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

HJ 834-2017

**Soil and sediment. Determination of semivolatile organic
compounds. Gas chromatography /Mass spectrometry**

土壤和沉积物 半挥发性有机物的测定 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 Semi-volatile organic compounds in the determination of the development of this standard.

This standard specifies the determination of semi-volatile organic compounds 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 to Appendix D 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, Henan Province Environmental Monitoring Station, Henan Province Environmental Science Research Institute, Henan Entry and Exit

Inspection Quarantine Bureau Technology Center, Kaifeng City Environmental Monitoring Station, Institute of Hydrogeology and Geology, Chinese Academy of Geological Sciences, Henan Province Environment 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 semi - volatile organic compounds in soil and sediments - Gas chromatographic - 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 organic solvents and reference materials directly contact the skin Skin and clothing.

1 Scope of application

This standard specifies the determination of semi-volatile organic compounds in soil and sediments by gas chromatography-mass spectrometry.

The method is applicable to the soil and sediments of chlorinated hydrocarbons, phthalates, nitrosamines, ethers, halogen ethers, Ketones, anilines, pyridines, quinolines, nitroaromatic hydrocarbons, phenols including nitrophenols, organochlorine pesticides, polycyclic Aromatic hydrocarbons and other semi-volatile organic matter screening and quantitative analysis, for a particular class of compounds, should be selected in this foundation on the use of proprietary analytical methods.

The sampling volume was 20.0 g, the volume of volume was 1.0 ml. When the total scanning method was used, the detection limit was 0.06 mg/kg ~ 0.3 mg/kg, the determination of the lower limit of 0.24 mg/kg ~ 1.20 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 semi-volatile organic compounds in soil or sediments are extracted by suitable extraction methods (Soxhlet extraction, pressurized fluid extraction, etc.) Take the appropriate cleaning method (gel permeation chromatography or column purification) according to the sample matrix interference to purify the extract, Concentrated, constant volume, separated by gas chromatography, mass spectrometry. According to the retention time, fragment ion mass ratio and its abundance qualitative, 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

dichloromethane (CH₂Cl₂). pesticide residues.

4.3

Ethyl acetate (C₄H₈O₂). pesticide residues.

4.4

Cyclohexane (C₆H₁₂). Pesticide level.

4.5

dichloromethane - acetone mixed solvent. 1 1.

2 were mixed at a 1. 1 by volume ratio with dichloromethane (4.2) and acetone (4.1).

4.6

Gel Permeation Chromatography Mobile phase. mixed with 1 1 volume ratio with ethyl acetate (4.3) and cyclohexane (4.4), or by instrument the preparation of other solvent system.

4.7

nitric acid. rho (HNO₃) = 1.42 g/ml, excellent grade pure.

4.8

nitric acid solution. 1 1.

With excellent grade pure nitric acid (4.7) and experimental water by 1. 1 volume ratio.

4.9 Copper powder (Cu). purity 99.5%

Use the nitric acid solution (4.8) to remove the oxide on the surface of the copper powder before use, rinse off the acid with experimental water, and use acetone (4.1) After cleaning, and then use high-purity nitrogen slowly dry standby, each temporary treatment, to keep the copper powder surface bright.

4.10 semi-volatile organic standard stock solution. $\rho = 1000 \text{ mg/L} \sim 5000 \text{ mg/L}$, commercially available standard solution.

4.11 semi-volatile organic standard intermediate liquid. $\rho = .200 \text{ } \mu\text{g/ml} \sim 500 \text{ } \mu\text{g/ml}$.

The semi-volatile organic standard stock solution (4.10) was diluted with dichloromethane-acetone mixed solvent (4.5).

4.12 Internal standard stock solution. $\rho = 5000 \text{ mg/L}$

1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, ?-d12 and perylene-d12, commercially available standard solutions. You can also use other Nature of semi-volatile organic matter similar to the internal standard.

4.13 internal standard liquid. $\rho = .200 \text{ } \mu\text{g/ml} \sim 500 \text{ } \mu\text{g/ml}$.

The internal standard stock solution (4.12) was diluted with dichloromethane-acetone mixed solvent (4.5) and mixed.

4.14 Substitute stock solution. $\rho = 1000 \text{ mg/L}$ to 4000 mg/L .

Phenol-d6, 2-fluorophenol, 2,4,6-tribromophenol, nitrobenzene-d5, 2-fluorobiphenyl, 4'4'-terphenyl-d14 and other commercially available Quasi solution.

4.15 Substitute intermediate. $\rho = .200 \text{ } \mu\text{g/ml}$ to $500 \text{ } \mu\text{g/ml}$.

The stock solution (4.14) was diluted with dichloromethane-acetone mixed solvent (4.5) and mixed.

4.16 decafluorotriphenylphosphine (DFTPP). $\rho = 50 \text{ mg/L}$, commercially available standard solution.

The other concentrations were diluted with dichloromethane (4.2) to 50 mg/L and mixed.

4.17 Gel Permeation Chromatography Calibration Solution. Contains 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.

Note: All standard stock solutions from 4.10 to 4.17 should be stored in accordance with the manufacturer's instructions. All the intermediate liquids should be at -10°C Save as below. Before use should check the changes, once the evaporation or degradation should be re-preparation, should be used before the room temperature, mixing.