

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

**HJ 849-2017**

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**Water quality - Determination of ethylenethiourea - High  
performance liquid chromatography**

水质 乙撑硫脲的测定 液相色谱法

**(English Translation)**

**Issued on: August 28, 2017**

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**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 and the Law of the People's Republic of China on the Prevention and Control of Water Pollution, Barrier to human health, regulate the determination of water thiodide in water method, the development of this standard.

This standard specifies the liquid chromatography for the determination of ethylene thiourea in water.

This standard is the first release.

This standard is organized by the Environmental Monitoring Department of the Ministry of Environmental Protection and the Department of Science and Technology Standards.

The main drafting unit of this standard. Qingdao Environmental Monitoring Center Station and China Ocean University.

The main verification unit of this standard. Ministry of Agriculture, Agricultural Product Quality Supervision, Inspection and Testing Center (Qingdao), Shandong Province

Environmental Monitoring Central Station, Jinan City Environmental Monitoring Center Station, Tai'an City Environmental Protection Monitoring Station, Liaoning North Environmental Testing Technology Co., Ltd. and Green Island One Monitoring Co., Ltd.

The environmental protection department of this standard approved on August 28, 2017.

This standard has been implemented since November 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Determination of water quality of ethyl thiourea by liquid chromatography Warning. Ethylene thiourea is a carcinogen, the standard solution should be prepared in the operation of the fume hood, the operation should be required Wear protective equipment to avoid contact with skin and clothing.

## **1 Scope of application**

This standard specifies the liquid chromatography for the determination of ethylene thiourea in water.

This standard is applicable to the determination of ethylene thiourea in surface water, groundwater, domestic sewage and industrial waste water.

When the injection volume was 20  $\mu$ l, the detection limit was 3  $\mu$ g/L and the lower limit was 12  $\mu$ g/L.

## **2 Normative reference documents**

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

Technical specification for surface water and wastewater monitoring Technical specification for groundwater environmental monitoring

## **3 Principle of the method**

The aqueous thiourea in water was injected directly by liquid chromatography with an ultraviolet detector. According to the retention time Qualitative and external standard method.

## **4 Interference and elimination**

When the organic matter in the water sample interferes with the determination of ethylene thiourea, by changing the chromatographic conditions, so that the thiodide thiourea and dry Disassociation, or the water sample first with n-hexane extraction to remove part of the

interference, take the water phase for analysis.

## 5 Reagents and materials

The experimental water is pure water containing no organic matter.

### 5.1

methanol (CH<sub>3</sub>OH). liquid chromatography pure.

### 5.2

Ethylene thiourea. purity  $\geq$  98.5%.

### 5.3

Ethylene thiourea standard stock solution.  $\rho = 100 \mu\text{g/ml}$ .

Weigh 0.010 g (accurate to 0.1 mg) ethylene thiourea (5.2), dissolved in water to 100 ml, frozen at 4 ° C, Dark and sealed can be kept for 3 months, or direct purchase of certified standard solution.

### 5.4

Ethylene thiourea standard use of liquid.  $\rho = 10.0 \mu\text{g/ml}$ .

Accurately remove 1.00 ml of ethyl thiourea standard stock solution (5.3) and dilute to 10 ml with water.

## 6 Instruments and equipment

### 6.1

Sampling vials. 100 ml brown glass jar with a mouth plug.

### 6.2

Liquid Chromatograph. with UV detector or diode array detector.

26.3 Column. Reagent column with 5  $\mu\text{m}$  ODS, 25 cm in length and 4.6 mm in diameter, or other equivalent column.

### 6.4

Micro Samplers. 10  $\mu\text{l}$ , 50  $\mu\text{l}$  and 100  $\mu\text{l}$ .

### 6.5

Filtration. 0.22  $\mu\text{m}$  nylon filter or acetate filter.

### 6.6

Common laboratory equipment and equipment.

### 7.1 Sample collection and storage

Samples were collected according to the relevant regulations of HJ/T 91 and HJ/T 164. The collected water samples should be refrigerated at 4 ° C, protected from light and sealed. Save, within 30 d analysis is completed.

### 7.2 Sample preparation

The water sample was filtered through a 0.22 µm filter (6.5) and placed in a vial and tested.

**Note:** For samples containing organic matter, take 10 ml of water samples, with 2 ml of n-hexane extraction, take the water phase for determination.

### 7.3 Blank sample preparation

The sample was prepared in the same manner as in Sample Preparation Step (7.2).

## 8.1 Chromatographic reference conditions

Mobile phase A. methanol; mobile phase B. water; flow rate. 0.8 ml/min; column temperature. 30 °C; detection wavelength. 233 nm;

Injection volume. 20 µl. The elution procedure is shown in Table 1.

Table 1 elution program

Time (min) A (%) B (%) Note. After 6 minutes elution, the cleaning column can be used to clean the column. The cleaning time can be adjusted according to the complexity of the actual sample.

## 8.2 Calibration curve establishment

Respectively, take appropriate amount of thiodide standard use of liquid (5.4), diluted with water, the preparation of at least five concentrations of the standard series, the concentration of ethylene thiourea was 10.0, 50.0, 250, 500, 1000 µg/L (reference concentration). From low concentration to High concentration followed by the standard series of solution injection to the concentration of ethylene thiourea as the abscissa, the corresponding peak area or peak height Calculate the calibration curve.

## 8.3 Determination

The sample to be tested (7.2) was subjected to the same conditions as the calibration curve.