

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 850-2017

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

水质 硝磺草酮的测定 液相色谱法

(English Translation)

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Foreword

To implement the "Environmental Protection Law of the People's Republic of China" and "Water Pollution Prevention Law of the People's Republic of China" to protect the environment and protect the human body Health, regulate the determination of salt in the water method, the development of this standard.

This standard specifies the liquid chromatography for the determination of mesotrione 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 and inspection test center (Qingdao), Shandong Province Environmental Monitoring Center Station, Jinan City Environmental Monitoring Center Station, Tai'an City Environmental Protection Monitoring Station, Liaoning North Environmental Testing Technology Co., Ltd. and Qingdao in a monitoring limited the company.

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 mesotrione by liquid chromatography Warning.
Nitraquinone has a certain toxicity, the standard solution should be prepared in the

ventilation cabinet operation, the operation should be in accordance with the provisions
Wear protective equipment to avoid contact with skin and clothing.

1 Scope of application

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

This standard is applicable to the determination of mesohydrate in surface water, groundwater, domestic sewage and industrial wastewater.

When the injection volume is 20 μL , the detection limit is 0.01 mg/L and the lower limit is 0.04 mg/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 mannose in water was directly injected and analyzed 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 coexisting in water samples interferes with the determination of mesotrione, it is possible to change the chromatographic conditions, 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

phosphoric acid (H_3PO_4). $\rho(\text{H}_3\text{PO}_4) = 1.69 \text{ g/ml}$, excellent grade pure.

5.2

acetonitrile ($\text{C}_2\text{H}_3\text{N}$). liquid chromatography pure.

5.3

Methyrone. purity $\geq 99\%$.

5.4

standard dosage of mesotrione. $\rho = 100 \mu\text{g/ml}$.

Weigh 0.010 g (exact to 0.1 mg) of meso-ketone (5.3), dissolve with a small amount of acetonitrile (5.2), then water to 100 ml, frozen at 4 °C, dark and sealed can be stored for 3 months, or directly to buy certified standard solution.

5.5

Naphthaosterone standard use liquid I. $\rho = 10.0 \mu\text{g/ml}$.

Accurately remove 1.00 ml of meso-ketone standard stock solution (5.4) and dilute to 10 ml with water.

5.6

Naphthaosterone standard use of liquid II. $\rho = 1.00 \mu\text{g/ml}$.

Accurate removal of 1.00 ml of meso-ketone standard use of liquid (5.5), diluted with water to 10 ml.

26 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.

6.3 Columns. 5 μm ODS with a column length of 25 cm, an internal diameter of 4.6 mm, or other equivalent chromatography column.

6.4 Micro Samplers. 10 μl , 50 μl and 100 μl .

6.5 Membrane. 0.22 μm acetate filter or nylon filter.

6.6 Common laboratory equipment and equipment.

7 samples

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 7 d analysis is completed.

7.2 Sample preparation the pH of the water sample was adjusted to 3.0 to 4.0 with phosphoric acid (5.1). The water was filtered through a 0.22 μm filter (6.5) and placed in a sample Bottle, to be 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 Analysis steps

8.1 Chromatographic reference conditions Mobile phase A. acetonitrile; mobile phase B. water (take 1 L ultrapure water, add 100 µl of phosphoric acid (5.1) to pH 3.0);

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 11 minutes elution procedure for cleaning 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 meso-ketone standard liquid I (5.5) or standard use of liquid II (5.6), diluted with water, preparation 3 at least 5 concentration points of the standard series, the concentration of meso-ketone were 0.040,0.100,0.250,0.500,1.00, 4.00 mg/L (this is the reference concentration). From the low concentration to high concentration followed by the standard series of solution injection to the salt of mannose Concentration for the abscissa, the corresponding peak area or peak height for the vertical axis, the establishment of calibration curve.

8.3 Determination

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

8.4 blank test

The blank sample to be measured (7.3) was measured under the same conditions as the sample.

9 Results calculation and representation

9.1 Qualitative analysis According to the sample of meso-ketone and standard series of mesotrione retention time for qualitative, if necessary, the standard sample Product addition method, absorption ratio at different wavelengths or scanning ultraviolet absorption spectroscopy and other methods to assist qualitative.

The standard chromatogram of mesotrione is shown in Fig. 1 under the chromatographic conditions recommended in this standard (8.1).

Figure 1 Standard spectrum of mesotrione

9.2 Quantitative analysis the concentration of mesotrione in the water sample is calculated according to the following formula.

Where. - mass concentration of mesotrione in water samples, mg/L;