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WS

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

WS/T 107.2-2016

**Determination Part 2: inductively coupled of iodine in
urine-plasma mass spectrometry method
(English Translation)**

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Table of Contents

Foreword
1 Scope
2 Principle
3 Instrument
4 Reagents
5 Solution preparation
5.1 Tetramethylammonium hydroxide solution (0.25% by mass)
5.2 Triton X-100 (1% mass fraction)
5.3 Thinner (mixed solution of 0.25% tetramethylammonium hydroxide and 0.02% Triton X-100)
5.4 Iodine standard solution
5.6 Background urine
5.7 Instrument tuning fluid
5.8 Internal standard solution
6 Collection, transportation and storage of urine samples
7 Analysis steps
8 Calculation of analysis results
9 Method characteristics
9.1 Detection limit and measurement range
9.2 Precision
9.3 Accuracy
10 Key points of quality assurance and control

Foreword

WS/T 107 "Determination of Iodine in Urine" to be released in sections , Divided into the following two parts.

--- Part 1. arsenic-cerium catalytic spectrophotometry;

--- Part 2. Inductively coupled plasma mass spectrometry.

This part is the second part of WS/T 107.

This section is drafted in accordance with the rules given in GB/T 1.1-2009.

This section was drafted. Beijing Centers for Disease Control and Prevention, Fujian Province Center for Disease Control and Prevention, Shandong Province Center for Disease Control and Prevention, Shenzhen Center for Disease Control and Prevention, Peking University School of Medicine, Beijing Shijingshan District Center for Disease Control and Prevention.

1 Scope

This part of WS/T 107 specifies the method for the determination of iodine in urine by inductively coupled plasma mass spectrometry.

This section applies to the determination of total iodine concentration in urine.

2 Principle

After the sample solution is treated with tetramethylammonium hydroxide, the carrier gas (argon) is sent into the inductively coupled plasma torch flame by atomization, and the sample solution the processes of ionization, dissociation, atomization, ionization, etc. are mostly converted into positively charged positive ions, which enter the mass spectrometer through the ion collection system.

It is separated according to its mass-to-charge ratio and detected by the detector. The ion counting rate is directly proportional to the content of the analyte in the sample.

Eliminate matrix effects and achieve quantitative analysis of iodine content in samples.

3 Instrument

3.1

Inductively coupled plasma mass spectrometer.

3.2

Electronic balance (sensitivity 0.1mg).

4 Reagents

4.1

Pure water (H₂O, Mr = 18.0), resistivity greater than 18.0 Mohm · cm or deionized water (in accordance with GB/T 6682 level 1 water regulations).

4.2

Tetramethylammonium hydroxide [(CH₃)₄NOH, 25% by mass], electronic grade.

4.3

Triton X-100 (C₃₄H₆₂O₁₁, Mr = 647.0), reagent grade.

4.4

Potassium iodate (KIO₃, Mr = 214.0), reference reagent or standard reagent.

5.1 Tetramethylammonium hydroxide solution (0.25% by mass)

Take 1mL of tetramethylammonium hydroxide (4.2) and dilute it to 100mL with pure water (4.1).

5.2 Triton X-100 (1% mass fraction)

Take 1mL of Triton X-100 (4.3) solution and dilute it to 100mL with pure water (4.1).

5.3 Thinner (mixed solution of 0.25% tetramethylammonium hydroxide and 0.02% Triton X-100)

Before use, take 10mL of tetramethylammonium hydroxide (4.2) and 20mL of Triton X-100 (5.2), and dilute with pure water (4.1) to 1000mL.

WS/T 107.2-2016

5.4 Iodine standard solution

5.4.1

Iodine standard stock solution [$\rho(I) = 1000.0 \text{ mg/L}$]. Weigh accurately 0.1686g of potassium iodate dried at 105 ° C to 110 ° C to a constant weight, dissolve with pure water, and use Make up the volume of pure water to a 100mL brown volumetric flask. This solution can be stored in a refrigerator (4 ° C) for 8 months.

5.4.2

Iodine standard intermediate solution [$\rho(I) = 10.0 \text{ mg/L}$]. accurately remove 1,000mL of iodine standard stock solution (5.4.1) and place it in a 100mL volumetric flask, using 0.25 % Tetramethylammonium hydroxide solution (5.1) to volume. This solution can be stored in a refrigerator (4 ° C) for 6 months.

5.4.3

Iodine standard solution [$\rho(I) = 1.0 \text{ mg/L}$]. Accurately remove 10.000mL of iodine standard intermediate solution (5.4.2) and place it in a 100mL volumetric flask, using 0.25% Make up to volume with tetramethylammonium hydroxide solution (5.1). This solution can be stored in a refrigerator (4 ° C) for 3 months.

Accurately transfer 0.001mL, 1.000mL, 5.000mL, 10.000mL iodine standard solution (5.4.3) and 1.50mL, 3.000mL, 6.000mL the iodine standard intermediate solution (5.4.2) was

placed in a 100mL volumetric flask, and the volume was adjusted to the mark with 0.25% tetramethylammonium hydroxide (5.1).

The quasi-series concentrations were 0.0 µg/L, 10.0 µg/L, 50.0 µg/L, 10.0 µg/L, 150.0 µg/L, 30.0 µg/L, and 600.0 µg/L.

5.6 Background urine

A clear urine sample with an iodine content below 70 µg/L.

5.7 Instrument tuning fluid

According to the requirements of the instrument operating instructions, take an appropriate amount of instrument tuning solution. It is recommended to use a mixed tuning solution lithium (Li), yttrium (Yttrium), cerium (Ce), thallium (Ti), and cobalt (Co) at a concentration of 10 µg/L.

5.8 Internal standard solution

The concentration of the standard substance of tellurium single element solution is 100 µg/mL. Before use, dilute with tetramethylammonium hydroxide solution (5.1) to the corresponding concentration according to the sensitivity of the instrument.

Note: If the sample injection volume and internal standard injection volume are 20. 1, the concentration of tellurium in the internal standard is recommended to be 3.0 mg/L; if the sample injection volume and internal standard injection volume are at 1. 1, the concentration of tellurium in the internal standard is recommended to be.200 µg/L.

6 Collection, transportation and storage of urine samples

Collect no less than 5mL of urine and place it in a polyethylene plastic or glass test tube with a tightly sealed mouth to prevent evaporation. Urine samples are collected and shipped on site There is no need to consider special storage conditions during the transfer process, it can be stored for 2 weeks at room temperature; the sample can be stored for 2 months at 4 ° C; polyethylene plastic Store the sample, and it can be stored for 4 months at -20 ° C after being sealed.

7 Analysis steps

7.1

Main reference conditions of the instrument. radio frequency (RF) power is 1320W; carrier gas flow rate is 1.10L/min; sampling depth is 7mm; atomization the room temperature is 2 ° C; the sampling cone and interception cone are nickel cones; the atomizer is a high-salt or concentric atomizer.