

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

HJ 805-2016

**Soil and Sediment - Determination of polycyclic aromatic
hydrocarbon by Gas chromatography - Mass Spectrometry
Method**

土壤和沉积物 多环芳烃的测定 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 Preparation of polycyclic aromatic hydrocarbons, the development of this standard.

This standard specifies the determination of 16 polycyclic aromatic hydrocarbons 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 Ministry of Environmental Protection Science and Technology Standards Division.

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

The standard verification unit. Henan Province Environmental Science Research Institute,

Xinxiang City Environmental Monitoring Station, Zhengzhou City Environmental Monitoring Station, opened Closed city environmental monitoring station, Chinese Academy of Geological Sciences Institute of hydrogeology and geology, environmental monitoring center in Henan Province.

The Environmental Protection Department of this standard is approved on June 24, 2016.

This standard has been implemented since August 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Determination of polycyclic aromatic hydrocarbons in soil and sediments - Gas chromatography - mass spectrometry Warning. The organic solvents and reference materials used in the experiment are toxic and hazardous substances, the standard solution preparation and the sample pretreatment process should be 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 polycyclic aromatic hydrocarbons in soil and sediments by gas chromatography-mass spectrometry.

This standard is applicable to the determination of 16 polycyclic aromatic hydrocarbons in soil and sediments. The target compounds include naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, (A) anthracene, benzo (b) fluoranthene, benzo (k) fluoranthene, benzo (a) pyrene, dibenzo (a, h) anthracene, benzene and (g, h, i) perylene and indene (1,2,3, -c, d) pyrene.

When the sampling volume is 20.0 g and the volume of volume after concentration is 1.0 ml, the detection limit of the method is 0.08 mg/kg to 0.17 mg/kg, with a lower limit of 0.32 mg/kg to 0.68 mg/kg. See Appendix A. for details.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard quasi.

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 Technical specification for soil environmental monitoring Extraction of pressurized fluid from soil and sediment organic matter

3 Principle of the method

Polycyclic aromatic hydrocarbons in soil or sediments are extracted using a suitable extraction method (Soxhlet extraction, pressurized fluid extraction, etc.) Body interference to choose the appropriate purification method (copper powder desulfurization, silica gel column, magnesium silicate column or gel permeation chromatography) on the extract purification, Concentrated, constant volume, by gas chromatography separation, mass spectrometry. Through the mass spectrometry with the standard material, retention time, fragment ion mass ratio and its abundance Comparison of qualitative and 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

pentane (C₅H₁₂). pesticide residues.

4.6

Cyclohexane (C₆H₁₂). Pesticide level.

4.7

Acetone-n-hexane mixed solvent. 1 1.

2 was mixed at a 1. 1 volume ratio with n-hexane (4.2) and acetone (4.1).

4.8 Dichloromethane-pentane mixed solvent. 2 3

Mixed with dichloromethane (4.3) and pentane (4.5) at a volume ratio of 2. 3.

4.9 Dichloromethane-n-hexane mixed solvent. 1 9

Mixed with methylene chloride (4.3) and n-hexane (4.2) at a 1. 9 volume ratio.

4.10 Gel permeation chromatography Mobile phase. Ethyl acetate (4.4) -cyclohexane (4.6) Mixing solvent (1 1), or by instrumentation His solvent system.

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

4.12 nitric acid solution. 1 1 (v/v), prepared with nitric acid (4.11).

4.13 Copper powder (Cu). purity of 99.5%

Use the nitric acid solution (4.12) to remove the oxide on the surface of the copper powder before use. Rinse the acid with the test water and rinse with acetone (4.1) After the dry with nitrogen, each pretreatment, keep the copper powder surface bright.

4.14 polycyclic aromatic hydrocarbons standard stock solution. rho = 1000 mg/L ~ 5000 mg/L, commercially available standard solution.

4.15 Polycyclic Aromatic Hydrochloride Standard Intermediate. rho =.200 µg/ml to 500 µg/ml

The polycyclic aromatic hydrocarbon standard stock solution (4.14) was diluted with acetone-n-hexane mixed solvent (4.7).

4.16 Internal standard stock solution. rho = 5000 mg/L

Naphthalene-d₈, acenaphthene-d₁₀, phenanthrene-d₁₀, -d₁₂ and perylene-d₁₂, commercially available standard solutions. Other properties can also be used similar to the semi-volatile Organic matter to do internal standard.

4.17 Internal standard liquid. rho =.200 µg/ml ~ 400 µg/ml

The internal standard stock solution (4.16) was diluted with acetone-n-hexane mixed solvent (4.7).

4.18 Substitute stock solution. rho =.2000 mg/L to 4000 mg/L, commercially available standard solution.

2-fluorobiphenyl and p-terphenyl-d₁₄; also available deuterated polycyclic aromatic hydrocarbons as substitutes.

4.19 Substitute Intermediate. rho = 500 µg/ml

The stock solution (4.18) was diluted with acetone-n-hexane mixed solvent (4.7).

4.20 decafluorotriphenylphosphine (DFTPP). rho = 50 mg/L, commercially available