

# Rapid determination of iodine content in drinking water by isopropyl sensitization and inductively coupled plasma mass spectrometry (ICP-MS)

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**Abstract.** A subject for the rapid determination of iodine content in drinking water by isopropyl sensitization and ICP-MS was established. The samples were dissolved in 1% ammonia solution, <sup>127</sup>I was selected as the mass number to be measured and <sup>159</sup>Tb as an internal standard. At the same time, 4% (V/V) isopropanol was used to enhance aerosol gasification efficiency, 2% NH<sub>3</sub>·H<sub>2</sub>O was used to effectively eliminate memory effects of iodine by cleaning sample for 180 seconds. The results show that, the detection limits of iodine in this method are 0.007 mg·kg<sup>-1</sup> and it is superior to other literature. The standard curve has a good linear relationship and the recovery rates of testing results range are from 96.00% to 112.3%. The content of iodine in standard substance water samples was determined by ICP-MS method. The iodine content in water samples from seven drinking water sources in Lianyungang area was analyzed between 14~21 µg·L<sup>-1</sup>. It was in line with the allowable value of 10~300 µg·L<sup>-1</sup> of the sanitary standard for drinking water, suggesting that the iodine content of drinking water resources in Lianyungang area was in a safe range.

## 1 Introduction

Iodine is closely related to the growth and metabolism of the human body. It is one of the trace elements with important biological effects. Lack, excessive or unbalanced intake of iodine can cause physiological abnormalities and lead to diseases.<sup>[1,2]</sup> The iodine that the human body needs mainly comes from food and water<sup>[3]</sup>. Although most areas of the country are short of iodine or moderate in iodine, there are still about 30 million people in 11 provinces living in high water iodine environment<sup>[4]</sup>. At present, the commonly used method for determining water iodine content is cerium sulfate catalytic spectrophotometry<sup>[5]</sup>, but the detection range is narrow and the limitations are strong. There are many methods for measuring total iodine in water samples, most of which have a narrow detection range and cannot meet the requirements of high-iodine or low-iodine areas<sup>[6,7]</sup>. Lack of standard materials, low content, easy contamination of the matrix, and active and easy oxidation of iodine are the main difficulties in the determination of trace ultra-trace iodine in water.

Inductively coupled plasma mass spectrometry (ICP-MS) is currently recognized as the best multi-element simultaneous analysis technology<sup>[8]</sup>, although the first ionization energy of non-metals such as bromine, iodine,

and arsenic is somewhat high, such as the first ionization of iodine. It can be 10.45 eV, and there is only about 25% ionization in the instrument, but compared with other analytical methods, it still has a relatively high sensitivity<sup>[9]</sup>. At present, ICP-MS determination of iodine is mostly used to detect biological samples. For example, Vanhoeet al<sup>[10]</sup> determine milk powder samples, Larsen et al<sup>[11]</sup> determine food samples, Cox et al<sup>[12]</sup> determine vegetable samples, Buchert et al<sup>[13]</sup> determine milk samples, and Baumann et al<sup>[14]</sup> determine fresh milk, etc., but there are only a few reports on the detection of iodine in water<sup>[15]</sup>.

This experiment established for the first time the method of alkalization of ammonia water medium combined with isopropanol sensitization signal, and inductively coupled plasma mass spectrometry to directly determine trace iodine in drinking water sources. The iodine of water samples of seven drinking water sources in Lianyungang area was analyzed with satisfactory results.

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## 2 Experimental part

### 2.1 Instruments and reagents

electronic balance BS210S (Beijing Sartorius Balance Co., Ltd.); ICP-MS inductively coupled plasma mass spectrometer (ICP-MS) (Bruker ICP-MS M90); Sc standard solution (purchased from the National Standard Material Center);

Iodine standard solution (purchased from the National Standard Material Center); Watsons distilled

water (purchased by Lotte Mart, Cangwu Road, Xinpu District, Lianyungang); nitric acid (merk nitric acid, Germany); iodine standard solution (purchased from the National Standard Material Center); ammonia (excellent grade pure, Sinopharm Chemical Reagent Co., Ltd.)

### 2.2 Instrument

The working conditions of the inductively coupled plasma mass spectrometer are shown in Table 1.

Table 1. Working parameter of ICP-MS.

