

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

HJ 963-2018

**Solid waste - Determination of 47 organophosphorous and
pyrethroid pesticides - Gas chromatography mass
spectrometry**

固体废物 有机磷类和拟除虫菊酯类等 47 种农药的测定 气相色谱-质谱法
(English Translation)

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Foreword

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 Environmental Pollution by Solid Waste Protect the environment, protect human health, and regulate 47 kinds of pesticides such as organophosphorus and pyrethroids in solid waste and its leachate the method of determination is to develop this standard.

This standard specifies the gas phase color of 47 pesticides such as organophosphorus and pyrethroids in solid waste and its leachate.

Spectral-mass spectrometry.

Appendix A of this standard is a normative appendix, and Appendix B~Appendix C is an informative appendix.

This standard is the first release.

This standard is formulated by the Environmental Monitoring Department and the Science and Technology Standards Department.

This standard was drafted. Zhejiang Environmental Monitoring Center.

This standard is verified by. Jiangsu Environmental Monitoring Center, Hubei Environmental Monitoring Center Station, Hangzhou Environmental Monitoring Center Station, Ningbo Environmental Monitoring Center, Shaoxing Environmental Monitoring Center Station and Jiaxing Environmental Protection Monitoring Station.

This standard is approved by the Ministry of Ecology and Environment on July 29, 2018.

This standard has been implemented since January 1, 2019.

This standard is explained by the Ministry of Ecology and Environment.

1 Determination of 47 pesticides such as organophosphorus and pyrethroids in solid waste

Gas chromatography-mass spectrometry Warning. The organic solvents and reference materials used in the experiment are toxic and hazardous substances, standard solution preparation and sample preparation.

The process should be carried out in a fume hood; protective equipment should be worn as required to avoid direct contact with skin and clothing.

1 Scope of application this standard specifies the gas phase of 47 pesticides such as organophosphorus and pyrethroids in solid waste and its leachate.

Chromatography-mass spectrometry.

This standard applies to the determination of 47 pesticides such as organophosphorus and pyrethroids in solid waste and its leachate. Its organophosphorus and pyrethroid pesticides have also been verified by this method.

Under the condition of selective ion (SIM), when the solid waste sample volume is 10.0 g and the constant volume is 1.0 ml, the target the detection limit of the method is 0.2-0.9 mg/kg, and the lower limit of determination is 0.8-3.6 mg/kg. Solid waste leachate sample when the product volume is 500 ml and the volume is 1.0 ml, the detection limit of the target is 0.006-0.02 mg/L. The lower limit of determination is 0.024 ~ 0.08 mg/L. See Appendix A for details.

2 Normative references

This standard refers to the following documents or their terms. For undated references, the valid version applies to this standard.

HJ 782 solid waste organic matter extraction pressurized fluid extraction HJ/T 20 Technical Specifications for Sampling and Sample Preparation of Industrial Solid Waste HJ/T 298 Hazardous Waste Identification Technical Specification HJ/T 299 solid waste leaching toxicity leaching method sulfuric acid nitric acid method HJ/T 300 solid waste leaching toxicity leaching method acetic acid buffer solution method

3 Principle of the method

After extraction, purification, concentration and constant volume of organophosphorus and pyrethroid pesticides in solid waste and its leachate, it was separated by gas chromatography and detected by mass spectrometry. Qualitative according to mass

spectrum, retention time, fragment ion mass-to-charge ratio and abundance ratio Standard method of quantification.

4 Reagents and materials

Analytically pure reagents in accordance with national standards were used for analysis, unless otherwise stated.

The pure water of the standard.

4.1

Acetone (C₃H₆O). pesticide residue level.

24.2 n-Hexane (C₆H₁₄). pesticide residue grade.

4.3

Dichloromethane (CH₂Cl₂). pesticide residue grade.

4.4

Acetonitrile (C₂H₃N). pesticide residue grade.

4.5

Hexane-acetone mixture. 1 1.

4.6

Acetonitrile-toluene mixture. 3 1.

4.7

Standard stock solution. pyrethroids, organophosphorus and heterocyclic pesticides, rho=500 mg/L.

Commercially available certified standard solutions can be purchased directly or formulated with standard materials. Store as required by the standard solution certificate.

4.8

Internal standard stock solution. rho=1000 mg/L.

Select tetrachloromethylene xylene (TCMX) as the internal standard solution, you can directly purchase the commercially available certified standard solution, or use the standard Quasi-substance preparation.??-d10, phenanthrene-d10, can also be used on the premise that the method requirements are met and the target compound is not interfered with.

Other internal standards such as ?-d12 and ?-d12.

4.9

Mass spectrometry tuning solution. decafluorotriphenylphosphine (DFTPP), rho = 5 mg/L.

Commercially available certified standard solutions can be purchased directly or prepared with standard materials and diluted with dichloromethane (4.3).

4.10

anhydrous sodium sulfate (Na_2SO_4). excellent grade pure.

Bake in a muffle furnace at 450 ° C for 4 h, cool, and seal in a ground glass bottle.

4.11

Diatomaceous earth. 0.6 to 0.9 mm (30 to 20 mesh).

Bake in a muffle furnace at 450 ° C for 4 h, cool, and seal in a ground glass bottle.

4.12

Quartz sand. 150 ~ 830 μm (200 ~ 100 mesh).

Bake in a muffle furnace at 450 ° C for 4 h, cool, and seal in a ground glass bottle.

4.13

Graphitized carbon black packing cartridge. 500 mg/6 ml.

4.14

Aminopropyl bonded silica packing cartridge. 500 mg/3 ml.

4.15

High purity nitrogen. purity $\geq 99.999\%$.

4.16

High purity helium. purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Gas Chromatography-Mass Spectrometry. The gas chromatograph section has a split/splitless inlet for temperature programming. Mass spectrometry There is an electron bombardment ionization (EI) source.

5.2

Column. quartz capillary column, 30 m x 0.32 mm x 0.25 μm , stationary phase is 14% cyanopropyl phenyl-86% Polysiloxane, or other equivalent column.

5.3

Extraction device. Soxhlet extractor, pressurized fluid extraction device or other equivalent extraction device.