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The Antimony-Cerium Catalytic Photometric Detection Method for High Concentration Iodine in Urine

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Abstract

To establish a antimony-cerium (Sb-Ce) catalytic spectrophotometric method for high-concentration urinary iodine(HUI) detection. The catalytic effect of iodine on the redox reaction of Sb-Ce was utilized. Within the high iodine mass concentration range of 300 μg/L to 1200 μg/L, the optimal sampling volume and water bath temperature range were investigated. Validation experiments including method standard curve linearity, precision, accuracy, and method comparison were conducted. The optimal sampling volume was determined to be 100 μL. For urinary iodine concentrations of 300 μg/L to 1200 μg/L, the correlation coefficients of the fitted standard curve ranged from -0.9993 to -0.9998. The intra- and inter-batch precision RSDs for actual urine samples were 0.6%-1.4% and 1.2%-2.1%, respectively. Measurement results for national first-grade urinary iodine reference materials fell within the reference range, with relative standard deviations of 1.2% and 0.7%. The spiked recovery rates ranged from 99.6% to 103.5%. Method comparison results showed no statistically significant differences. This method exhibits excellent precision and accuracy, making it suitable for HUI determination.

Keywords High urinary iodine concentration·Antimony cerium catalysis·High Concentration Iodine in Urine· Spectrophotometry

Introduction

Iodine is an essential trace element for the human body, playing an irreplaceable role in the synthesis of thyroid hormones, regulation of body metabolism, and neural development. Both iodine deficiency and excess can cause serious health problems[1]. Iodine deficiency can lead to goiter, cretinism, and varying degrees of brain development disorders[2], while long-term iodine excess may induce thyroid autoimmune diseases[3], hyperthyroidism or hypothyroidism, and even increase the risk of thyroid cancer. Therefore, accurately assessing the iodine nutritional status of the population is of crucial significance for guiding scientific iodine supplementation[4], preventing and treating iodine-related diseases, and formulating public health policies. Urinary iodine[5-6] is the most commonly used biological indicator for evaluating recent iodine intake in the human body. The World Health Organization and other international institutions also consider it as one of the preferred indicators for evaluating the iodine nutritional status of the population

[7].

Currently, the classic method for urinary iodine detection, arsenic cerium catalytic spectrophotometry (Sandell-Kolthoff reaction) [8-12], is simple to operate and has low equipment costs, making it suitable for application at the grassroots level. However, the use of the highly toxic substance arsenic trioxide increasingly restricts its application. Inductively coupled plasma mass spectrometry [13-16]has the advantages of high sensitivity, good accuracy, and a wide linear range, but due to its expensive equipment and complex operation. Ion chromatography with pulsed amperometric detection[17] and differential pulse voltammetry[18] are applied in determination of urine iodine.But these are difficult to be widely promoted for large-scale sample monitoring at the grassroots level. Our research group has previously reported an arsenic-free urinary iodine Sb-Ce catalytic spectrophotometric method[19-20], but it is only applicable to the determination of urinary iodine samples below 300 μg/L. With the implementation of iodized salt policies, the increased use of iodine-containing health supplements and contrast agents, the situation of HUI samples in specific monitoring scenarios is increasing. In some high-water-iodine areas in China, there are also high-iodine urine samples in the population's iodine nutritional monitoring. However, the existing spectrophotometric detection methods suitable for grassroots use often face problems such as insufficient linear range, cumbersome dilution in pretreatment, and easy introduction of operational errors when dealing with such high-concentration samples. This may lead to inaccurate assessment of the risk of iodine excess and affect the reliability of large-scale population monitoring data. Therefore, these have become an urgent technical need in the current field of iodine nutritional monitoring and related disease research.

Based on the previous research on the Sb-Ce method for urinary iodine, our research group aims to optimize a detection method with a wider linear range, simple operation, and stable results for high-concentration urinary iodine samples. We would also conduct thorough validation of the methodological performance to provide reliable technical support for the precise assessment of iodine excess in the population's iodine nutritional monitoring system.

Methods

Principle

Iodine can catalyze the redox reaction between potassium antimonyl tartrate and ammonium cerium sulfate. In the solution, Ce^{4+} (yellow) is reduced to Ce^{3+} (colorless), causing the solution to colorless.

$$[SbO(C_4H_4O_6)]^- + 2Ce^{4+} \rightarrow [SbO(C_4H_4O_6)]^+ + 2Ce^{3+}$$

The higher the concentration of iodide ions, the faster the decolorization reaction rate. Under certain reaction temperature and time conditions, the logarithm of the absorbance value of the solution after the reaction is linearly related to the iodine mass concentration. By substituting the absorbance value of the sample solution to be tested into this equation, the iodine mass concentration can be calculated.

