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

GBZ/T 312-2018

**Determination of N-methyl acetamide in urine -- Gas
chromatography method**

尿中 N-甲基乙酰胺的测定 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 was drafted. Zhejiang Academy of Medical Sciences, Zhejiang Provincial Center for Disease Control and Prevention, Guangdong Provincial Hospital for Occupational Diseases, Shanghai Disease Prevention and Control Center.

1 Scope

This standard specifies gas chromatography for the determination of N-methylacetamide in urine.

This standard applies to the determination of N-methylacetamide in the 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 WS/T 97 urinary creatinine spectrophotometric method WS/T 98 urinary creatinine by

reversed-phase high performance liquid chromatography

3 Principle

After the urine sample (hereinafter referred to as the urine sample) is frozen, centrifuged, and precipitated with methanol, the N-A in the urine is separated by a polar capillary column.

Acetylamine, detected by a nitrogen-phosphorus detector, is characterized by retention time and peak area is quantified.

4 Instruments

4.1

Covered polyethylene plastic bottle. 50mL.

4.2

Covered polyethylene plastic centrifuge tube. 5mL, 10mL.

4.3

Centrifuge. 5000r/min.

4.4

Microinjector. 1 μ L.

4.5

Gas chromatograph with nitrogen and phosphorus detector, autosampler.

Instrument operation reference conditions.

- a) Column. 30 m x 0.53 mm x 1.0 μ m bonded/crosslinked polyethylene glycol (PEG-20M);
- b) Column temperature. initial temperature 100 ° C, hold for 1 min, raise temperature to 170 ° C at 25 ° C/min, hold for 3 min, heat up to 10 ° C/min to 210 ° C, kept for 3 min;
- c) vaporization chamber temperature. 250 ° C;
- d) detection chamber temperature. 320 ° C;
- e) carrier gas (nitrogen) flow. 6.0 mL/min;
- f) Split ratio. 10.1.

5 Reagent

5.1

Experimental water. chromatographic identification of interference-free peaks.

5.2

Methanol solution. 80% (volume fraction), chromatographic identification of interference-free peaks.

5.3

Standard solution. Accurately weigh a certain amount of N-methylacetamide (content $\geq 99\%$, dissolved in warm water and then take it), diluted with methanol solution Release and make up to a certain volume, make a standard stock solution, store in a refrigerator at $4\text{ }^{\circ}\text{C}$, and set aside. When used, it is diluted with methanol solution to a concentration of Standard application solution 1 at 1.00 mg/mL and standard application solution 2 at a concentration of 0.020 mg/mL .

6 Sample collection, transportation and storage

Cover the polyethylene plastic bottle to collect 50mL of the end of the working hours of occupational exposure to dimethylacetamide workers, and tighten the cap after the chamber it can be stored in a refrigerator at $-18\text{ }^{\circ}\text{C}$ for two weeks.

7 Analysis steps

7.1

Blank inspection. Take 1.0mL water and transfer to a 5mL centrifuge tube, add 4.0mL methanol solution, mix and measure for determination.

No interference peaks.

7.2

Sample processing. After the frozen urine sample is thawed at room temperature, mix it, and take 10 mL of urine into a 10 mL stoppered centrifuge tube to 3000 Centrifuge for 10 min at r/min , accurately absorb 1.0 mL of the supernatant and transfer to a 5 mL stoppered centrifuge tube, add 4.0 mL of methanol solution, mix, and then Centrifuge for 10 min at 3000 r/min and the supernatant was assayed.

7.3

Standard curve drawing. Accurately add 0.0 mL, 0.10 mL, 0.20 mL, 1.00 mL of standard application solution 2 in a 10 mL volumetric flask.

And 0.10mL, 0.50mL of the standard application solution 1, add water 2.0mL, dilute to the mark with methanol solution, formulated to a concentration of $0.0\mu\text{g/mL}$, N-methylacetamide standard series of $0.20\text{ }\mu\text{g/mL}$, $0.40\text{ }\mu\text{g/mL}$, $2.00\text{ }\mu\text{g/mL}$, $10.0\text{ }\mu\text{g/mL}$, $50.0\text{ }\mu\text{g/mL}$; reference instrument operation Conditions, the gas chromatograph was adjusted to the optimum measurement state, $0.2\text{ }\mu\text{L}$ was injected, and each standard series was measured, and each concentration was repeatedly measured three times.

Qualitative retention time, with the concentration of N-methylacetamide ($\mu\text{g/mL}$) as the abscissa, the mean value of the peak area measured is plotted on the ordinate.

Curve or calculate regression equations.

7.4

Sample determination. The urine sample supernatant solution is determined by the operating conditions of the assay standard series, and the measured peak area value is determined by a standard curve or the regression equation was used to obtain the concentration ($\mu\text{g/mL}$) of N-methylacetamide in the sample solution. If the concentration of N-methylacetamide in the sample exceeds the measurement range Dilute, can be determined by dilution with methanol solution, multiplied by the dilution factor.

8 Calculation

8.1

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

$$C=5C_0 \quad (1)$$

In the formula.

C-- concentration of N-methylacetamide in urine, in milligrams per liter (mg/L);

5-- dilution factor for pretreatment of urine sample;

C_0 --Measured concentration of N-methylacetamide in the urine sample solution in micrograms per milliliter ($\mu\text{g/mL}$).

8.2

In order to calibrate the dilution of urine, creatinine should be measured as soon as possible after urinary sample collection according to WS/T 97 or WS/T 98. See formula (2) for the correction formula.

In the formula.

m--Concentration of N-methylacetamide in urine samples after creatinine correction, in milligrams per gram of creatinine (mg/g creatinine);

C--the concentration of N-methylacetamide in urine, in milligrams per liter (mg/L);

Cr--urinary creatinine concentration in grams per liter (g/L).

9 Description

9.1

The determination range of this method is $0.20\mu\text{g/mL} \sim 50.0 \mu\text{g/mL}$, the detection limit is