Working parameter	set value	Working parameter	value
Plasma flow velocity/L/min	16.5	Sampling depth / mm	6.5
Auxiliary gas flow velocity/L/min	2.00	Peristaltic pump rate/ rpm	4
Protective gas flow velocity/L/min	0.20	Stable delay time/s	10
Atomizer velocity/L/min	1.00	residence time/ ms	10000
Scanning times /times	50	Sample interval cleaning time/s	60
Analysis element	<sup>127</sup> I	Total acquisition time/s	20
Radiofrequency power/ kW	1.40		

### 2.3 Water sample collection and processing

Water samples from water sources were taken in cities and towns surrounding Lianyungang City. The locations of the sampling points are shown in Table 2. Use a water collector to take a water sample from below the water surface, put it into a 500mL plastic bottle, and bring it back to the laboratory refrigerator for storage. Before use, the original water sample is centrifuged and filtered, and then acidified or alkalinized.

**2.3.1 Acidification treatment.** Accurately pipette 98.00 mL of filtered water sample and add nitric acid to a 100.00 mL PE volumetric flask to be tested. The final water sample contains 2% nitric acid.

**2.3.2 Alkalization treatment.** Accurately pipette 99.00 mL of filtered water sample and add ammonia water to a 100.00 mL volumetric flask to be tested. The final water sample contains 1% ammonia water.

**2.3.3 Direct treatment.** Take 100 ml of filtered water for testing.

Table 2. Coordinates of sampling Points.

place	Qiantan	Waterworks of Maokou	Waterworks of Haizhou	Waterworks of West Ganyu City	Dayangzhuang	Siying
coordinate	119°13'7.93"E 34°36'45.39"N	119°13'7.84"E 34°36'44.82"N	119°13'7.05"E 34°36'45.77"N	119°13'7.47"E 34°36'44.63"N	119°13'7.47"E 34°36'44.63"N	119°13'7.47"E 34°36'44.63"N

## 3 Results and discussion

### 3.1 Sample medium selection

The signal intensity of iodine in the water sample is related to the medium. In the experiment, the iodine

signal intensity and RSD% in the water sample were used as indicators, and ammonia water and nitric acid were used as the media respectively to compare the effects of the two media on the iodine signal in the water. The results are shown in Figure 1.

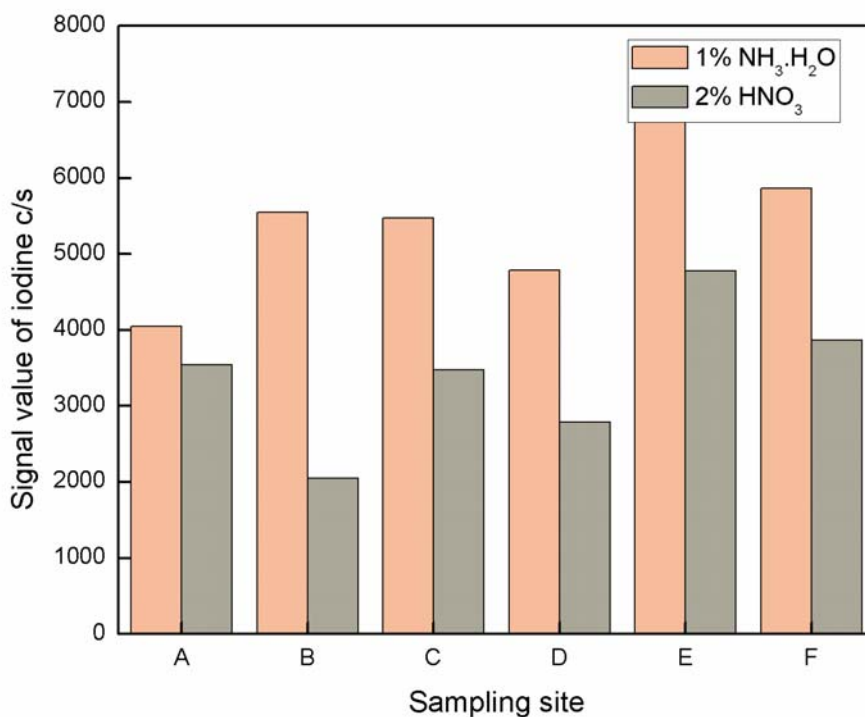


Fig.1 signal value of iodine in 1%NH<sub>3</sub>·H<sub>2</sub>O vs 2%HNO<sub>3</sub> medium

It can be seen from the figure that the signal intensity of <sup>127</sup>I in the ammonia medium is almost double the value of nitric acid. The RSD% value of the water sample in 1% ammonia medium is between 0% and 5%, while the RSD% value in the 2% nitric acid medium is between 20% and 41%. The results indicate the signal stability and the signal sensitivity of iodine are better in ammonia medium than in nitric acid medium.

