, which were obtained by a series standard solutions (11 points, from 0 μg/L to 100 μg/L). Every kind of HAAs had its own standard curve and all the regressive coefficients were above 0.99. For 1,2-dibromopropane was used as internal standard, precision of this method was assured to some extent. From Fig. 2, it could be seen that there was no interference peak in actual water samples, for drinking water was rather clean or pure and there was hardly interfering peak nearby.

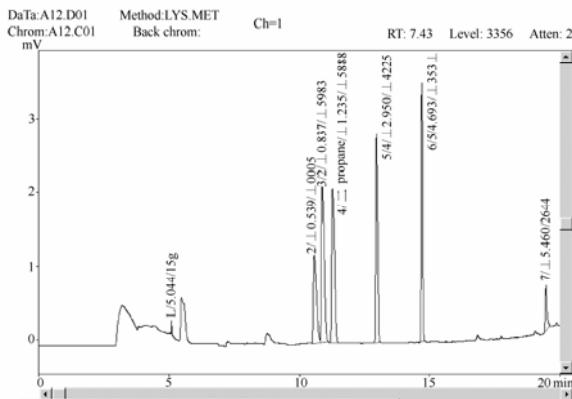


FIG. 2. GC of HAAs and internal standard in an actual sample when DB-1 column was used.

Method Validating

When DB-1 column was used, 20 μg/L standard reagents were added in 15 water samples, and each sample was taken for double analysis, recoveries of four HAAs were got and are listed in Table1. The lowest detection limits (LDLs) were calculated by following formula:

$$\text{LDLs} = 3 * S_b / S$$

Where S_b denotes absolute difference, and S is the slope of standard curve. Namely, LDLs equals to noise 3. When HP-5 column was used, 5 $\mu\text{g/L}$ standard reagents was added in 25 water samples and each sample was taken for double analysis, average recoveries and LDLs of five HAAs were calculated and results were listed also in Table 1. Recoveries were all in the range of EPA regulations and the lowest detection limits (except MCAA) were much higher than possible content of HAAs in actual water. Therefore, the method was practical in drinking water analysis for HAAs.

TABLE 1

Average Recoveries and LDLs of Five HAAs

	Column	MCAA	MBAA	DCAA	TCAA	DBAA
Recoveries	DB-1	-	91.00	91.15	96.68	94.69
(%)	HP-5	123.57	97.46	85.04	91.53	111.97
LDLs	DB-1	-	0.241	0.101	0.119	0.105
($\mu\text{g/L}$)	HP-5	2.273	0.279	0.080	0.020	0.030

RESULTS

Water samples from five cities in different area of China were collected and analyzed from 1999 to 2003. Kinds of source water were investigated, such as water from river or reservoir. In the waterworks, the treatment process consisted of coagulation, sedimentation, sand or anthracite filtration and chlorination. Chlorine was used in pre-oxidation process and disinfection in water facilities was liquid chlorine or chlorine-ammonia. During chlorine-ammonia disinfection, the Cl_2 -to-N ratio was 3:1-4:1. Residual chlorine in finished water usually varied from 0.8-1.2 mg/L (total chlorine), no more than 1.5 mg/L.

City H in Southeast China

Q river is the main source water of city H and the other is T stream, which is a small stream in the northwest of H and feeds to waterworks E as a water source. The other four waterworks are all depending on Q river. Samples from different water facilities and one distribution system were collected in April 2002, July 2002 and February 2003, held in portable icebox and taken to Beijing, then pretreated in the Environmental Sciences and Engineering Department of Tsinghua University.

Liquid chlorine and ammonia are both added before coagulation tank together with aluminum sulfate as flocculant. Results showed DCAA and TCAA were both detected in finished water and distribution system water and their concentrations were not high.

TABLE 2

Concentrations of HAAs in Finished Water From Five Waterworks ($\mu\text{g/L}$)

Seasons	A	B	C	D	E
Spring	4.84	5.65	4.78	5.31	5.43
Summer	3.39	5.14	5.98	6.35	5.16
Winter	3.41	-	-	-	-

From Table 2, it could be seen that the concentration of HAAs in drinking water here was in the range of 3.3-6.4 $\mu\text{g/L}$. During sampling period, TOC of source water was between 4-5.5 mg/L.

Fig. 3 shows concentrations of HAAs (DCAA and TCAA) changing in distribution system of waterworks A in summer. It is clear that at the end of the net which is 3.2 km far from the waterworks, it reached its highest content, increased by 50%. This was because residual chlorine continued to react with organic matters in the water along the pipeline and the concentration of HAAs continuously increased.

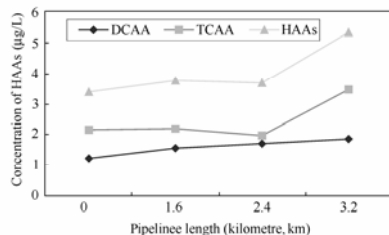


FIG. 3. Concentrations of HAAs in distribution system of city H.

