

ICS 13.100  
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**NATIONAL OCCUPATIONAL HEALTH STANDARD OF THE  
PEOPLE'S REPUBLIC OF CHINA**

**GBZ/T 310-2018**

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**Determination of 1-bromopropane in urine -- Headspace-gas  
chromatography method**

尿中1-溴丙烷的测定 顶空-气相色谱法

**(English Translation)**

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### Foreword

This standard is formulated in accordance with the Law of the People's Republic this standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This standard is mainly drafted by. Wuxi Eighth People's Hospital (Wuxi Occupational Disease Prevention and Treatment Hospital), Jiangsu Provincial Center for Disease Control and Prevention, Tianjin Center for Disease Control and Prevention, School of Public Health, Fudan University, School of Public Health, Peking University.

### 1 Scope

This standard specifies headspace-gas chromatography for the determination of 1-bromopropane in urine.

This standard applies to the determination of 1-bromopropane in urine of 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

### 3 Principle

Urine sample (hereinafter referred to as urine sample) is added with anhydrous sodium sulfate, after headspace treatment, separated by gas chromatography column, hydrogen flame ionization test the detector is detected by the retention time and quantified by the external standard method.

### 4 Instruments

#### 4.1

Electronic analytical balance (one in ten thousand).

#### 4.2

Volumetric flask, 10mL.

#### 4.3

Headspace bottle, 10mL.

#### 4.4

micro syringe, 10  $\mu$ L.

#### 4.5

Automatic Headspace Sampler.

#### 4.6

Gas Chromatograph, Hydrogen Flame Ionization Detector.

### 5 Reagent

5.1 1-bromopropane, chromatographically pure.

#### 5.2

Methanol, chromatographically pure.

#### 5.3

anhydrous sodium sulfate, analytically pure.

5.4 1-Bromopropane standard stock solution. Add about 5 mL of methanol to a 10 mL volumetric flask and accurately add 20.0  $\mu$ L with a micro syringe.

Bromopropane (chromatographically pure, 1  $\mu$ L of 1-bromopropane having a mass of 1.3537 mg at 20 ° C), diluted to the mark with methanol, this solution was 2.71 mg/mL Standard stock solution of 1-bromopropane.

5.5 1-Bromopropane standard application solution. dilute 1-bromopropane standard stock

solution with methanol to prepare a concentration of 10.0 µg/mL, 100 µg/mL Standard application solution.

## 6 Sample collection, transportation and storage

### 6.1

According to GBZ/T 295 for urine sample collection, the urine sample should be  $\geq 50$  mL after the collection of the glass bottle.

### 6.2

Sample transportation. Add 2g anhydrous sodium sulfate to the 10mL headspace bottle, take 4mL of urine from the collected urine sample, add the headspace bottle, and press the headspace bottle.

Refrigerated after the cover.

### 6.3

Sample storage. The sample has a shelf life of 7d at -20 °C.

## 7 Analysis steps

### 7.1

Instrument Operation Reference Conditions. Gas Chromatograph. The column is a FFAP capillary column, 30 m x 0.32 mm x 0.25 µm; the column temperature is initial Warm at 35 °C for 3.5 min, ramp up to 120 °C at 25 °C/min for 1 min; vaporization chamber temperature is 200 °C; hydrogen flame away the sub-detector temperature was 250 °C; the carrier gas (nitrogen) flow rate was 1.00 mL/min; the split ratio was 1.8. Automatic headspace sampler. headspace bottle Heating temperature is 70 °C; heating time is 20 min; shaking rate is 250 rpm; syringe temperature is 80 °C; injection volume is 0.5 mL (gas body).

### 7.2

Preparation and determination of the working curve. 4 mL of control urine was added to the 10 ml headspace bottle (no 1-bromopropane contact population mixed urine) Liquid), add 2g anhydrous sodium sulfate, add standard application solution to make standard series with concentration of 0µg/L~1000µg/L, compact top Empty caps, put into the automatic headspace sampler, refer to the operating conditions of the instrument, each series is measured in parallel 3 times, calculate the concentration of 1-bromopropane Area average. The working curve is plotted with the concentration as the abscissa and the peak area average as the ordinate.

### 7.3

Sample processing and determination. Place the headspace bottle into the automatic headspace sampler, and follow the same operation curve drawing method. Sample blank, sample the product solutions were measured in parallel for 3 times, and the retention time was qualitatively determined. After calculating the peak area average, the 1-bromopropane concentration was obtained from the working curve. If sample the concentration of the analyte to be tested exceeds the measurement range, and can be measured by dilution with blank urine, and the calculation is multiplied by the dilution factor.

## 8 Calculation

### 8.1

Calculate the concentration of 1-bromopropane in urine according to formula (1).

$$OCC = (1)$$

In the formula.

C- concentration of 1-bromopropane in urine in micrograms per liter (g/L);

C<sub>0</sub> - The concentration of 1-bromopropane in the urine calculated by the regression equation in micrograms per liter (g/L).

### 8.2

Correct the sample results according to the specific gravity correction method.

## 9 Description

### 9.1

The detection limit of the method is 5.0 µg/L, and the lower limit of the method is 17.0 µg/L; the method range is 17.0 µg/L to 1000 µg/L.

The precision of the method ranged from 4.8% to 6.6% (n=6). The recoveries of the method were in the range of 96.8 % to 104.8 % (the spiked concentration was 100 µg/L, 200 µg/L, 500 µg/L).

### 9.2

During the sample collection and preservation process, the loss of 1-bromopropane should be prevented, and the headspace cap should be refrigerated and transported at 4 °C, as soon as possible;

The immediate measurement needs to be stored at -20 ° C for a shelf life of 7 days. The frozen urine sample was returned to room temperature before measurement.