### 3.2 sensitization

According to the literature report, adding a certain amount of ethanol to the solution by ICP-MS can enhance the signal of iodine. The signal enhancement of iodine by methanol, ethanol and isopropanol in the concentration range of 1%~5% was investigated in the presence of 1% ammonia with the iodine signal value of the concentration of 20 μg·L<sup>-1</sup> <sup>127</sup>I as an index. The results are shown in figure 2.

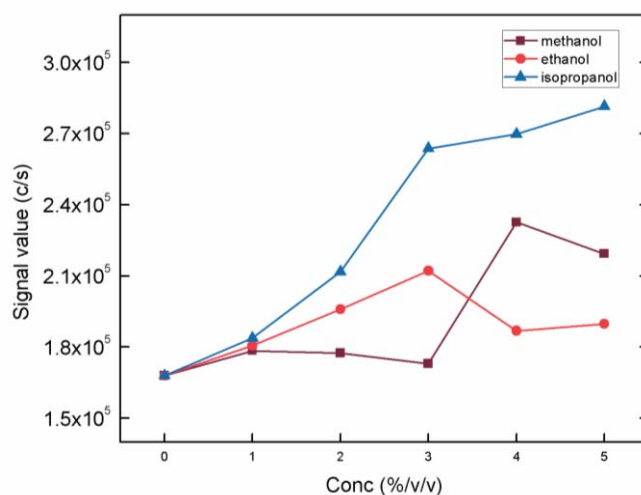


Fig.2 Signal value of 20 μg·L<sup>-1</sup> iodine under different alcohols media

As can be seen from the diagram, adding a small amount of methanol, ethanol, isopropanol can improve the signal sensitivity of iodine. Compared with several alcohols, the signal increase of isopropanol was the most obvious change with the concentration, and the iodine signal value was the largest when the content of isopropanol increased to 4% (V/V).

### 3.3 selection of internal standard

If the sample contains internal standard elements, it will interfere with the calibration of the analysis results, and if the concentration of internal standard elements is increased to counteract the interference, the correction of low signal value will be lost because of the high signal value of internal standard.<sup>[16]</sup> In order to eliminate matrix effect and to correct the influence of instrument response variation on the determination, internal standard elements such as <sup>159</sup>Tb or <sup>89</sup>Y are needed. The results showed that good results were obtained when the concentration of 5.0 μg·L<sup>-1</sup> terbium was added online as internal standard. Its internal standard signal value is about 400000 c/s, which can effectively compensate for the change of instrument condition in the testing process.

### 3.4 optimization of cleaning media and cleaning time

Some problems, such as serious memory effect, great fluctuation of Analytical Signal, poor accuracy and precision, often occur in the determination of iodine in samples by ICP-MS. When iodine is determined in the form of iodide in HNO<sub>3</sub> medium, the analytical error can be as high as 750%.<sup>[16]</sup> The main reason is that the determination of iodine by ICP-MS is complicated by the different forms of iodine, volatility and pollution. In order to reduce the memory effect of iodine, we tried to choose a suitable cleaning solution to clean the injection system. Li Bing et al<sup>[9]</sup> reported that the choice of dilute NH<sub>3</sub>·H<sub>2</sub>O cleaning injection system significantly reduced the memory effect of iodine and bromine. The 100 μg·L<sup>-1</sup> iodine standard solution was chosen as the test solution, and the signal intensity of 100 μg·L<sup>-1</sup> iodine standard solution in pure water is about 4.0 × 10<sup>6</sup> c/s. The cleaning effect and cleaning time of 2% HNO<sub>3</sub>, pure water and 1%~5% NH<sub>3</sub>·H<sub>2</sub>O were compared respectively. The signal intensity of 100 ppb iodine standard solution cleaned by different cleaning solution changed with time. The cleaning effect and cleaning time of 2% HNO<sub>3</sub>, pure water and 1%~5% NH<sub>3</sub>·H<sub>2</sub>O were compared respectively, and the signal intensity of 100 ppb iodine standard solution cleaned by different cleaning solution was changed with time. The results of different cleaning solutions are shown in figure 3.