City D and City M in the Southwest Area

People of city D is living on surface water. The area is rich in water. There are several water treatment facilities and drinking water from the Sixth waterworks was carefully studied from October 1999 to November 2000. HAAs analysis system was established in Water Quality Section of Drinking Water Company in city D and then water samples could be prepared and analyzed as soon as they were taken. During this period, TOC was about 2 mg/L.

Chlorine was adopted as a disinfectant during the experimental period and there were two chlorine-adding points, namely before sedimentation and before entering clear water tank. No HAAs existed in source water and two species of HAAs were detected in finished water and distribution water. It proved again that DCAA and TCAA were two main kinds of HAAs in drinking water in China. The concentration of HAAs in finished water varied from 1.64 $\mu\text{g/L}$ (in winter) to 12.85 $\mu\text{g/L}$ (in summer).

Because in winter and summer, the dose of residual chlorine did not vary much, it could be concluded that increasing organic matters in source water and higher temperature in summer had obvious effects on formation of HAAs.

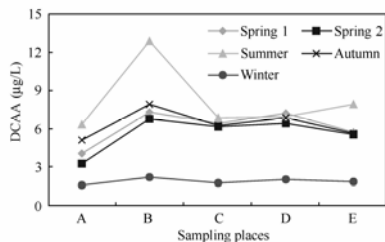


FIG. 4. Change of DCAA in distribution system of city D.

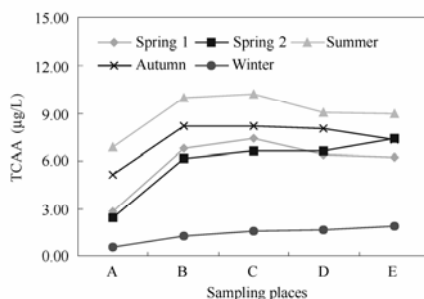


FIG. 5. Change of TCAA in distribution system of city D.

It could be known from Figs. 3 and 4 that the contents of DCAA and TCAA in distribution system were increasing after water left the waterworks. The highest value of DCAA occurred at place B, which was the changing point of net, 5 km away from the facility. Similar to DCAA, the concentration of TCAA rose with the length of pipeline firstly. At the entrance of city, namely place C which is 27 km far and starting from it there is civil net, TCAA was up to its highest value, about 1.5-2.6 times of the finished water. Then the content of TCAA changed a little or just the same. Similar phenomena were present in smaller distribution systems of city H, at the end of the system the highest content of HAAs occurred.

Because the sixth waterworks is in the suburb, far from the city zone, retention time of clear water tank is not long. Before water left the facility, reaction between disinfectant and organic substances were not complete. During the transferring process, the reaction continued. Thus, with the time passing by, DCAA and TCAA reached their highest concentrations far from the facility. Conclusion can be drawn that finished water is not definitely the supervising point of HAAs, for in distribution system, reaction is continuing.

There is another fact worth attention. Along the conventional water treatment processes HAAs were not removed. It appears that once HAAs are formed, it is difficult to get rid of them from the drinking water. Such situation warns of removing precursors of disinfection by-products (DBPs) before DBPs coming into being.

City M is more southern than city D, in another beautiful and fancy province. Water samples from there were collected in July 2002, kept in portable icebox and brought to Beijing. In the lab of Tsinghua University, pretreatment was done as soon as samples reached. Except 1# sample was finished water, the others were all distribution water samples.

TABLE 3

Results of HAAs Content in Water of City M ($\mu\text{g/L}$)

Sample Number	1	2	3	4	5
MBAA	2.20	4.54	ND*	2.31	4.95
DCAA	ND*	2.64	0.43	1.09	1.42
TCAA	4.57	2.26	10.98	9.61	4.00
DBAA	1.17	1.10	1.06	ND*	2.81
HAAs	7.94	10.54	12.47	13.01	13.18

Note. *ND=not detected.

It is the first time that brominated HAAs were detected since the beginning of the research in China. The content of MBAA was up to nearly 5 $\mu\text{g/L}$ and DBAA 2.8 $\mu\text{g/L}$. At the same time, DCAA and TCAA were in the range of 0.4-11 $\mu\text{g/L}$.

City P and City T in North China

Survey in city P was conducted in 2002. Raw water was from reservoir M and there was no prechlorination. Only DCAA and TCAA were found in the finished water, the content of HAAs ranging from 4 $\mu\text{g/L}$ to 10 $\mu\text{g/L}$. Trihalomethane, another kind of DBPs was detected also and whose level in finished water varied from 15 $\mu\text{g/L}$ to 30 $\mu\text{g/L}$, higher than HAAs. Nevertheless, as HAAs possess higher carcinogenic risk^[20], problem of HAAs is worthwhile much more attention.

City T is near city P, but its source water is from a river. Three main waterworks were chosen as objects and samples were collected from January to March in 2003. There is a prechlorination process. Experimental data showed that in finished water, the concentration of HAAs was between 5.4 $\mu\text{g/L}$ and 14.1 $\mu\text{g/L}$. Here there were obviously more TCAA than DCAA, about 65%-75% of total HAAs. No distinct difference was found between the three chosen water facilities. Compared with the other facilities mentioned above, the levels of HAAs in drinking water in city T were a bit higher than those in city P and city H.