Reagents and Solutions

Semi-hydrated stibate of potassium ($KSbC_4H_4O_7 \cdot 1/2H_2O$, analytical purity, Aladdin); Tetra-hydrated ammonium cerium sulfate [$(NH_4)_4Ce(SO_4)_4 \cdot 4H_2O$, analytical purity; State Pharmaceutical Group]; Potassium iodide (KI, superior purity, State Pharmaceutical Group);

Concentrated sulfuric acid (H_2SO_4, superior purity, State Pharmaceutical Group); Sodium hydroxide (NaOH, superior purity, State Pharmaceutical Group); Sodium chloride (NaCl, superior purity, State Pharmaceutical Group); Deionized water was used in the whole study; Analytical reference material for iodine content in human urine (GBW09109m, GBW09110e, National iodine deficiency disease reference laboratory of china). Details were listed as follows:

-Ammonium persulfate solution (1.0 mol/L): Weighed 114.1g of ammonium persulfate, dissolved it in 400 ml of pure water. After mixing evenly and allowing it to return to room temperature, added pure water to make the total volume 500 ml.

-Antimony potassium tartrate solution (2.75 mmol/L): In a 15 ml beaker, weighed 2.86 g of sodium hydroxide, 0.46 g of hemihydrate stibosulfate potassium and 0.05 g of sodium chloride. Dissolved them with 10 ml of pure water, stirred to dissolve until clear and transparent. Transferred and washed the beaker and glass rod with 200 ml of pure water multiple times until the volume reaches 500 ml volumetric flask. Mixed evenly, then slowly added 160 ml of 2.5 mol/L sulfuric acid solution, cooled to room temperature, and made up to the mark with pure water. Mixed evenly and stored in a reagent bottle at room temperature, teady-to-use upon preparation.

-Ammonium cerium(IV) sulfate solution [$c(Ce^{4+})$ = 0.025 mol/L]: Weighed 16.7 g of tetrahydrated ammonium cerium(IV) sulfate and dissolved it in 700 ml of sulfuric acid solution (2.5 mol/L). Diluted with water to a final volume of 1000 ml in a volumetric flask, mixed well, and stored in a brown bottle in a dark place at room temperature.

-Iodine stock solution (100 μg/mL): Weighed accurately 0.1686g of potassium iodate that had been dried at 105℃ - 110℃ until constant weight. Dissolved it in 0.2% sodium chloride solution and made up the volume to 1000ml with 0.2% sodium chloride solution. Stored it in a brown bottle and it could be kept for 6 months at room temperature.

-Iodine standard series solution (300 μg/L- 1200 μg/L): When ready for use, accurately transferred 0.3 ml, 0.4 ml, 0.6 ml, 0.8 ml, 1.0 ml, and 1.2 ml of the iodine standard stock solution [ρ(I) = 100 μg/ml] into 100 ml volumetric flasks respectively. Diluted to the mark with 0.2% sodium chloride solution and mixed well to obtain the mass concentrations of iodine in the standard series solution of 300 μg/L, 400 μg/L , 600 μg/L, 800 μg/L, 1000 μg/L and 1200 μg/L.

Study Subjects

For collecting urine samples, thirty healthy subjects were recruited from the Clinical Physical Examination Center of the Second Affiliated Hospital of Anhui Medical University. All the subjects had no history of thyroid disease, were not taking thyroid medication, and did not suffer from kidney disease. The research protocol was approved by the Ethics Committee of the Second Affiliated Hospital of Anhui Medical University. All urine samples were by subjects, and informed consent was obtained.

Establishment and Validation of Sb-Ce Catalytic Photometric Detection Method for High Concentration Iodine in Urine

Establishment of Sb-Ce Catalytic Photometric Detection Method

In order to obtain the most optimal detection method for iodine in urine using the Sb-Ce catalytic

reaction, we selected the optimal conditions. The method was established by considering the influence of sample size, the relationship between the temperature of a constant-temperature water bath and the reaction time.