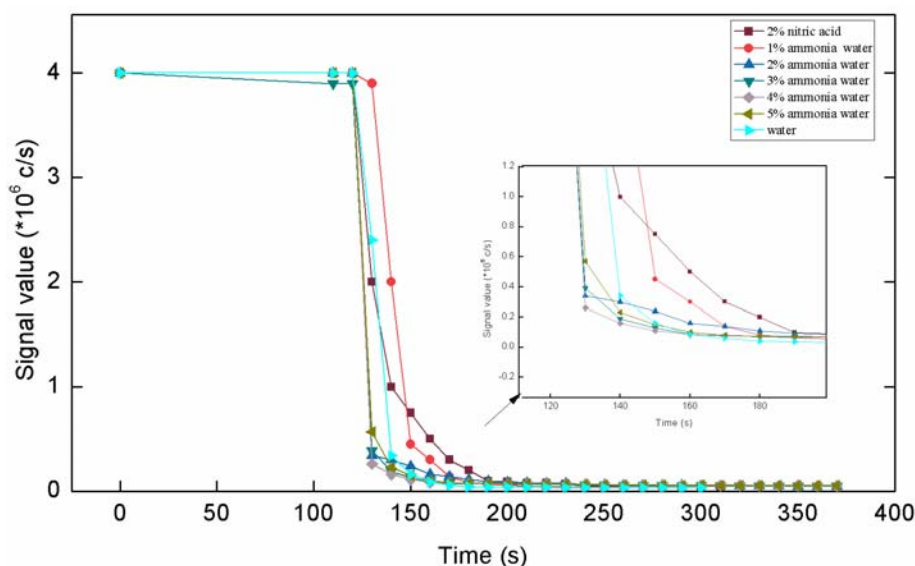


Fig. 3 cleaning effect under different cleaning solutions

As can be seen from Fig.2, all cleaning solutions above 200 seconds can reduce the signal to the value of the blank signal. 5% (V/V) ammonia water and 2% (V/V) nitric acid were the slowest, pure water and 1% (V/V) NH<sub>3</sub>·H<sub>2</sub>O were the second, and 2%~5% (V/V) NH<sub>3</sub>·H<sub>2</sub>O could make the iodine signal decrease rapidly after 130 seconds time cleaning. In the medium of blank ultra-pure water, the blank value of ammonia water is higher than that of nitric acid. It is inferred that the matrix of ammonia water itself causes some interference,

which may explain why the cleaning effect is not improved synchronously after the concentration of ammonia water increases. The results showed that When 2% (V/V) ammonia water was used to clean the injection system, the iodine signal value decreased to lower than 1.0 × 10<sup>5</sup> c/s when the cleaning time reached 180 seconds when the injection tube was inserted into the cleaning liquid, after that, the signal value which changes with time decreases slowly to the size of the blank signal value (~0.5 × 10<sup>5</sup> c/s).

### 3.5 optimization of analytical performance

#### 3.5.1 Standard working curve

Preparation of standard solution: 0, 1.0  $\mu\text{g}\cdot\text{L}^{-1}$ , 2.0  $\mu\text{g}\cdot\text{L}^{-1}$ , 4.0  $\mu\text{g}\cdot\text{L}^{-1}$ , 8.0  $\mu\text{g}\cdot\text{L}^{-1}$ , 10  $\mu\text{g}\cdot\text{L}^{-1}$  standard solutions were

prepared with 100  $\mu\text{g}\cdot\text{L}^{-1}$  iodine standard solution as mother solution and 1% (V/V) ammonia as medium, after washing with 2% (V/V) ammonia for 180 seconds, the working curve was drawn as shown in figure 4.

