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HJ

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

HJ 921-2017

**Soil and sediment--Determination of organochlorine
pesticides--Gas chromatography**

土壤和沉积物 有机氯农药的测定 气相色谱法

(English Translation)

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Foreword

In order to carry out "Environmental Protection Law of the People's Republic of China", protect the environment, protect human health, standardize the soil and sediments Determination of organochlorine pesticides, the development of this standard.

This standard specifies the determination of soil and sediment 23 kinds of organochlorine pesticides by gas chromatography.

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. Jiangsu Provincial Environmental Monitoring Center.

This standard verification unit. Taizhou City Environmental Monitoring Center Station, Henan Province Environmental Monitoring Center, Nanjing Environmental Monitoring Center Station, Dalian City Environmental Monitoring Center, Nanjing Environmental

Protection Research Institute of Environmental Protection and Exit Inspection and Quarantine of Nanjing Electrical and Electronic Product Laboratory.

This standard MEP approved on December 28, this standard since April 1, 2018 come into operation.

This standard is interpreted by the MEP.

Soil and sediments - Determination of organochlorine pesticides - Gas chromatography
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 organochlorine pesticides in soil and sediment by gas chromatography.

This standard applies to soil and sediment alpha - BHC, HCB, gamma - BHC, beta - BHC, delta - BHC, sulfur Dan I, aldrin, endosulfan II, epichlorohydrin,, o, p'-didehydrin, gamma-chlordane, alpha-chlordane, trans-p, p'-Drip, o, p'-Drip, Dieldrin, Endrin, o, p'-DDT, p, p'-p, p - DDT, mirex and other 23 kinds of organochlorine pesticides determination. If other organochlorine pesticides through the verification, can also be measured using this standard.

When the sample volume was 10.0 g, the detection limit of 23 organochlorine pesticides was 0.04 µg/kg ~ 0.09 µg/kg.

The limit is 0.16 µg/kg ~ 0.36 µg/kg. See Appendix A for details.

2 Normative references

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

GB 17378.3 Marine monitoring code - Part 3. Sample collection, storage and transport GB 17378.5 Marine monitoring code - Part 5. Sediment analysis HJ 494 water sampling technical guidance HJ 613 Determination of soil dry matter and moisture Gravimetric method HJ 783 Extraction of organic matter from soils and sediments by pressurized fluid extraction HJ/T 91 Technical Specifications for Surface Water and Sewerage Monitoring HJ/T 166 Soil Environmental Monitoring Technical Specifications

3 Method principle

Organochlorine pesticides in soils or sediments are extracted, purified, concentrated, and fixed. After using the electron capture detector Phase detection. According to the retention time of qualitative, external standard method.

4 Reagents and materials

Unless otherwise stated, analytical grade reagents that meet national standards are used. Experimental water for the new preparation of pure water or distilled water.

4.1

n-Hexane (C₆H₁₄). Chromatographic purity.

4.2

Acetone (CH₃COCH₃). Chromatography pure.

4.3

Dichloromethane (CH₂Cl₂). Chromatographic purity.

4.4

anhydrous sodium sulfate (Na₂SO₄). excellent grade pure.

Baking in a muffle furnace at 450 ° C for 4 h, cooling and placing in a glass jar with a grit stopper and holding in a desiccator.

4.5

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

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

4.6

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

Mix with acetone (4.2) and n-hexane (4.1) at a volume ratio of 1.9.

4.7

Organochlorine pesticide standard stock solution. $\rho = 10 \text{ mg/L} \sim 100 \text{ mg/L}$.

Buy a commercially available certified standard solution, store at 4 ° C in a dark, airtight refrigerator or with reference to a standard solution certificate. Make Use should be returned to room temperature and shake.

4.8

Organochlorine pesticides standard solution. $\rho = 1.0 \text{ mg/L}$.

Dilute organochlorine pesticide standard stock solution with n-hexane (4.1) (4.7). Avoid light at 4 °C closed refrigeration, storage period of six months.

4.9

Magnesium silicate solid phase extraction column. commercially available, 1000 mg/6 ml.

4.10

quartz sand. 270 µm ~ 830 µm (50 mesh ~ 20 mesh).

Baking in a muffle furnace at 450 °C for 4 h, cooling and placing in a glass jar with a grit stopper and holding in a desiccator.

4.11

Diatomaceous earth. 37 µm to 150 µm (400 mesh to 100 mesh).

Baking in a muffle furnace at 450 °C for 4 h, cooling and placing in a glass jar with a grit stopper and holding in a desiccator.

4.12

glass wool or glass fiber filter.

400 °C in a muffle furnace baking 1 h, cooled and placed in a sealed glass jar sealed.

4.13

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

4.14

Endrin and p, p'-DDT mixed standard solution. $\rho = 1.0$ mg/L.

Commercially available standard endrin and p, p'-DDT standard solutions were purchased and diluted with n-hexane (4.1). Sealed in the dark at 4 °C cold storage.

5 Instruments and equipment

5.1

Gas Chromatograph. With Electronic Capture Detector (ECD) with Split/Splitless Inlet for Programmable Temperature.

5.2 Column

5.2.1

Column 1. Column length 30 m, inner diameter 0.32 mm, film thickness 0.25 µm, stationary phase 5% polydiphenylsiloxane and 95% polydimethylsiloxane, or other equivalent column.