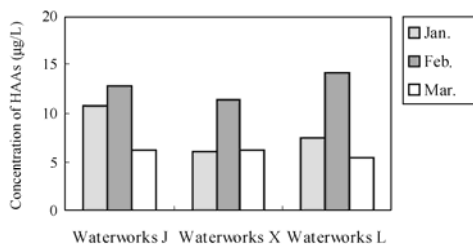


FIG. 6. Contents of HAAs in finished water from three waterworks in city T.

DISCUSSIONS

Based on the data acquired in China so far, the content of DCAA was from 1.58 $\mu\text{g/L}$ to 6.31 $\mu\text{g/L}$ in finished water and from 0.4 $\mu\text{g/L}$ to 12.85 $\mu\text{g/L}$ in distribution water, and the content of TCAA was 0.56 $\mu\text{g/L}$ to 6.89 $\mu\text{g/L}$ in finished water and 1.26-10.98 $\mu\text{g/L}$ in distribution water. MBAA and DBAA were also found in the drinking water, though not as common as DCAA and TCAA. Their concentrations were 2.20-4.95 $\mu\text{g/L}$ and 1.10-2.81 $\mu\text{g/L}$, respectively. The concentrations of HAAs ranged from 3.39 $\mu\text{g/L}$ to 12.30 $\mu\text{g/L}$ in finished water and from 3.68 $\mu\text{g/L}$ to 23.09 $\mu\text{g/L}$ in distribution water.

It is noticeable that raw water of city M is surface water with large catchment area where there are abundant vegetations and the sampling happened to take place in summer when rich nutrients were likely to enter water and at the same time turbidity of water was relatively high. So natural bromide level here may be higher than that in other places and after chlorine was added, bromide was oxidized to bromine, which was then hydrolysed to hypobromous acid. Such kind of acid would react with organic matters in the water and then

form MBAA and DBAA. Since MBAA in the water is more than DBAA, it could be supposed that the concentration of Br⁻ is not very high in the raw water. Because of the long distance between sampling places and lab, it is inconvenient to take more samples for further study. If there is other chances, the content of Br⁻ should be measured, its specific origin should be looked for and its effect on HAAs formation should be studied carefully.

It could be found that the highest concentrations of DCAA and TCAA occurred in Canada^[1], 10-60 times higher than those in China and 4-39 times than those in Australia^[16]. But in Australia, there existed a higher content of MCAA in drinking water^[16]. Situation of USA indicated that its concentration of HAAs was lower than that in Canada but higher than that in Australia and China^[17-19].

Different kinds of disinfectants, distinct dose of disinfectants or different kinds of organic materials and pollutants (which may act as precursors of DBPs) in source water are all possible causes for these results in different countries. For example, in USA, total chlorine might be up to 6.5 mg/L and median value was 2.4 mg/L, and free chlorine maximum concentration 4.4 mg/L and mean 1.1 mg/L^[18,19], much higher than those in China. Because there is not enough chlorine, not many reactions between chlorine and organic chemicals happen, and so less HAAs form.

Higher content of HAAs usually happens during single chlorine disinfection. Survey of five cities in different areas of China could also be proof of this phenomenon. Where chlorine-ammonia disinfection is used (for example, City H), there are relatively lower levels of HAAs. It has been testified that chloramine process could obviously decrease the formation of DBPs and higher Cl₂-to-N ratios might increase the content of chlorinated DBPs^[21,22].

On the other hand, where the source water is not river changing with seasons, such as in city H, the contents of HAAs are only a bit affected by seasons. There is different situation where river changes with season. For example, in city D HAAs content in summer was nearly 8 times higher than that in winter. Because in summer, a lot of humic substances may possibly enter the water body from upstream mountainous areas. So it could be determined that the quality of source water is the key factor for HAAs formation.

CONCLUSIONS

From the above results and discussion, it can be concluded that DCAA and TCAA are the main kinds of HAAs in drinking water of China, though their concentrations are much lower than those in Canada or Australia. MBAA and DBAA have also been found in the drinking water, not as common as DCAA and TCAA. Concentrations of HAAs are usually no more than 25 µg/L in drinking water of China and are certainly under the limits of Sanitary Standard for Drinking Water Quality in China according to the data obtained.

Nevertheless, further information is needed to draw final conclusions about HAAs pollution situation because China covers so large an area and regional diversities are imaginable. Varying synthetic pollutants and NOM in different source waters should be considered and studied carefully because they are possible precursors of DBPs. A wider survey of HAAs in drinking water should be conducted in the nation. Keeping the balance between microbiological and chemical safety is important in water treatment process to minimize the formation of DBPs and at the same time, ensure the safety of water supply^[3]. It is a stringent challenge for engineers and scientists related to problems based on water quality.

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