

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

### HJ 1183-2021

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**Water quality Determination of omethoate, methamidophos,  
acephate and phoxim liquid chromatography-triple quadrupole  
mass spectrometry**

水质 氧化乐果、甲胺磷、乙酰甲胺磷、辛硫磷的测定 液相色谱-三重四极杆质谱  
法

**(English Translation)**

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### 1 Scope of application

This standard specifies the liquid chromatography-triple quadrupole mass spectrometry method for the determination of omethoate, methamidophos, acephate and phoxim in water.

This standard applies to omethoate, methamidophos, acephate and phoxim in surface water, groundwater, domestic sewage and industrial wastewater.

Determination.

When the injection volume was 5.0  $\mu\text{L}$ , the detection limit of the method was 0.5  $\mu\text{g/L}$ , and the lower limit of determination was 2.0  $\mu\text{g/L}$ . See Appendix A for details.

### 2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this

standard.

HJ/T 91 Technical Specification for Surface Water and Sewage Monitoring HJ 91.1  
Technical Specification for Wastewater Monitoring HJ 164 Technical Specification for  
Groundwater Environmental Monitoring

### 3 Principles of the method

The samples were directly injected after filtration or purification, and separated and detected by liquid chromatography-triple quadrupole mass spectrometry. According to retention time and characteristic ions Qualitative, internal standard method quantitative.

### 4 Interference and cancellation

#### 4.1

When there is matrix interference in the sample, it can be reduced or eliminated by optimizing the chromatographic conditions, diluting the sample, or reducing the injection volume.

#### 4.2

When the sample contains organic substances that interfere with the determination of the target substance, part of the interference can be removed by solid-phase extraction purification.

### 5 Reagents and Materials

Unless otherwise stated, analytical reagents that meet national standards were used in the analysis, and the experimental water was newly prepared without target compounds.

pure water.

#### 5.1

Acetonitrile ( $\text{CH}_3\text{CN}$ ). chromatographically pure.

#### 5.2

Methanol ( $\text{CH}_3\text{OH}$ ). chromatographically pure.

#### 5.3

Ethyl acetate ( $\text{CH}_3\text{COOCH}_2\text{CH}_3$ ). chromatographically pure.

#### 5.4

Hydrochloric acid.  $\rho = 1.19 \text{ g/ml}$ , excellent grade.

**5.5**

Sodium Hydroxide (NaOH).

**5.6**

Ammonium formate (HCOONH<sub>4</sub>).

**5.7**

Standard products of omethoate, methamidophos, acephate, and phoxim. the purity is not less than 99.0%.

**5.8**

Standard products of methamidophos-d<sub>6</sub>, omethoate-d<sub>6</sub> and phoxim-d<sub>5</sub>.the purity is not less than 99.0%.

**5.9**

Acetonitrile solution.  $\phi(\text{CH}_3\text{CN}) = 50\%$ .

Mix acetonitrile (5.1) and water in a 1.1 volume ratio.

**5.10**

Acetonitrile-ethyl acetate mixed solution.  $\phi(\text{CH}_3\text{CN}) = 50\%$ .

Mix acetonitrile (5.1) and ethyl acetate (5.3) in a 1.1 volume ratio.

**5.11**

Methanol solution.  $\phi(\text{CH}_3\text{OH}) = 80\%$ .

Mix methanol (5.2) and water in a volume ratio of 8.2.

**5.12**

Hydrochloric acid solution.  $\phi = 50\%$ .

Hydrochloric acid (5.4) and water were mixed in a volume ratio of 1.1.

**5.13**

Sodium hydroxide solution.  $c(\text{NaOH}) = 0.1 \text{ mol/L}$ .

Weigh 0.4 g of sodium hydroxide (5.5) and dissolve it in a small amount of water, dilute to 100 ml, and mix well.

**5.14**

Ammonium formate solution.  $c(\text{HCOONH}_4) = 5.0 \text{ mmol/L}$ .

Weigh 0.16 g of ammonium formate (5.6) and dissolve it in a small amount of water, dilute to 500 ml, and mix well.

#### 5.15

Ammonium formate-acetonitrile solution.  $c = 5.0 \text{ mmol/L}$ .

Weigh 0.16 g of ammonium formate (5.6) and dissolve it in a small amount of acetonitrile (5.1), dilute to 500 ml, and mix.

#### 5.16

Mixed standard stock solution of omethoate, methamidophos, acephate, and phoxim.  $\rho = 1\,000 \text{ mg/L}$ .

Weigh 100.0 mg of omethoate, methamidophos, acephate, and phoxim standard (5.7) respectively, dissolve them in a small amount of acetonitrile (5.1).

solution, transfer to a 100 ml volumetric flask, make up to the mark with acetonitrile (5.1), and mix well. Aliquot into brown vials, seal,  $-15^{\circ}\text{C}$  the following storage in the freezer and protected from light has a shelf life of 6 months. The certified standard solution can also be directly purchased and stored according to the standard solution certificate requirements.

#### 5.17

Mixed standard solution of omethoate, methamidophos, acephate and phoxim.  $\rho = 10.0 \text{ mg/L}$ .

Pipette appropriate amount of omethoate, methamidophos, acephate, and phoxim mixed standard stock solution (5.16), dilute with acetonitrile solution (5.9) Release, refrigerate below  $4^{\circ}\text{C}$  and protect from light, the shelf life is 2 months.

#### 5.18

Internal standard stock solution.  $\rho = 100 \text{ mg/L}$ .

Weigh 10.0 mg of methamidophos-d6, omethoate-d6, and phoxim-d5 standard (5.8), dissolve with a small amount of acetonitrile (5.1), transfer to Transfer to a 100 ml volumetric flask, make up to the mark with acetonitrile (5.1), and mix well. Dispense into brown sample vials, seal, cool below  $-15^{\circ}\text{C}$  Refrigerated and protected from light, the shelf life is 6 months. The certified standard solution can also be directly purchased and stored according to the standard solution certificate requirements.

#### 5.19

Internal standard solution.  $\rho = 0.50 \text{ mg/L}$ .

Pipette an appropriate amount of the internal standard stock solution (5.18), dilute it with acetonitrile solution (5.9), and store it under  $4^{\circ}\text{C}$  in the dark, with a shelf life of 2 months.

#### 5.20

Solid phase extraction column I. The packing is octadecyl-bonded silica gel, or an extraction column with equivalent column efficiency, with a specification of 500 mg/6 ml.