

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 770-2015

**Water quality. Determination of Phenoxy carboxylic acids
herbicide by High Performance Liquid Chromatography. Tandem
Mass Spectrometry**

水质 苯氧羧酸类除草剂的测定 液相色谱串联质谱法

(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, standard water phenoxy carboxylic acid herbicides monitoring methods, the development of this standard.

This standard specifies the determination of phenoxy carboxylic acid herbicides in water by liquid chromatography/tandem mass spectrometry.

This standard is the first release.

Appendix A is a normative appendix, Appendix B is an informative appendix.

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

The main drafting unit of this standard. Jiangsu Province Environmental Monitoring Center.

The standard method of verification unit. Suzhou City Environmental Monitoring Center Station, Dalian Environmental Monitoring Center Station, Jiangsu Entry-Exit Inspection and Quarantine Bureau Food Laboratory, Changzhou Environmental Monitoring Center, Jinan Environmental Monitoring Center, Shimadzu Enterprise Management (China) Co., Ltd. Shanghai Analysis center.

The Environmental Protection Department of this standard approves on November 20, 2015.

This standard is implemented as of December 15, 2015.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of phenoxy carboxylic acid herbicides Liquid chromatography/tandem mass spectrometry Warning. Phenoxy carboxylic acid herbicides are toxic organic compounds, the experimental process of the use of organic solvents with strong volatility. Minute Analysis of personnel in the experimental operation, should be in the fume hood operation; should be required to wear protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of phenoxy carboxylic acid herbicides in water by liquid chromatography/tandem mass spectrometry.

This standard applies to surface water, groundwater and waste water
2-methyl-4-chlorophenoxyacetic acid, 2,4-dichlorophenoxyacetic acid, 2- (2-methyl-Chlorophenoxy) propionic acid, 2- (2,4-dichlorophenoxy) -propionic acid (or 2,4-propionic acid), 2,4,5-trichlorophenoxyacetic acid, 2- (2,4,5-trichloro Phenoxy) -propionic acid (or 2,4,5-propionic acid), 4- (2,4-dichlorophenoxy) -butyric acid and 4- (2-methyl-4-chlorophenoxy) Butyric acid Determination of phenoxy carboxylic acid herbicides.

When the injection volume is 10 µl, the detection limit of the method is 0.3 µg/L ~ 0.5 µg/L, and the detection limit is 1.2 µg/L ~ 2.0 Mg/L, see Appendix A.

When the volume of the sample was 100 ml, the volume of the volume was 1.0 ml after concentration and the volume of the sample was 10 µl.

Limited to 0.006 µg/L to 0.009 µg/L, with a lower limit of 0.024 µg/L to 0.036 µg/L, as described in Appendix A.

2 Normative reference documents

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

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

3 Principle of the method

The phenoxy carboxylic acid herbicides in water were enriched by direct injection or solid phase extraction and separated by liquid chromatography/tandem mass spectrometry. according to Retention time and characteristic ion peak characterization, internal standard method quantification.

4 Reagents and materials

Unless otherwise stated, analytical reagents and experimental water are used in accordance with national standards.

4.1 Standard solution

4.1.1

Phenoxy carboxylic acid herbicide standard stock solution. $\rho = 100 \mu\text{g/ml}$.

Can be directly purchased certified standard solution can also be prepared with standard materials. The stock solution is stored in the dark at 0°C to 4°C or referenced to the manufacturer product manual. Use should be restored to room temperature, and shake.

4.1.2

phenoxy carboxylic acid herbicides standard use of liquid. $\rho = 10.0 \mu\text{g/ml}$ (reference concentration).

The standard stock solution (4.1.1) of the phenoxy carboxylic acid compound is diluted with acetonitrile (4.2) as required. Standard use of liquid at 4°C cold, avoid Light preservation, shelf life of two months. Use should be restored to room temperature, and shake.

4.1.3

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

The internal standard is 2,4-dichlorophenoxyacetic acid- $^{13}\text{C}_6$, can be directly purchased certified standard solution can also be prepared with the standard material. The stock solution is at zero $^\circ\text{C} \sim 4^\circ\text{C}$ Keep away from light or refer to the manufacturer's product description. Use should be restored to room temperature, and shake.

4.1.4

Internal standard liquid. $\rho = 10.0 \mu\text{g/ml}$ (reference concentration).

The internal standard stock solution (4.1.3) was diluted with acetonitrile (4.2) as needed. Internal standard use of liquid at $0^\circ\text{C} \sim 4^\circ\text{C}$ dark preservation, shelf life Two months. Use should be restored to room temperature, and shake.

4.2 acetonitrile (CH_3CN). pesticide residues.

4.3 methanol (CH_3OH). pesticide residues.

4.4 Methanol/water mixed solution. 1 4.

4.5 acetonitrile/water mixed solution. 1 4.

4.6 Ammonium acetate solution. $\text{C}(\text{CH}_3\text{COONH}_4) = 0.002 \text{ mol/L}$.

Weigh 0.077 g of ammonium acetate in 500 ml of experimental water and 0.22 μm filter

(4.13).

4.7 Sulfuric acid (H₂SO₄). ρ (H₂SO₄) = 1.84 g/ml.

4.8 Sodium hydroxide (NaOH).

4.9 Sulfuric acid solution. 1 1.

Measure 50 ml of concentrated sulfuric acid (4.7) and slowly add to 50 ml of water.

4.10 Sodium hydroxide solution. ρ (NaOH) = 0.4 g/ml.

Weigh 40 g of sodium hydroxide (4.8) in water and set to 100 ml.

4.11 solid phase extraction column. filler for the divinyl benzene and N-vinyl pyrrolidone copolymer (HLB) or the same column efficiency of the extraction column, specifications for the 6 ml/500 mg.

4.12 Nitrogen. Purity $\geq 99.99\%$.

4.13 Filtration. 0.22 μ m Teflon or other material equivalent filter.

5 Instruments and equipment

5.1

solid phase extraction device. automatic or manual, flow rate can be adjusted.

5.2

Concentration device. KD concentrator or nitrogen blowing concentrator and other equipment equivalent performance.

5.3

Liquid Chromatography/Tandem Mass Spectrometer. Equipped with electrospray ionization source (ESI).

5.4

Column. C18 or equivalent reversed-phase high performance liquid chromatography column with reference specifications of 100 mm x 2.1 mm, 1.7 μ m.

5.5

Microinjectors. 10 μ l, 50 μ l, 100 μ l, 250 μ l.

5.6

Common laboratory equipment and equipment.