An experiment was conducted to investigate the influence of sample volumes of standard series solutions of 25 μL, 50 μL, 100 μL, 150 μL and 200 μL on the reaction time of the system and the correlation coefficient of the standard curve fitting, and an experiment was conducted to investigate the influence of standard series solutions on the reaction time of the system and the correlation coefficient of the standard curve under different reaction temperatures.

Validation of Sb-Ce Catalytic Photometric Detection Method

The method was validated by evaluating linear relationship of the standard curve, precision experiments, accuracy and method comparison experiment. These high concentration urine samples preparation in these experiments were the same as the establishment of the standard solutions.

-Linearity: Within the range of 300 μg/L- 1200 μg/L for the quality concentration of urinary iodine, six independent batches of experiments were conducted. In each batch, six standard points were used. A linear fitting was performed by taking the quality concentration of urinary iodine as the abscissa and the logarithm of the absorbance value as the ordinate.
-Precision experiments: Batch internal and batch inter-laboratory precision experiments were conducted on three actual urine samples with different iodine concentrations, and the mean values and relative standard deviations (RSD) were calculated.
-Accuracy: Six parallel determinations were conducted on the national reference material for urinary iodine (GBW09111b, GBW09112b) within the same batch. The mean values and relative standard deviations (RSD) were calculated. Three actual urine samples with different iodine concentrations were selected. Different concentrations of iodine standard solutions were added to each sample, and the concentrations after addition were within the range of the standard curve. Each sample was determined in triplicate, and the mean concentrations before and after addition were obtained for each sample. The recovery rates of each group after addition were calculated.
-Method comparison experiment: The urine samples were from 2025 iodine deficiency disease monitoring in Yingquan County, Fuyang City, Anhui Province, including 20 re-tested samples of children's urine iodine and 10 re-tested samples of pregnant women's urine. These samples were tested using this method and the WS/T 107.1-2016 "Determination of Iodine in Urine - Part 1: Arsenic-Cerium Catalytic Spectrophotometry" respectively for actual sample detection. The standard curve range for both was 300 μg/L - 1200 μg/L.

Experimental Operation Procedure and Iodine Concentration of Sb-Ce Catalytic Photometric Detection Method for high concentration Urine Iodine Determination

Add 0.10 ml of the iodine standard series solution or the actual urine sample to a glass test tube （15mm×120mm）. The standard series tubes should be arranged in descending order of concentration. Each tube is then added with 1 ml of 1.0 mol/L persulfate ammonium solution, mixed well, and placed in an electrically heated constant temperature rotary drying

oven(DHG-9075A, China, Yiheng Instrument Co., Ltd.) at 100 ℃ for digestion for 60 minutes. After removal, let it cool naturally to room temperature. Each tube is then added with 2.5 ml of 2.75 mmol/L stibium potassium tartrate solution, mixed well, and placed in a 30 ℃ constant temperature water bath(IB20Pro, German, IKA Instrument Co., Ltd.) for 15 minutes. Every 30 seconds, rapidly added 0.3 ml of 0.025 mol/L cerium ammonium sulfate solution to the center of each test tube, mixed immediately by a shaker(MS2 minishaker, German, IKA Instrument Co., Ltd.) , and returned it to the constant temperature water bath. After 20 minutes of reaction with the 1200 μg/L iodine standard tube, used a 1 cm cuvette, with pure water as the reference, detected at a wavelength of 400 nm. Taked readings from each tube per 30 seconds and record the absorbance values by a spectrophotometer(SP-723, China, Shanghai Spectroscopic Instrument Co., Ltd.)(see Fig.1).

