

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

HJ 912-2017

**Solid waste--Determination of organochlorine pesticides--Gas
chromatography mass spectrometry**

固体废物 有机氯农药的测定 气相色谱-质谱法

(English Translation)

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Foreword

In order to carry out "Law of the People's Republic of China on Environmental Protection" and "Law of the People's Republic of China on Prevention and Control of Environmental Pollution by Solid Wastes" Protect the environment, protect human health, standardize the determination of organochlorine pesticides in solid waste and leachate, and develop this standard.

This standard specifies the determination of solid waste and its leachate 23 kinds of organochlorine pesticides by gas chromatography - mass spectrometry.

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

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. Henan Environmental Monitoring Center.

This standard verification unit. Xinxiang City Environmental Monitoring Station, Henan Province Environmental Science Research Institute, Zhengzhou City Environmental Protection Supervisor Center Station, Kaifeng City Environmental Monitoring Station, Institute of Hydrogeology and Environmental Geology, Chinese Academy of Geological Sciences and Henan Province Environmental Monitoring Center.

This standard MEP approved on December 29, 2017.

This standard since April 1, 2018 come into operation.

This standard is interpreted by the MEP.

Solid waste - Determination of organochlorine pesticides - Gas chromatography - mass spectrometry Warning. The organic solvents and reference materials used in the experiment are toxic substances, standard solution preparation and sample preparation should be carried out in a fume hood; operation should be worn protective clothing as required to avoid direct contact with the skin and clothing.

1 Scope of application

This standard specifies the determination of solid waste and its leachate organochlorine pesticides by gas chromatography - mass spectrometry.

This standard applies to solid waste and leaching solution of alpha-BHC, HCB, beta-BHC, gamma-BHC, delta-BHC Hexachlorocyclohexane, Aldrin, Ephedrine B, alpha-Chlordane, Endosulfan I, gamma-Chlordane, Dieldrin, p, p'-Endosulfan II, p, p'-Drip, o, p'-DDT, Endrin aldehyde, Endosulfan sulfate, p, p'-DDT, Endrin, Methoxy DDT, mirex and other 23 kinds of organochlorine pesticides determination. If other organochlorine pesticides through the verification, can also use this Standard determination.

When the amount of ash solid waste sample was 10 g and the volume of constant volume was 1.0 ml, the detection limits of 23 organochlorine pesticides 0.02 ~ 0.09 mg/kg, the lower limit of determination was 0.08 ~ 0.36 mg/kg. When the sludge solid waste sampling volume of 2 g, the volume of the body the product detection limit of 23 organochlorine pesticides was 0.2-0.8 mg/kg when the product was 1.0 ml. The lower limit of determination was 0.8-3.2 mg/kg.

When the sample volume of the leachate is 100 ml and the volume of constant volume is 10 ml, the detection limits of the 23 organochlorine pesticides are 0.04 ~ 0.1 mg/L, the lower limit of determination is 0.16 ~ 0.4 mg/L.

Methods for detection of solid waste and leachate detection limits and lower limit see Appendix A.

2 Normative references

This standard references the following documents or the terms. For undated references, the effective version applies to this book standard.

HJ 765 solid waste organic matter extraction microwave extraction method HJ 782 Solid waste - Extraction of organic matter - Pressurized fluid extraction HJ/T 20 Industrial solid waste sampling Sampling specifications HJ/T 298 Hazardous Waste Identification Technical Specifications Industrial solid waste - Extraction toxicity Toxicity - Sulfuric acid nitrate method Industrial solid waste - Extraction toxicity Toxicity - Acetic acid buffer solution method

3 Method principle

Organochlorine pesticides in solid wastes and leachate were extracted, purified, concentrated, and fixed, then separated by gas chromatography Spectrum detection. According to mass spectrometry, retention time, fragment ion mass ratio and its abundance qualitative, internal standard method.

4 Reagents and materials

Unless otherwise stated, analytical grade reagents that meet national standards are used. Experimental water for the newly prepared pure water or Distilled water.

4.1

acetone (C₃H₆O). pesticide grade.

4.2

n-hexane (C₆H₁₄). pesticide grade.

4.3

Dichloromethane (CH₂Cl₂). pesticide residues.

Ethyl acetate (C₄H₈O₂). pesticide residues.

4.5

cyclohexane (C₆H₁₂). pesticide grade.

4.6

ether (C₄H₁₀O). pesticide residues.

4.7

n-hexane - acetone mixed solvent I. 1 1.

Mix with n-hexane (4.2) and acetone (4.1) at 1.1 volume ratio.

4.8

n-hexane - acetone mixed solvent II. 9 1.

Mix with 9.1 volumes by volume with n-hexane (4.2) and acetone (4.1).

4.9

Dichloromethane - acetone mixed solvent. 1 1.

Mix with methylene chloride (4.3) and acetone (4.1) at 1.1 volume ratio.

4.10

n-hexane - ether mixed solvent I. 94 6.

Mix with n-hexane (4.2) and diethyl ether (4.6) at a 94.6 volume ratio.

4.11

n-hexane - ether mixed solvent II. 85 15.

Mix with 85.15 by volume with n-hexane (4.2) and ether (4.6).

4.12

n-hexane - ether mixed solvent III. 1 1.

Mix with n-hexane (4.2) and diethyl ether (4.6) at a 1.1 volume ratio.

4.13

n-hexane - dichloromethane mixed solvent I. 1 1.

Mix with n-hexane (4.2) and methylene chloride (4.3) at 1.1 volume ratio.

4.14

n-hexane - dichloromethane mixed solvent II. 74 26.

Mix with n-hexane (4.2) and methylene chloride (4.3) in a volume ratio of 74.26.

4.15

gel permeation chromatography mobile phase.

Mix with ethyl acetate (4.4) and cyclohexane (4.5) at a volume ratio of 1.1, or prepare other solvents according to the instruction manual system.

4.16

nitric acid. $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$, excellent grade pure.

4.17

nitric acid solution. 1 1.

Mix with nitric acid (4.16) and water at a volume ratio of 1.1.