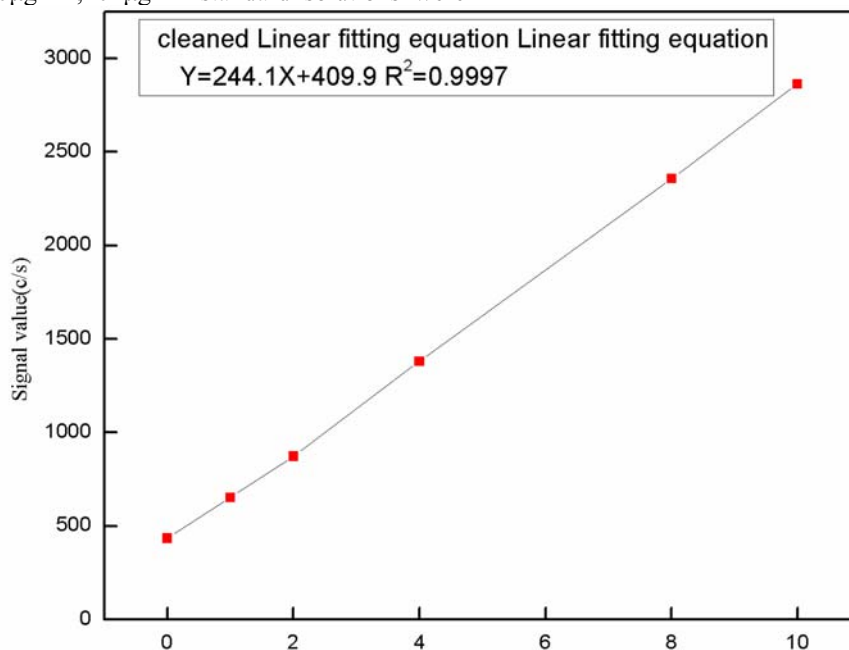


Fig.4 Signal concentration working curve of iodine

As can be seen from the figure 4, the Correlation Coefficient of the iodine standard liquid correlation curve is above the 0.999 ( $R^2=0.9997$ ), which meets the linear requirement.

#### 3.5.2 method detection limit

It was reported that Zuo Shumei et al [17] used 2% (V/V) nitric acid as the medium to determine the iodine content in water samples and the lowest detection limit reached to 0.018  $\mu\text{g}\cdot\text{L}^{-1}$ . Li Bing et Al [9] used 1%(V/V) ammonia as the medium to determine the iodine content in geological samples, also the lowest detection limit

reached to 0.018  $\mu\text{g}\cdot\text{L}^{-1}$ . The detection limit ( $n= 11$ ) of the method is 0.007  $\mu\text{g}\cdot\text{L}^{-1}$ , which is lower than the method reported in the literature.

### 3.6 recovery

50  $\mu\text{g}\cdot\text{L}^{-1}$  iodine standard solution was used as the standard solution, 95.00ml of the test solution was transferred to 100.00 ml volumetric flask, 5.00 ml standard solution was added, and then mixed thoroughly. The results are shown in Table 3.

Table 3. Recovery test of iodine.

Element	Source of water sample	Background value / $\mu\text{g}$	Scalar addition/ $\mu\text{g}$	Recovery value/ $\mu\text{g}$	Recovery rate/%
$^{127}\text{I}$	Qiantan	1.416	0.300	1.744	109.3
	Waterworks of Maokou	1.996	0.300	2.312	105.3
	Waterworks of Haizhou	2.017	0.300	2.341	108.0
	Waterworks of West Ganyu City	1.686	0.300	2.023	112.3
	Dayangzhuang	2.399	0.300	2.687	96.00
	Siyang	2.049	0.300	2.378	109.6

The recovery rate of iodine content in different samples was between 96.0% and 112.3% , which was in accordance with GB/T23942-2009(80% ~ 120%) .

### 3.7 Comparison of test methods

The iodine content in water was determined by the method of this experiment and the national standard method respectively with the iodine standard substance Gbw09113 as the analysis sample. The results are shown in Table 4.

Table 4. The iodine content of each sample is shown in the following figure ( $\mu\text{g}\cdot\text{L}^{-1}$ ).

Determination frequency	1	2	3	4	5	6
National Standard method*/ $\mu\text{g}\cdot\text{L}^{-1}$	10.5	10.2	9.9	8.9	8.6	9.2
This test/ $\mu\text{g}\cdot\text{L}^{-1}$	10.1	10.3	9.8	9.5	8.8	8.9
Allowable range of certified reference material/ $\mu\text{g}\cdot\text{L}^{-1}$	9.5±1.2					

\* GB/T 13025.7-2012

The experimental results show that the iodine content of the two methods is within the allowable range of the

standard material, and can meet the actual determination of water samples. There was no significant difference between the two methods of t-test.