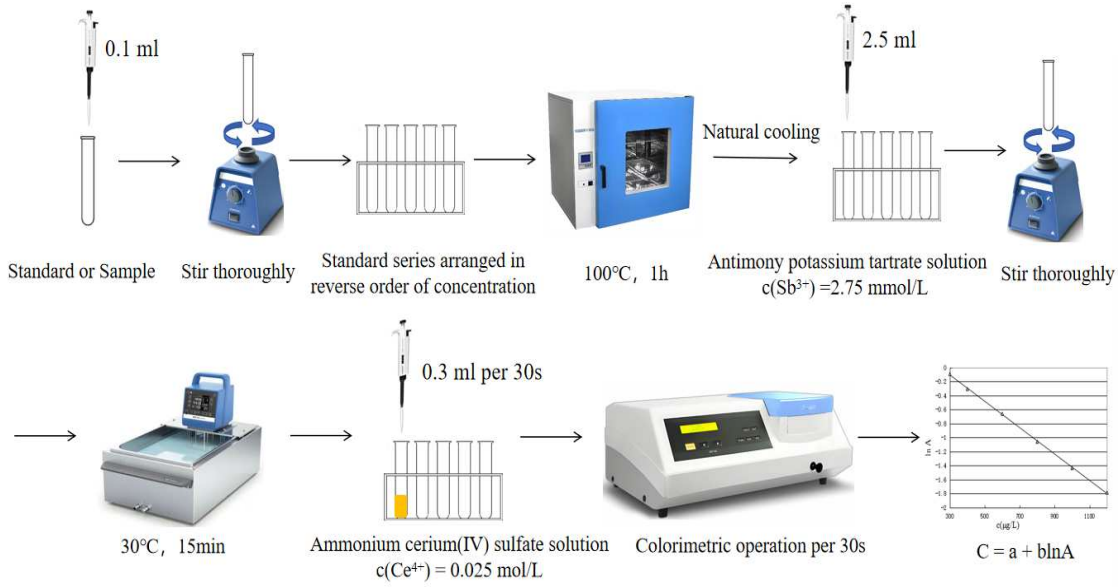


Fig.1 Experimental operation procedure of Sb-Ce Catalytic method for high concentration urine iodine

-Standard curve method: Drawed a standard curve in a semi logarithmic coordinate system. The iodine concentration of the iodine calibration solution was taken as the horizontal coordinate, and the logarithm of the absorbance value was taken as the vertical coordinate. By using the absorbance value of the tested sample, the concentration of urinary iodine sample could be obtained on the standard curve.

-Regression equation method: A linear equation could be obtained by fitting the negative logarithm of the standard series solution concentration to the absorbance value. C = aln A + b (where C was the iodine mass concentration of the standard series solution, a was the slope of the fitted curve, A was the absorbance value, and b was the intercept of the fitted curve). Substituted the absorbance value of the urine sample into the fitted linear equation to calculate its iodine mass concentration.

Statistical Anaylsis

All statistical analyses were conduced by Excel 2019(Microsoft Corporation, Redmond, WA, USA).The Figures were conduced by Powerpoint 2019(Microsoft Corporation, Redmond, WA, USA). All the data about the iodine concentration and the absorbance in the linearity of the standard curve test was presented as mean and relative standard deviation. All the data of recovery rate were presented as the percentage by the following formula:

Recovery rate (%) = (measured value of spiked sample − measured value of original sample) ∗100/ adding standard matter amount

The standard curve regression equation was calculated using Excel 2019. Relative standard deviation (RSD) was the percentage of standard deviation divided by the mean. Using paired t-test to compare the differences between two methods based on different principles, the test was two-sided, and $p<0.05$ is defined as significant.

Quality Control

The RSD requirement for parallel sample precision experiments was less than 10%. The recovery rate of biological samples should be between 75% and 110%; Our choice is 90-110%. The absolute value of the correlation coefficient between the standard curve fitting regression equation C=a+blgA or C=a+blnA should not be less than 0.999. The experimental environment and all experimental equipment should avoid contamination by iodine. Strictly follow the relevant methods for collecting, transporting, and storing samples to avoid contamination from iodinated substances.

Results

Establishment of Sb-Ce Catalytic Photometric Detection Method

Sample volume affects the experiment

An experiment was conducted to investigate the influence of different sample volumes on the reaction time and the correlation coefficient of curve fitting when the reaction temperature was 30℃ and the absorbance value was within a similar range after the reaction with a 1200 μg/L standard solution tube. The results are shown in Table 1. When the sample volume was 25 μL, the correlation coefficient was poor. When the sample volume was 50 μL or more, the standard curve fitting showed a good linear relationship. As the sample volume increased, the reaction time gradually shortened. At 100 μL and 150 μL, the difference in absorbance between the 300 μg/L tube and the 1200 μg/L tube was large, suggesting good sensitivity. Considering the need for manual operation and the comparison color reaction time, this method adopts the optimal sample volume of 100 μL.

