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HJ

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

HJ 835-2017

**Soil and sediment. Determination of organochlorine pesticides.
Gas chromatography/Mass spectrometry**

土壤和沉积物 有机氯农药的测定 气相色谱-质谱法

(English Translation)

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Foreword

For the implementation of the "People's Republic of China Environmental Protection Law" to protect the environment, protect human health, regulate soil and sediment in the determination of organochlorine pesticides, the development of this standard.

This standard specifies the determination of 23 organochlorine pesticides in soil and sediments by 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 Environmental Monitoring Department of the Ministry of Environmental Protection, the Secretary for Science and Technology Standards.

The main drafting of this standard. Henan Province Environmental Monitoring Center.

The standard verification unit. Xinxiang City Environmental Monitoring Station, Henan Province Environmental Science Research Institute, Zhengzhou City, environmental protection monitoring Heart station, Kaifeng City Environmental Monitoring Station, Institute of Hydrogeology and Geology, Chinese Academy of Geological Sciences, Henan Province Environmental Monitoring Center.

The Environmental Protection Department of this standard approved on July 18, 2017.

This standard has been implemented since September 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Determination of organochlorine pesticides in soil and sediments - Gas chromatography - mass spectrometry Warning. The organic solvents and reference materials used in the test

are toxic and hazardous substances, standard solution preparation and sample pretreatment should be carried out in the fume hood; operation should be required to wear protective equipment, to avoid direct contact with skin and clothing.

1 Scope of application

This standard specifies the determination of organochlorine pesticides in soil and sediments by gas chromatography-mass spectrometry.

This standard is applicable to the determination of 23 organochlorine pesticides in soil and sediments. Targets include. alpha- 6606, hexachlor Benzene, beta-hexachloride, gamma-six six six, delta-six six six, heptachlor, aldrin, epoxidized heptachlor, alpha-chlordane, alpha-endosulfan, gamma-Dibenzo, p, p'-DDE, endrin, beta-endosulfan, p, p'-DDD, endosulfan sulfate, aldrin aldehyde, o, p'-DDT, Endrin ketone, p, p'-DDT, methotrexate, mirex. This organism is also applicable to other organochlorine pesticides.

When the sampling volume is 20.0 g and the volume of the volume after concentration is 1.0 ml, the detection limit is 0.02 mg/kg to 0.09 mg/kg, with a lower limit of 0.08 mg/kg to 0.36 mg/kg. See Appendix A.

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.

Specification for ocean monitoring - Part 3. Sample collection, storage and transport
Specification for marine monitoring - Part 5. Sediment analysis Determination of Dry Matter and Moisture in Soil Extraction of pressurized fluid from soil and sediment organic matter
Technical specification for soil environmental monitoring

3 Principle of the method

The organochlorine pesticides in soil or sediments are extracted using a suitable extraction method (Soxhlet extraction, pressurized fluid extraction, etc.) According to the sample matrix interference to choose the appropriate purification method (copper powder desulfurization, magnesium silicate column or gel permeation chromatography), to mention Take liquid purification, and then concentrated, constant volume, by gas chromatography separation, mass spectrometry. According to the standard substance mass spectrometry, retention time, Debris ion mass ratio and its abundance characterization. Internal standard method.

4 Reagents and materials

Unless otherwise stated, analytical reagents conforming to national standards are used for analysis. Experimental water for the new preparation of ultra-pure water or distilled water.

4.1

acetone (C₃H₆O). pesticide residues.

4.2

n-hexane (C₆H₁₄). pesticide residues.

4.3

dichloromethane (CH₂Cl₂). pesticide residues.

4.4

ethyl acetate (C₄H₈O₂). pesticide residues.

4.5

Cyclohexane (C₆H₁₂). Pesticide level.

24.7 N-Hexane - Acetone Mixed Solvent I. 1 1

Mix with 1. 1 by volume using n-hexane (4.2) and acetone (4.1).

4.8 N-Hexane-Acetone Mixed Solvent II. 9 1 Mixed with n-hexane (4.2) and acetone (4.1) at a volume ratio of 9. 1

4.9 Dichloromethane - acetone mixed solvent. 1 1 Mix with methylene chloride (4.3) and acetone (4.1) at a 1. 1 volume ratio.

4.10 N-Hexane-Ethyl Ether Mixed Solvent I. 94 6 Mix with 94. 6 volume with n-hexane (4.2) and diethyl ether (4.6).

4.11 n-Hexane-Ether Mixed Solvent II. 85 15 Using n-hexane (4.2) and diethyl ether (4.6) at a volume of 85.15.

4.12 N-Hexane-Ether Mixed Solvent III. 1 1 Mix with 1. 1 volume with n-hexane (4.2) and diethyl ether (4.6).

4.13 n-hexane-dichloromethane mixed solvent I. 1 1 Mix with 1. 1 volume with n-hexane (4.2) and dichloromethane (4.3).

4.14 n-hexane-dichloromethane mixed solvent II. 74 26 Mixed with hexanes (4.2) and dichloromethane (4.3) at a volume of 74.26.

4.15 Gel Permeation Chromatography Mobile phase. mixed with ethyl acetate (4.4) and cyclohexane (4.5) at 1 1 volume ratio, or by instrument the preparation of other solvent

system.

4.16 nitric acid. ρ (HNO_3) = 1.42 g/ml, excellent grade pure.

4.17 Nitric acid solution. 1 1.

With excellent grade nitric acid (4.16) and experimental water by 1. 1 volume ratio mixed configuration.

4.18 organic chlorine pesticide standard stock solution. ρ = 1000 mg/L ~ 5000 mg/L, commercially available standard solution.

4.19 Organochlorine pesticide standard intermediate liquid. ρ = .200 mg/L ~ 500 mg/L.

The organochlorine pesticide standard stock solution (4.18) was diluted with n-hexane-acetone mixed solvent I (4.7) and mixed.

4.20 Internal standard stock solution. ρ = 5000 mg/L Selection of pentachloronitrobenzene or phenanthrene-d10 or ?-d12 as internal standard. A commercially available standard solution. Other compounds can also be used to do Internal standard.

4.21 Internal standard broth. ρ = 500 mg/L Measure the amount of 1.0ml internal standard stock (4.20) in 10 ml volumetric flask, with n-hexane - acetone mixed solvent I (4.7) dilute Release to the marking, mixing.

4.22 Substitute stock solution. ρ = .2000 mg/L to 4000 mg/L, commercially available standard solution.

Decachlorobenzene or 2,4,5,6-tetrachloro-xylene and dibutyl fumarate should be used.

4.23 Substitutes Intermediate. ρ = .200 mg/L to 400 mg/L the mixture was diluted with n-hexane-acetone mixed solvent I (4.7) on the standard stock solution (4.22) and mixed.

4.24 decafluorotriphenylphosphine (DFTPP) solution. ρ = 50 mg/L, commercially available standard solution.

The other concentrations were diluted with dichloromethane (4.3) to a concentration of 50 mg/L.

4.25 Gel Permeation Chromatography Calibration solution. containing corn oil (25 mg/ml), di (2-diethylhexyl) phthalate (1 mg/ml), methotrexate (200 mg/L), perylene (20 mg/L) and sulfur (80 mg/L). Commercially available.

3 Note. All standard stock solutions from 4.18 to 4.25 should be stored in the manufacturer's product description.

-10 °C below the dark preservation. Before use should check the changes, once the evaporation or degradation should be re-preparation, should be used before the room temperature, mixing.