Table 5. Determination of iodine content in various water samples ( $\mu\text{g}\cdot\text{L}^{-1}$ ).

Result	Qiantan	Waterworks of Maokou	Waterworks of Haizhou	Waterworks of West Ganyu City	Dayangzhuang	Siying
Content/ $\mu\text{g}\cdot\text{L}^{-1}$	14.0	20.2	20.4	16.9	24.4	20.7
RSD/%	2.6	3.5	2.1	2.6	3.1	3.2
**	10~300					

\*\* 《Hygienic Standard for drinking water》 (GB5749-2006) Allowable iodine level

### 3.8 sample determination

The content of iodine in water sample was determined by inductively coupled plasma mass spectrometry (ICP-MS) with ammonia solution-isopropanol sensitizing method. The results of the analyzed certified reference materials are within the allowable error range of the

standard values (RSD%<5%) . The iodine content in water samples from seven drinking water sources in Lianyungang was analyzed. The iodine content was between 14.0 and 20.7 $\mu\text{g}\cdot\text{L}^{-1}$ , which was in accordance with the allowable value of 10 ~ 300  $\mu\text{g}\cdot\text{L}^{-1}$  in the hygienic standard for drinking water.

Table 6. contrast of several determination methods of iodine.

Experimental method	Medium	Detection limit ( $\mu\text{g}\cdot\text{L}^{-1}$ )	Linear range
Sulfuric acid catalytic Spectrophotometer <sup>[5]</sup> / $\mu\text{g}\cdot\text{L}^{-1}$	1%Ammonia water	1	1~10 10~100
	2%		
Li Bing <sup>[9]</sup> / $\mu\text{g}\cdot\text{L}^{-1}$	Nitric acid	0.018	/
Zuo Shumei <sup>[17]</sup>		0.018	/
Guo Jingjing <sup>[18]</sup>		0.15	0~500

This experimental method/ $\mu\text{g}\cdot\text{L}^{-1}$	1% (v/v) Nitric acid +4% (v/v) Isopropanol	0.007	0.1~100
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## 4 Discuss

The establishment of a rapid and accurate method for the determination of iodine in drinking water by ICP-MS is of great significance to the safety of iodine in drinking water. Our experimental method is superior to that reported in the literature in Detection limit of  $0.007\mu\text{g}\cdot\text{L}^{-1}$ . Iodine is volatile, especially in acidic solutions to form volatile substances, such as HI or I<sub>2</sub>. Therefore, it is necessary to use TMAH, NaOH and NH<sub>4</sub>OH to stabilize iodine in solution. H. ISNARD et al [19] compared the sensitivity, stability and elution time of iodine in HNO<sub>3</sub>, NaOH and TMAH. It was found that the signal sensitivity in NaOH and TMAH was better than that in HNO<sub>3</sub>. The effects of dilute nitric acid and dilute ammonia water on the signal intensity and the repeatability of the results were compared. The final results show that when 1% (v/v)NH<sub>3</sub>·H<sub>2</sub>O was used as the medium, the iodine signal in the water sample detected by ICP-MS was enhanced by about 1/3, at the same time, the precision of repeated determination is greatly increased. It is inferred that the memory effect of point is decreased by inhibiting the volatilization of HI in iodine measurement under weak base condition. At the same time, the iodine signal in water was improved by Matrix, and the signal values of ethanol, methanol and glycerol were compared. In order to eliminate the memory effect of iodine further, the cleaning solution was selected, and it was determined that the memory effect of iodine could be effectively eliminated by adding 2% (v/v)NH<sub>3</sub>·H<sub>2</sub>O cleaning solution in 180 seconds between the standard sample, the sample to be tested and the sample, the linear relationship of serial standard solution can reach 0.9997. At the same time, the iodine signal in water was improved by Matrix, and the signal values of ethanol, methanol and glycerol were compared. It is confirmed that 4% isopropanol sensitizes the signal effectively. Common interference factors in the quantitative analysis by ICP-MS include the effects of acids, oxides and hydroxide, isotopes, complex ions and double charge ions. The influence of nitric acid on iodine may come from two aspects: When the sample solution contains nitric acid, phosphoric acid and sulfuric acid, it may form cations including ArN<sup>+</sup>, PO<sup>+</sup>, ArP<sup>+</sup>, SO<sup>+</sup>, ArS<sup>+</sup> ClO<sup>+</sup>, ArC<sup>+</sup> et al, these ions interfere with the determination of Si, Fe, Ti, Ni, Ga, Zn, Ge, V, Cr, As and Se. This interference can be eliminated by selecting another isotope ion of the analyte. In addition, iodine is unstable in acidic media. In ICP, the oxides of the metallic elements are completely dissociable, but near the sampling cone, the temperature is slightly lower and the residence time is longer, thus providing an opportunity for reoxidation. The strength ratio of Ce<sup>+</sup> and CeO<sup>+</sup> can be used to estimate the influence of oxide, and the position of sampling cone can be adjusted to reduce the influence of oxide. At the same time, the presence of oxide and hydroxide can interfere with the determination