Table 1 Sample volume affects the experiment

Reaction time (min)	50	36	20	14	10.5

Sample volume（μL）	25	50	100	150	200
Urine iodine standard series solution (μg/L)	Absorbance value A				
300	0.8825	0.8807	0.9718	0.9796	0.9211
400	0.7633	0.7344	0.7702	0.7831	0.7428
600	0.6004	0.5074	0.5234	0.514	0.5078
800	0.4913	0.3529	0.3508	0.3521	0.3450
1000	0.3645	0.2399	0.2442	0.2444	0.2419
1200	0.2811	0.1694	0.1656	0.1679	0.1726
Linear relationship r	-0.9987	-0.9999	-0.9997	-0.9994	-0.9995

Experiment on the relationship between constant temperature water bath temperature and reaction time

This experiment investigated the influence of the temperature of the constant temperature water bath ranging from 15℃ to 40℃ on the reaction time and the correlation coefficient of the fitting curve. As shown in Table 2, after the reaction in the 1200 μg/L standard solution tube, the absorbance values were within a similar range. The higher the reaction temperature, the faster the reaction rate of the system, and the linear relationship was good. In this experiment, considering the possible influence of indoor environmental temperatures in different seasons on the reaction time, 30℃ was selected as the experimental condition.

Table 2 Experiment on the relationship between water bath temperature and reaction time

Temperature (℃)	Reaction time (min)	The absorbance value urine iodine standard series solution						Linear relationship r
		300	400	600	800	1000	1200	
15	47	0.9543	0.7698	0.5020	0.3285	0.2302	0.1570	-0.9994
20	33	0.9605	0.7746	0.5173	0.3460	0.2331	0.1576	-0.9999
25	26	0.9240	0.7460	0.4964	0.3356	0.2350	0.1639	-0.9995
30	20	0.9198	0.7625	0.4958	0.3321	0.2244	0.1535	-0.9998
35	14	0.8626	0.6956	0.4664	0.3186	0.2171	0.1483	-0.9999
40	9	0.7869	0.6200	0.4131	0.2769	0.1912	0.1273	-0.9996

Validation of Sb-Ce Catalytic Photometric Detection Method

The linear range of the iodine standard series using solution

The iodine standard series using solution was within the range of 300 μg/L to 1200 μg/L. The

reaction time is 20 minutes, and the reaction temperature was 30℃. Six different batches of standard curve series solutions were fitted, and the results are shown in Table 3. The correlation coefficients of curve fitting were between -0.9993 and -0.9998, and the RSD of absorbance values at each concentration among different batches was less than 10%. An example of a standard curve was shown in Fig. 2.

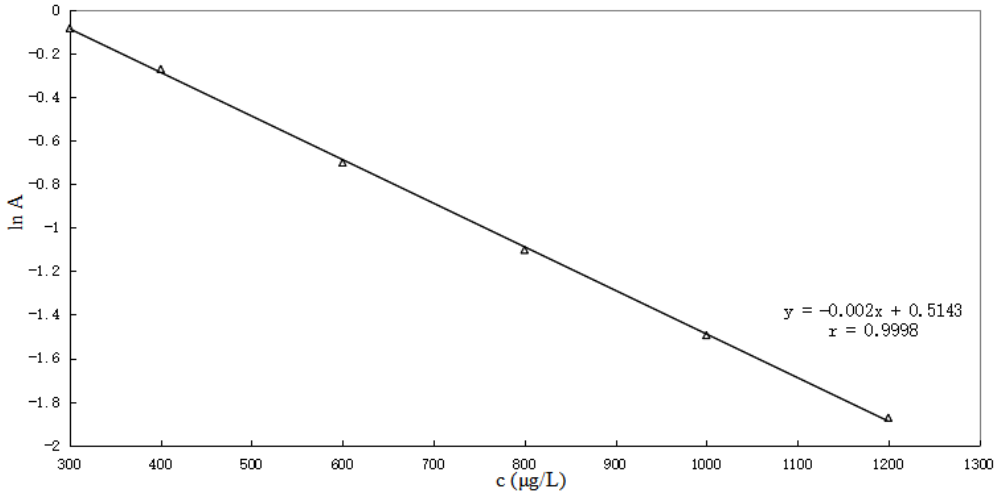


Fig.2 An example of a standard curve of the Sb-Ce catalytic photometric method for high concentration iodine in urine

Table 3 The linear range of the iodine standard series using solution

Standard curve iodine concentration[a] (μg/L)	Measure the absorbance values of different batches						Mean (μg/L)	RSD(%)
	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6		
300	0.9359	0.9198	0.9626	0.9711	0.8827	0.9085	0.9301	3.6
400	0.7491	0.7625	0.7458	0.7766	0.7469	0.7349	0.7526	2.0
600	0.4864	0.4958	0.4872	0.5017	0.484	0.5159	0.4952	2.5
800	0.3208	0.3321	0.3149	0.327	0.3271	0.3467	0.3281	3.3
1000	0.2164	0.2244	0.2079	0.2194	0.2253	0.2382	0.2219	4.6
1200	0.1476	0.1535	0.1436	0.1508	0.1521	0.1678	0.1526	5.4
r	-0.9996	-0.9998	-0.9993	-0.9995	-0.9998	-0.9998	--	--

r coefficient of determination, RSD relative standard deviation

[a] The standard curves are linear over the concentration range of 300 μg/L-1200 μg/L

