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WS

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

WS/T 635-2018

**Determination of urinary arsenic speciation - liquid
chromatography-atomic fluorescence spectrometry
(English Translation)**

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Foreword

This standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard was drafted. Beijing Municipal Center for Disease Control and Prevention, Chongqing Municipal Center for Disease Control and Prevention, Guangdong Provincial Center for Disease Control and Prevention, Anhui Provincial Center for Disease Control and

Prevention, Beijing Chaoyang Hospital, Shanxi Provincial Center for Disease Control and Prevention.

1 Scope

This standard specifies the determination of arsenic in urine (arsenite [As (III)], arsenate [As (V)], monomethyl arsenic (MMA) and Liquid chromatography-atomic fluorescence method for methyl arsenic (DMA).

This standard applies to urinary arsenic species (arsenite [As (III)], arsenate [As (V)], monomethyl arsenic (MMA) and dimethyl Determination of arsenic (DMA)).

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document.

For undated references, the latest edition (including all amendments) applies to this document.

GB/T 6682 Analytical laboratory water specifications and test methods

3 Principle

The urine sample is diluted in a certain ratio and separated by liquid chromatography. The separated target compound is in an acidic medium and potassium borohydride.

(KBH₄) reaction, formation of gaseous arsenic compounds, determination by atomic fluorescence spectrometer, retention time qualitative, peak height or peak area Draw a standard curve method to quantify.

4 Instruments

4.1

Glassware should be soaked in nitric acid solution (1 4) for 24 h, rinsed repeatedly with water, and finally rinsed with pure water.

4.2

Liquid Chromatography-Atomic Fluorescence Spectrometer.

4.3

pH meter (accuracy 0.01).

4.4

Electronic balance (sensing 0.1 mg).

4.5

Ultrasonic cleaner.

4.6

Centrifuge, speed ≥ 8000 r/min.

4.7

Pure water preparation instrument.

5 Reagent

5.1

Experimental water and reagent requirements. Unless otherwise stated, the reagents used in this method are excellent grade pure, pure water is the first grade specified in GB/T 6682 water.

5.2

Hydrochloric acid (HCl, $\rho = 1.19$ g/mL).

5.3

Potassium hydroxide (KOH).

5.4

Potassium borohydride (KBH_4).

5.5

diammonium hydrogen phosphate $[(\text{NH}_4)_2\text{HPO}_4]$.

5.6

Formic acid (HCOOH , 99%).

5.7 Reference material. Arsenite (AsO_3)

3-), arsenate (AsO_4

3-), monomethyl arsenic ($\text{CH}_3\text{AsO}_3^-$), dimethyl arsenic ($\text{C}_2\text{H}_5\text{AsO}_2$) all are certified standard substances.

6.1 Current Carrying, Hydrochloric Acid Solution (7.93)

Measure 70 mL of hydrochloric acid and dilute to 1000 mL with pure water.

6.2 reducing agent (20 g/L potassium borohydride solution - 3.5 g/L potassium hydroxide solution) Weigh 3.5 g of potassium hydroxide dissolved in 900 mL of pure water, add 20 g of potassium borohydride, dilute to 1000 mL with pure water, mix uniform.

6.3 Formic acid solution (10 90)

Dilute 10 mL of formic acid to 100 mL with pure water.

6.4 Mobile phase (15 mmol/L diammonium phosphate solution)

Weigh 1.98 g of diammonium hydrogen phosphate dissolved in 1000 mL of pure water, adjust the pH to 6.0 with formic acid solution (10 90), ultrasonic degassing 5 min, spare.

6.5 Arsenic Form Standard Solution Preparation

6.5.1

Standard storage solution of arsenic form [$\rho(\text{As})=10.0 \text{ mg/L}$]. accurately weigh the appropriate amount of arsenite, arsenate, monomethyl arsenic and dimethyl Four kinds of arsenic standard materials of arsenic, prepared with pure water to prepare arsenic form standard stock solution with arsenic concentration of 10.0 mg/L, placed at 4 °C Store in the refrigerator.

6.5.2

Arsenic form mixed standard solution [$\rho(\text{As})=1.0 \text{ mg/L}$]. accurately transfer 10.0 mg/L of four kinds of arsenic single standard stock solutions respectively Place 1.00 mL in a 10 mL volumetric flask and dilute to volume with pure water.

6.5.3

Arsenic form mixing standard use series solution [$\rho(\text{As})=0\sim100.0 \mu\text{g/L}$]. accurately transfer a certain amount of 1.0 mg/L arsenic form Standard solution, diluted with pure water to 0.0 $\mu\text{g/L}$, 2.0 $\mu\text{g/L}$, 5.0 $\mu\text{g/L}$, 10.0 $\mu\text{g/L}$, 50.0 $\mu\text{g/L}$, 100.0 $\mu\text{g/L}$ the arsenic form mixed standard series uses the solution, which is currently used.

7 Sample collection, transportation and storage

Collect not less than 10 mL of urine, place it in a clean polypropylene plastic or glass test tube and seal tightly to prevent evaporation. Collecting urine on site After the sample is stored and transported at 4 °C; the sample should be measured as soon as possible and stored at 4 °C for 30 d.

8.1 Pretreatment of the sample

Take 1 mL of uniform urine sample diluted 3 times with pure water, filter with 0.45 μm water filter and measure on the machine. If there is a precipitate in the urine sample, the urine sample was centrifuged for 10 min (8000 r/min), and a certain amount of supernatant was taken according to the above method. If the arsenic form concentration in the sample exceeds the upper limit of the standard curve is determined by diluting the urine sample to a certain ratio.