

ICS 13.100
CCS C 60



**NATIONAL OCCUPATIONAL HEALTH STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GBZ/T 303-2018

Replacing WS/T 18-1996

**Determination of lead in urine -- Graphite furnace atomic
absorption spectrometry method**

尿中铅的测定 石墨炉原子吸收光谱法

(English Translation)

Issued on: August 16, 2018

Implemented on: January 1, 2019

Issued by: National Health and Family Planning Commission

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Foreword

This standard is formulated in accordance with the Law of the People's Republic this standard replaces WS/T 18-1996 "Method for the determination of lead in urine by graphite furnace atomic absorption spectrometry".

Compared with WS/T 18-1996, the main changes are as follows.

----The operating conditions of the instrument are changed to the reference conditions for instrument operation;

---- Improved matrix modifier, standard preparation, and measurement range;

---- Added the requirement for sample dilution.

This standard is mainly drafted by. China Center for Disease Control and Prevention, Occupational Health and Poison Control Institute, Guangdong Provincial Occupational Disease Prevention and Treatment Institute, Shandong Provincial Institute of Labor Health and Occupational Diseases, Jiangsu Provincial Center for Disease Control and Prevention.

1 Scope

This standard specifies the graphite furnace atomic absorption method for the determination of lead in urine.

This standard applies to the determination of lead in urine in occupational exposure personnel.

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.

General rules for biological monitoring of GBZ/T 295 occupational population GB/T 6682
Analytical laboratory water specifications and experimental methods

3 Principle

Urine sample (hereinafter referred to as urine sample) with palladium chloride matrix modifier, measured by graphite furnace atomic absorption spectrometry at 283.3 nm
Determine the concentration of lead.

4 Instruments

4.1

Covered polyethylene plastic bottle, 100 mL.

4.2

stoppered polyethylene centrifuge tube, 1.5 mL.

4.3

Volumetric flask, 10 mL.

4.4

Microsampler,.200 μ L, 1000 μ L.

4.5

Atomic absorption spectrophotometer with graphite furnace, background correction and lead hollow cathode lamp. Refer to Table 1 for instrument operation reference conditions.

Table 1 Instrument operation reference conditions

step

temperature

Heating time

Residence time

Carrier gas flow

mL/min

Dry 90 1 30 250

Dry 120 15 30 250

Ashing 800 10 20 250 Atomic 1900 0 5 0 Clear 2100 1 3 250

5 Reagent

5.1

Experimental water. According to GB/T 6682.

5.2

Nitric acid, $\rho_{20}=1.42$ g/mL, excellent grade pure.

5.3

Nitric acid solution, 1% (volume fraction).

5.4

Matrix modifier. 0.33 g of palladium chloride (excellent grade) is slightly soluble in 10 mL of nitric acid, shake well, and diluted to 500.0 mL with water. Storage Stored in a brown reagent bottle and stored in the refrigerator for one year.

5.5

Normal human urine. normal human urine that does not touch lead.

5.6

Lead standard solution. The lead standard solution standard substance with the national standard substance certificate number is used.

5.7

Lead Standard Application Solution. Dilute the lead standard solution to 1.0 $\mu\text{g/mL}$ lead standard application solution with 1% nitric acid.

6 Sample collection, transportation and storage

6.1

Sample collection. Collect about 50 mL of urine sample once in a polyethylene plastic bottle. After measuring the specific gravity as soon as possible, add in proportion to 1% Nitric acid, mix well.

6.2

Sample blank. Take 5 mL of 1% nitric acid in a plastic tube with a blank.

6.3

Samples and sample blanks should be placed in a clean container and shipped under refrigerated conditions. It can be stored in the refrigerator at -18 ° C for 1 month.

7.1 Preparation of the standard series of lead working curves

Take 7 10 mL volumetric flasks and add 0.00 mL, 0.20 mL, 0.40 mL, 0.80 mL, 1.20 mL, 1.60 mL, 2.00 mL, respectively.

Lead standard application solution, each with palladium chloride matrix modifier to 10 mL, configured to be 0.0 µg/L, 20.0 µg/L, 40.0 µg/L, 80.0 µg/L, 120.0 µg/L, 160.0 µg/L, 200.0 µg/L lead standard series, see Table 2.

Table 2 Preparation of the standard series of lead working curves

Pipe number	0	1	2	3	4	5	6	Lead standard application solution /mL	0.00	0.20	0.40	0.80	1.20	1.60	2.00	Matrix improver /mL	10.0	9.80	9.60	9.20	8.80	8.40	8.00	Lead concentration/(µg/L)
	0.0	20.0	40.0	80.0	120.0	160.0	200.0	Before the injection, the lead standard series and the normal human urine sample were processed in the same ratio to prepare a standard series of lead working curves.																

7.2.1 Lead working curve standard series pretreatment method

0.2 mL of each standard solution was added to a stoppered polyethylene centrifuge tube to add 0.2 mL of normal human urine, and the mixture was mixed for measurement.

7.2.2 Sample pretreatment method

The acidified urine sample was taken out of the refrigerator, and after returning to room temperature, shake well. Pipette 0.2 mL into a stoppered polyethylene centrifuge tube 0.2 mL palladium chloride matrix modifier, mixed for measurement. When the sample concentration exceeds the linear range, the sample should be appropriately diluted and injected.

When diluting the sample, be aware that the sample, blank, and standard series of treatment methods should be consistent. The measurement results do not need to calculate the dilution factor.

7.2.3 Blank Preprocessing Method

0.2 mL of 1% nitric acid was added to a stoppered polyethylene centrifuge tube to add 0.2 mL of palladium chloride matrix modifier, and the mixture was mixed for measurement.