[b] RSD is less than 10%, which meets the requirements of quality control

Intra-batch precision experiment

Within the iodine mass concentration range of 300 μg/L to 1200 μg/L, six parallel determinations were conducted on three different iodine concentration actual urine samples. The mean values and relative standard deviations were calculated. The results were shown in Table 4. The RSD range

was 0.6% to 1.4%, all of which were less than 5%.

Table 4 Intra-batch precision experiment

Sample number	Testing result (μg/L)						Mean (μg/L)	RSD[a]（%）
1	376	374	370	373	366	362	370	1.4
2	546	551	547	546	546	541	546	0.6
3	979	984	994	998	998	982	989	0.9

[a]RSD is less than 10%, which meets the requirements of quality control

Inter-assay precision experiment

Within the iodine concentration range of 300 μg/L to 1200 μg/L, six independent batches of measurements were conducted on three different iodine concentration actual urine samples. Each batch included three parallel determinations. The mean values and relative standard deviations were calculated. The results were shown in Table 4. The RSD range was 1.2% to 2.1%, all of which were less than 5%, as shown in Table 5.

Table 5 Inter-assay precision experiment

Sample number	The average values of each batch of test results (μg/L)						Mean (μg/L)	RSD[a]（%）
	Batch 1	Batch 2	Batch 3	Batch 4	Batch 5	Batch 6		
1	354	360	373	352	362	355	359	2.1
2	538	551	533	543	537	529	539	1.4
3	974	991	976	976	981	955	976	1.2

[a]RSD is less than 10%, which meets the requirements of quality control

Standard substance determination experiment

Six parallel determinations were conducted on the national urine iodine primary standard substances GBW09111b and GBW09112b. The mean values and relative standard deviations (RSD) were calculated, as shown in Table 6. The determination results all fell within the reference value range, with RSDs of 1.2% and 0.7% respectively.

Table 6 Standard substance determination experiment

Urine iodine reference material number	Reference value (μg/L)	Test results（μg/L）	Mean (μg/L)	RSD(%)

GBW09111b	525±17	530	517	518	530	531	532	526	1.2
GBW09112b	794±28	799	803	802	808	795	809	803	0.7

Actual urine sample spiked recovery experiment

For 3 actual urine samples with different iodine concentrations, different concentrations of iodine standard solutions were added to each sample, and the concentrations after addition were within the range of the standard curve. Each sample was analyzed in triplicate. The average concentrations before and after addition of the samples were obtained, and the spiked recovery rate range was calculated to be 99.6% - 103.5%, as shown in Table 7.

Table 7 Actual urine sample spiked recovery experiment[a]

Sample[b]	The range of urinary iodine baseline measurement results(μg/L, n^c=3)	Background mean value (μg/L, n^c=3)	Adding standard matter amount (μg/L)	Average measured value (μg/L, n^c=3)	Average of recovery rate[d]（%）
Low	319 ~ 321	320	200	527	103.5
Medium	428 ~ 438	433	400	835	100.5
High	519 ~ 531	526	500	1024	99.6

[a] The experiment was repeated for three times

[b]The samples of urine were divided into low-concentration sample group, medium--concentration sample group, and high-concentration sample group according to the iodine concentration after standard addition less than 600 μg/L, from 600 μg/L to 900 μg/L, and more than 1000 μg/L.

[c]The three parallel samples are taken for each experiment to get the average value

[d]The recovery rate should be from 75% to 110%, the result of the work meets the requirements of quality control. Recovery rate (%) = (measured value of spiked sample − measured value of original sample) ∗100/ adding standard matter amount

Method comparison experiment

The current method and WS/T 107.1-2016 "Determination of iodine in urine - Part 1: Arsenic-cerium catalytic spectrophotometric method" [9] (referred to as the standard method) were respectively applied to determine the high iodine concentration of 30 rechecked urine samples from students and pregnant women in the high iodine areas of Anhui Province within the iodine mass concentration range of 300 μg/L - 1200 μg/L. By comparing the test results, as shown in Table 8, there was no statistically significant difference (t = 0.577, P = 0.568).

