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

GBZ/T 311-2018

**Determination of toluene diamine in urine -- Gas
chromatography method**

尿中甲苯二胺的测定 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. Tianjin Municipal Center for Disease Control and Prevention, Wuxi City Occupational Disease Prevention and Treatment Hospital of Jiangsu Province, Shenzhen Occupational Disease Prevention and Treatment Institute, Henan Institute of Occupational Diseases Prevention and Control, Xinxiang City Institute of Occupational Disease Prevention and Control.

1 Scope

This standard specifies gas chromatography for the determination of toluene diamine in urine.

This standard applies to the determination of toluene diamine 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.

GB/T 6682 Analytical laboratory water specifications and test methods WS/T 97 urinary creatinine spectrophotometric method WS/T 98 urinary creatinine by reversed-phase high performance liquid chromatography

3 Principle

In the urine sample (hereinafter referred to as the urine sample), the chelate of toluenediamine (TDA) is hydrolyzed by hydrolysis with sulfuric acid to form TDA, in the base.

Extracted with toluene under the conditions, derivatized with heptafluorobutyric anhydride, separated by polar capillary column, and detected by electron capture detector (ECD) Measured, characterized by retention time, and peak area quantified.

4 Instruments

4.1

Vortex mixer.

4.2

Centrifuge.

4.3

Constant temperature water bath.

4.4

Gas Chromatograph, Electron Capture Detector.

Instrument operation reference conditions.

- a) Column. Rtx-50, 30 m x 0.25 mm x 0.25 μ m;
- b) Column temperature. 160 $^{\circ}$ C;
- c) vaporization chamber temperature. 290 $^{\circ}$ C;
- d) detector temperature. 320 $^{\circ}$ C;
- e) carrier gas flow rate. 1.00 mL/min;
- f) Split ratio. 20.1.

5 Reagent

5.1

Experimental water. Performed in accordance with GB/T 6682.

5.2 2,4-TDA, chromatographically pure.

5.3

Toluene, chromatographically pure.

5.4

Heptafluorobutyric anhydride, 98%.

5.5

Sodium hydroxide solution, 450 g/L.

5.6

Hydrochloric acid solution, 1.0 mol/L.

5.7

Sulfuric acid solution, 3.0 mol/L.

5.8

Buffer solution. Weigh 27.2 g of potassium dihydrogen phosphate, dissolve it with 200 mL of water, and adjust the pH to 7.0 with sodium hydroxide solution.

5.9 2,4-TDA standard stock solution. accurately weigh 0.0500 g 2,4-TDA in a 50 mL volumetric flask, dissolve and volume with hydrochloric acid solution to the mark, prepare a standard stock solution with a concentration of 1.0 mg/mL.

5.10 2,4-TDA standard solution. dilute the 2,4-TDA standard stock solution with hydrochloric acid solution to prepare a standard solution with a concentration of 0.040 µg/mL.

liquid.

6 Sample collection, transportation and storage

6.1

Environmental conditions. The urine collection site should be clean and tidy, and there is no toluene diisocyanate (TDI) pollution source.

6.2

Sample collection. Collect urine samples from the end of the batch with clean polyethylene plastic bottles. The collection of urine samples should be greater than 50 mL. Collect duplicate samples if necessary. Product, one as a backup sample.

6.3

Sample blank. 2 samples of the urine collection bottle with the same batch number as the sample collection were randomly selected as the sample blank.

6.4

Sample transport. Store the collected samples and sample blanks in a clean container at 4 °C for refrigerated transport.

6.5

Sample storage. The sample can be stored for 7 days at -20 °C.

7 Analysis steps

7.1

Determine the urinary creatinine concentration as soon as possible according to the creatinine assay (see WS/T 97 or WS/T 98).

7.2

Sample processing. Pipette 2.0 mL of urine sample into a 10 mL centrifuge tube, add 1.5 mL of sulfuric acid solution, shake well, cover well, and place in boiling water.

Heated in the bath for 8 h, taken out and cooled to room temperature, add 5 mL of sodium hydroxide solution, add 2.0 mL of toluene, shake for 2 min, at 1500 r/min Centrifuge for 10 min. Take about 1.5 mL of toluene layer, place in another centrifuge tube, add 20.0 L heptafluorobutyric anhydride, shake for 2 min, place 5 min, add 1.0 mL buffer, shake for 2 min to remove excess heptafluorobutyric anhydride, place for 2 min, take the toluene layer for injection measurement.

7.3

Standard curve drawing. Take 5 plug colorimetric tubes and add 0.00 mL, 0.50 mL, 1.00 mL, 1.50 mL, 2.00 mL respectively.

Standard solution, each adding hydrochloric acid solution to 2.00 mL, formulated into 0.000 µg/mL, 0.010 µg/mL, 0.020 µg/mL, 0.030 µg/mL, 0.040 µg/mL standard series. In addition to heating, the standard series of processes are the same as the sample. Adjust the gas chromatograph according to the operating conditions of the instrument to the best measurement state, take 1.0 L toluene layer for injection, determine the standard series, repeat the determination of each concentration 3 times, with the peak area mean to TDA A standard curve was drawn at the concentration (g/mL).

7.4

Sample determination. Take the toluene layer of the treated sample at 1.0 L and calculate the TDA from the standard curve based on the measured peak area.

concentration.