

ICS 13.080
CCS Z 18

HJ

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

HJ 614-2011

**Soil. Determination of tetramethylene disulphotetramine. Gas
chromatography method
(English Translation)**

Issued on: April 15, 2010

Implemented on: October 1, 2011

Issued by: Ministry of Ecology and Environment

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Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China", protect the environment, protect human health, and standardize the determination method of soil poisoning and rat strength, Develop this standard.

This standard specifies gas chromatography for the determination of tetramine in soil.

This standard is the first release.

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard is mainly drafted by. Changchun Environmental Monitoring Center Station.

This standard is verified by. Shenyang Environmental Monitoring Center Station, Harbin Environmental Monitoring Center Station, Jilin Entry-Exit Inspection and Quarantine Bureau Technology Center, Jilin Province Environmental Monitoring Center Station, Dalian Environmental Monitoring Center and Jilin Province Product Quality Supervision and Inspection Institute.

This standard was approved by the Ministry of Environmental Protection on April 15, 2011.

This standard has been implemented since October 1, 2011.

This standard is explained by the Ministry of Environmental Protection.

Determination of soil tetramine by gas chromatography Warning. The tetramine is a highly toxic substance. The sample preparation process should be carried out in a fume hood. The operator should wear protective equipment to avoid Contact skin and clothing.

1 Scope of application

This standard specifies gas chromatography for the determination of tetramine in soil.

This standard applies to the determination of soil poisoning.

When the sample size is 5 g, the detection limit of this method is 3.5 µg/kg, and the lower limit of determination is 14 µg/kg.

2 Normative references

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard.

quasi.

HJ 613 Determination of dry matter and moisture in soils - Gravimetric method HJ/T 166 Technical Specifications for Soil Environmental Monitoring

3 Principle of the method

The tetramine in the soil was extracted with ethyl acetate, and the extract was purified and concentrated, separated by gas chromatography, and detected by a nitrogen-phosphorus detector.

Leave time to be qualitative and quantify by external standard method.

4 Reagents and materials

Analytical purification reagents conforming to national standards are used for analysis, unless otherwise stated. The experimental water is freshly prepared distilled water or removed.

Sub-water, chromatographic test without interference peaks.

4.1

Ethyl acetate. excellent grade.

4.2

anhydrous sodium sulfate.

Heat at 300 ° C for 2 h, placed in a desiccator and stored for cooling.

4.3

Tetramine standard stock solution. $\rho=200$ mg/L, directly purchase certified standard solution.

4.4

Standard test solution for tetramine. $\rho=20$ mg/L.

Accurately measure 1.0 ml of tetramine strong standard stock solution (4.3) into a 10 ml volumetric flask, dilute to volume with ethyl acetate (4.1), and mix.

4.5

Quartz sand. 30 ~ 60 mesh, heated at 300 ° C for 2 h before use.

4.6

Glass wool.

After washing with water, it was dried at 105 ° C, washed with ethyl acetate (4.1), dried, and placed in a desiccator for cooling and storage.

4.7

Activated carbon. 30-50 mesh, activated at 300 ° C for 2 h before use, placed in a desiccator for cooling and storage.

4.8

High purity nitrogen. purity $\geq 99.999\%$.

4.9

High purity hydrogen. purity $\geq 99.999\%$.

4.10

High purity air. purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Gas Chromatograph. with nitrogen and phosphorus detector.

5.2

Column 1. 30 m x 0.25 mm, film thickness 0.25 μm , 5% phenyl 95% methylpolysiloxane capillary column. Or other equivalent capillary Column.

5.3

Column 2. 30 m x 0.25 mm, film thickness 0.25 μm , 35% phenyl 65% methylpolysiloxane capillary column. Or other equivalent capillary Column.

5.4

Soxhlet extractor. 250 ml.

5.5

Nitrogen blowing instrument..200 ml with nitrogen blowing tube.

5.6

Glass purification column. hollow glass column with a length of about 10 cm and an inner diameter of 1.2 cm. Fill a small layer of glass wool (4.6) on the bottom of the hollow glass column.

Add 2 g of anhydrous sodium sulfate (4.2), 1.0 g of activated carbon (4.7) and 2 g of anhydrous sodium sulfate (4.2), using 10 ml of ethyl acetate (4.1) Pre-rinse and discard the eluent. A schematic diagram of the glass purification column is shown in Figure 1.

Anhydrous sodium sulfate Activated carbon Glass wool Anhydrous sodium sulfate

Figure 1 purification device diagram

5.7

Analytical balance. Accuracy is 0.01 g.

5.8

separatory funnel. 150 ml.

5.9

Vial. 2 ml, screw glass.