Table 8 Method comparison experiment

Sample number[a]	The method (μg/L)	The standard method[b] (μg/L)	Sample number[a]	The method (μg/L)	The standard method[b](μg/L)
1	566	561	16	964	960
2	471	463	17	450	450
3	862	876	18	557	571
4	344	332	19	472	486
5	959	947	20	867	870
6	608	605	21	417	402
7	429	429	22	486	474
8	872	884	23	339	335
9	978	986	24	954	945
10	393	408	25	331	335
11	420	415	26	498	498
12	850	859	27	887	887
13	340	333	28	923	937
14	570	565	29	295	316
15	461	452	30	576	590

[a]These urine samples were collected from 2025, during the monitoring of high iodine disease Yingquan County, Fuyang City, Anhui Province. They included 20 re-examination samples of children's urine iodine levels and 10 re-examination samples of pregnant women's urine iodine levels.

[b]WS/T 107.1-2016 "Determination of iodine in urine-Part 1: Arsenic-cerium catalytic spectrophotometric method"

Discussion

This study aimed to establish an accurate and reliable Sb-Ce method for the determination of HUI without arsenic and low toxicity within a high-quality urine iodine concentration range. The optimal sampling volume and reaction temperature of the method were investigated to assess their impact on the experimental results. The method was systematically verified to have excellent linear relationships within the high iodine concentration range of 300 μg/L to 1200 μg/L. The intra-assay and inter-assay precisions of the method were within the RSD range of less than 5%, and the determination results of the national urine iodine reference material were all within the reference value range. The relative standard deviations were 1.2% and 0.7% respectively. The recovery rate range was 99.6% to 103.5%, which met the requirements for the development of biological sample detection methods[21]. Moreover, the comparison and determination results with the currently effective health industry standard detection methods showed no statistically significant differences. This provides reliable technical support for accurately assessing excessive iodine status and improving the iodine nutrition detection system in the population.

Author Contribution All authors contributed to the study conception and design. Material preparation, data collection and analysis were performed by Shudong Xu, Xiuxiu Sun, Ruihan Zong and Meng Su. The samples were collected by Xing Zhong and Ye Ruan. The first draft of the manuscript was written by Shudong Xu. All authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

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Data Availability The data that support the findings of this study were available on request from the corresponding author.

Declarations

Ethics Approval Approval was granted by the Ethics Committee of the Second Affiliated Hospital of Anhui Medical University(No.: YX2025-135)

Consent to Participate Informed Informed consent was obtained from all individual participants included in the study.

Consent for Publication Not applicable.

Competing interests The authors declare no competing interests.

References

[1] Teng W, Shan Z, Teng X, Guan H, Li Y, Teng D, Jin Y, Yu X, Fan C, Chong W, Yang F, Dai H, Yu Y, Li J, Chen Y, Zhao D, Shi X,Hu F, Mao J, Gu X, Yang R, Tong Y, Wang W, Gao T, Li C (2006) Effect of iodine intake on thyroid diseases in China. N Engl J Med354(26):2783–2793.

[2]Qian M. Advances of the effects of iodine on brain development[J]. Chin J Endemiol, 2020,39(12)：923-926.DOI:10.3760/cma.j.cn231583-20191224-00364.

[3]Li S, Zha H, Cui Y, Sun L, Yuan Q. The relationship between excessive iodine during pregnancy and adverse pregnancy complications. Endocrine 88, 203-210 (2025). https://doi.org/10.1007/s12020-024-04134-2.

[4]Hooshmand, S., Yousefian, F., Rahimi, H. et al. Mohammadzadeh M. Dehghani R. Household Salt consumption and urinary iodine levels in Schoolchildren aged 8–10 in Darab City, Iran: 2022. Sci Rep 14, 28349 (2024). https://doi.org/10.1038/s41598-024-78799-0

[5]ICCIDD (2001) Ideal iodine nutrition: a brief nontechnical guide.IDD Newsletter 17:28-9. [3] WHO, UNICEF, ICCIDD (2008) Assessment of iodine deficiency disorders and monitoring their elimination. A guide for programmemanagers, 3rd edn.