of other ions, for example, <sup>40</sup>ArO and <sup>40</sup>CaO can interfere with <sup>56</sup>Fe, <sup>46</sup>CaOH can interfere with <sup>63</sup>Cu, <sup>42</sup>CaO can interfere with <sup>58</sup>Ni, etc. Therefore, it is necessary to choose the isotope that is not interfered with when the quantitative analysis. There are <sup>40</sup>Ar<sup>+</sup> interference <sup>40</sup>Ca<sup>+</sup>, <sup>58</sup>Fe interference <sup>58</sup>Ni, <sup>113</sup>In interference <sup>113</sup>Cd<sup>+</sup> and so on. Other interference, mainly compound ion interference and double charge ion interference and so on. The composite ions include, <sup>40</sup>ArH<sup>+</sup>, <sup>40</sup>ArO<sup>+</sup>, etc. For the elements with lower second ionization potential, the existence of double-charged ions will also affect the reliability of the measured values. In this experiment, isopropanol was used for the first time to enhance the sensitivity of iodine signal. The result was better than that reported in literature. The effect of different concentration of methanol, ethanol and isopropanol was compared, the results showed that low concentration of isopropanol had a strong effect on signal sensitizing of <sup>127</sup>I, and the effect of sensitizing was positively correlated with the concentration of <sup>127</sup>I at low concentration ( $\leq 4\%$ ). The mechanism may be related to the concentration of alcohol sensitizers and the number of carbon atoms. With the increase of isopropanol concentration, the inhibition of iodine signal is enhanced, which may be due to the energy consumption in decomposition of organic matter, thus reducing the ionization degree of iodine analyte and reducing the signal. The ionization energy of argon is 15.7 eV, the ionization energy of iodine is 10.08 eV, which is close to the ionization energy of carbon atom is 11.27 eV, that is, the ionization of iodine in the plasma is increased by the transfer of electrons from the more ionized iodine atoms to the carbon and carbon polyatomic ion.

The results show that <sup>127</sup>I is used as the target element and <sup>159</sup>Tb as the internal standard in the determination of iodine in water samples by ICP-MS, when 1% (v/v)NH<sub>3</sub>·H<sub>2</sub>O was used as solvent, 4% (v/v) isopropanol as sensitizer, and 2% (v/v)NH<sub>3</sub>·H<sub>2</sub>O was used as cleaning solution for more than 180 seconds, the method had high precision and good accuracy. The measured data of iodine content in Lianyungang drinking water source were within the standard range ( $10\sim 300\mu\text{g}\cdot\text{L}^{-1}$ ), and the measured results ( $14.01\sim 24.45\mu\text{g}\cdot\text{L}^{-1}$ ) showed that the iodine content in Lianyungang water source was safe and in the low value range.

## 5 Conclusion

The determination of the iodine in water resources can be carried out with an ammonia medium, 4% (v/v) isopropanol as sensitizer, followed by ICP-MS analysis. The quantification limit of this method is  $0.007\mu\text{g}\cdot\text{L}^{-1}$ , which is better than that reported in literature<sup>[5,9,17,18]</sup>. It was shown in this article that adding a certain amount of isopropanol to the solution by ICP-MS can enhance the signal of iodine effectively, better than ethanol as sensitizer reported in literature<sup>[21]</sup>. This method can be used for the determination of trace iodine in water resources.

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