

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Water quality. Determination of chlorothalonil and pyrethroid
insecticides. Gas chromatography mass spectrometry**

水质 百菌清及拟除虫菊酯类农药的测定 气相色谱—质谱法

(English Translation)

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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, Protection of human health, regulate the determination of chlorothalonil and pyrethroid pesticides in the determination of the method, the development of this standard.

This standard specifies the chlorothalonil and pyrethroid pesticide gas chromatography-mass spectrometry.

This standard is the first release.

Appendix A to this standard is a normative appendix, Appendix B and Appendix C are informative appendices.

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

The main drafting unit of this standard. National Environmental Analysis and Testing Center.

The standard verification unit. Hunan Province Environmental Monitoring Center Station, Hubei Province Environmental Monitoring Center Station, Pearl River Basin Water Environment Monitoring Measuring center, Xiangtan City Environmental Protection Monitoring Station, Changsha City Environmental Monitoring Center Station and Zhuzhou City Environmental Monitoring Center Station.

The Environmental Protection Department of this standard approves on August 21, 2015.

This standard is implemented on October 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

Determination of chlorothalonil and pyrethroid pesticides - Gas chromatography - mass spectrometry Warning. The solvent, internal standard and standard samples used in the experiment are toxic and harmful compounds. The solution should be prepared.

Wind cabinet, the operation should be required to wear protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the liquid-liquid extraction or solid-phase extraction/gas chromatography-mass spectrometry of chlorothalonil and eight pyrethroid pesticides Spectrum method.

This standard applies to surface water, groundwater, industrial wastewater and domestic sewage chlorothalonil and pyrethroid pesticide compounds Determination of matter.

The detection limit of the method was 0.005 µg/L to 0.05 µg/L, and the lower limit of determination was 0.020 Mg/L ~ 0.20 µg/L. When the sample volume was 500 ml, the detection limit was 0.005 µg/L ~ 0.08 µg/L.

Limited to 0.020 µg/L to 0.32 µg/L. See Appendix A. for details.

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 liquid extract or solid phase extraction method was used to extract chlorothalonil and pyrethroid pesticide in water samples. The extract was dehydrated, Concentrated, purified, constant volume, separated by gas chromatography, mass spectrometry. According to the retention time, the fragment ion mass ratio and its abundance Degree of qualitative and internal standard method.

4 Reagents and materials

Unless otherwise stated, analytical reagents and pure water that do not contain the target are used in the analysis.

4.1

dichloromethane (CH_2Cl_2). pesticide residues.

4.2

n-hexane (C_6H_{14}). pesticide residues.

4.3

methanol (CH_3OH). pesticide residues.

4.4

chlorothalonil standard solution. $\rho = 1\ 000\ \text{mg/L}$, the solvent is acetone, commercially available standard solution.

4.5

pyrethroid standard solution. $\rho = 1\ 000\ \text{mg/L}$, including deltamethrin, fenvalerate, cypermethrin, cyhalothrin, Chrysanthemum, fenpropathrin, ketamine and allyrin, the solvent is acetone, commercially available standard solution.

4.6

internal standard stock solution I. $\rho = 100\ \text{mg/L}$, including deuterated phenanthrene, deuterated pyrene and deuterium, the solvent is n-hexane. Can be purchased directly Certified standard solution can also be prepared with the standard material, diluted with n-hexane, $4\ ^\circ\text{C}$ below the sealed light preservation.

4.7

internal standard stock solution II. $\rho = 100\ \text{mg/L}$, including ^{13}C -PCB209, the solvent is n-hexane. Can be purchased directly to prove the standard solution, can also be prepared using standard materials, diluted with n-hexane, $4\ ^\circ\text{C}$ below the sealed light preservation.

24.8 anhydrous sodium sulfate (Na_2SO_4).

Burn at $400\ ^\circ\text{C}$ for 4 h, cooled into the grinding glass bottle, placed in the dryer to save.

4.9

standard solution liquid. $\rho = 10.0 \sim 100\ \text{mg/L}$.

Respectively, remove $100\ \mu\text{l}$ of chlorothalonil (4.4) and bifenthrin, fenpropathrin, ketamine, allylin standard solution (4.5), and then remove $1.00\ \text{ml}$ of deltamethrin, fenvalerate, cypermethrin and cyhalothrin standard solution (4.5) to $10\ \text{ml}$ Color volume bottle, with n-hexane (4.2) constant volume to the mark, mix. Is now available.

4.10

internal standard use of liquid. $\rho = 10.0 \sim 20.0\ \text{mg/L}$.

Remove the $1.00\ \text{ml}$ internal standard stock solution I (4.6) and the $2.00\ \text{ml}$ internal

standard stock solution II (4.7) to 10 ml volumetric flask, respectively, with n-hexane (4.2) constant volume to the mark, mix.

4.11

n-hexane acetone solution. 95 5.

4.12

Magnesium silicate column. The filler is magnesium silicate, 500 mg, and the column volume is 6 ml.

4.13

solid phase extraction column. the filler is lipophilic divinyl benzene and hydrophilic N-vinyl pyrrolidone two kinds of monomer by a certain proportion of poly Synthetic macroporous copolymer or equivalent type of filler, 1 000 mg, column volume of 6 ml, commercially available.

4.14

Carrier gas. helium, purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Sampling vials. 1 L or 2 L brown glass jar with a mouth plug.

5.2

Gas Chromatography Mass spectrometer. with capillary column and split/splitless inlet, programmable temperature, mass spectrometry with EI source.

5.3

Column. 5% phenyl-methylpolysiloxane with a column length of 30 m. The column was a fused silica with an inner diameter of 0.25 mm Capillary column with a film thickness of 0.25 μm . Or use other equivalent columns.

5.4

solid phase extraction device. solid phase extraction instrument, through the vacuum pump to adjust the flow rate.

5.5

Concentration device. rotary evaporator or KD concentrator, nitrogen blowing concentrator and other enrichment device.

5.6

separatory funnel. 2 L with polytetrafluoroethylene plug.