[6] Meng Q, Fu M, Yang R, Li JJ, Ji MN,Jin Q, Liu, MX., Zhang, HX., Guo, WX. Sample Size and Optimal Timing for Urine Collection Based on Diurnal Variability of Urinary

Iodine/Creatinine Ratio in Pregnant Women. Biol Trace Elem Res (2025). https://doi.org/10.1007/s12011- 025-04898-2.
[7]Sun L, Wang JH. Excessive iodine and autoimmune thyroid disease[J]. Chin J Endemiol, 2017, 36(11): 856-858. DOI:10.3760/cma.j.issn.2095-4255.2017.11.019.
[8]WHO/UNICEF/ICCIDD. Assessment of iodine deficiency disorders and monitoring their elimination, A guide for programme managers. Third edition. 2007.
[9]National Health Commission of the People's Republic of China. Determination of iodine in urine-Part 1: As^{3+}-Ce^{4+} catalytic spectrophotometry: WS/T 107.1-2016[S]. Beijing:Standards Press of China, 2016.
[10]Zhang YP, Yan YQ, Liu LJ, Sun YN, Li WD, Hua JL, Huang YH, Li XW, Zhao LS. Revised method with low usage amount of arsenic trioxide for testing urinary iodine by As(III)-Ce^{4+} catalytic spectrophotometry using ammonium persulfate digestion[J]. Chin J Endemiol, 2013, 32(01): 95-100. DOI:10.3760/cma.j.issn.2095-4255.2013. 01.026.
[11]Xu SD, Xu X, Zhao LS, Liu TT, Li WD. A study on the determination method of iodine content in urine by enzyme-lobeled instrument[J]. Chin J Endemiol, 2020, 39(03):221-224. DOI:10.3760/cma.j. cn231583-20190710-00194.
[12]Xu SD , Li WD , Zhao LS . The determination method of high concentration iodine in urine measured by automatic biochemical analyzer[J]. Anhui J Prev Med, 2015,21(6):381-383, 406.
[13]Jooste PL, Strydom E (2010) Methods for determination of iodinein urine and salt. Best Pract Res Clin Endocrinol Metab 24(1):77–88.
[14]Macours P, Aubry JC, Hauquier B, Boeynaems JM, Goldman S,Moreno-Reyes R (2008) Determination of urinary iodine by inductively coupled plasma mass spectrometry. J Trace Elem Med Biol 22(2):162–165.https://doi.org/10.1016/j.jtemb.2008.02.003.
[15]National Health Commission of the People's Republic of China. Determination of iodine in urine- Part 2: inductively coupled plasma mass spectrometry method: WS/T 107.2-2006[S]. Beijing: Standards Press of China, 2016.
[16]Xing HT, Fan JB, Jiang DG, Hu SG, Chen M, Cai WH, Lian XW, Su ZJ. A method for determination of urinary iodine by a pure water direct dilution-ICP-MS[J]. Chin J Endemiol, 2025, 44(08):679-683. DOI:10.3760/cma.j.cn 231583 -20250117-00039.
[17]Wafa Hourani, Mohammad Amayreh, Mohammed Khair Hourani,Determination of dopamine in blood serum and urine samples by differential pulse voltammetry at an iodine-coated platinum electrode,PHARMACIA,2023, 70(4): 993-998, https://doi.org/10.3897/pharmacia.70.e110336.
[18]Anna B, Maria K, Wojciech Dr, Ryszard K, Determination of total iodine in serum and urine samples by ion chromatography with pulsed amperometric detection–Studies on analyte loss, optimization of sample preparation procedures, and validation of analytical method,Journal of Chromatography B, 962, 2014, Pages 141-146,https://doi.org/10.1016/j.jchromb.2014.05.022.
[19]Xu SD, Xie JA, Ding G, Xu X, Li WD. A method for detecting urine iodine by antimony cerium catalytic spectrophotometry[J]. Chin J Endemiol, 2024,43(09): 750-754. DOI:10.3760/ cma.j.cn231583- 20240122-00023.
[20]Sun XX, Xie JA, Xu SD, Jiang J, Wei Y, Zong RH, Chen NN, Liu H. A detection method of arsenic free fully automatic online digestion iodine analyzer for urinary iodine[J]. Chin J Endemiol, 2025,44(11):920-924.DOI:10. 3760/cma.j. cn231583-20240925-00353.
[21]The Ministry of Health of the People's Republic of China. Guide for establishing occupational health standards-Part 5: Determination methods of chemicals in biological materials: GBZ/T

210.5-2008[S]. Beijing: People ' s Medical Publishing House , 2008:129-136.