

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

HJ 784-2016

**Soil and sediment. Determination of polycyclic aromatic
hydrocabons. High performance liquid chromatography**

土壤和沉积物 多环芳烃的测定 高效液相色谱法

(English Translation)

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Table of Contents

Preface

1 Scope of application

2 Normative reference document

Principle of Method

4 Reagents and materials

5 Instruments and equipment

6 Samples

7 Analysis Step

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 high performance liquid chromatography for polycyclic aromatic hydrocarbons (PAHs) compounds in soil and sediments.

This standard is the first release.

Appendix A of this standard is a normative appendix, Appendix B is an informative appendix.

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.

This standard is verified by. Institute of Hydrogeology and Geology and Environmental Geology, Chinese Academy of Geological Sciences, Zhengzhou Environmental Protection Monitoring Center Station, Kaifeng City Environmental Monitoring Station, Luoyang City Environmental Monitoring Station, Xinxiang City Environmental Monitoring Station.

This standard is approved by the Ministry of Environmental Protection on February 1, 2016.

This standard has been implemented since March 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Determination of polycyclic aromatic hydrocarbons in soil and sediments - High performance liquid chromatography Warning. part of the polycyclic aromatic hydrocarbons

are strong carcinogens, the operation should be required to wear protective equipment, to avoid contact with the skin and clothes. Solution preparation and sample pretreatment should be carried out in a fume hood.

1 Scope of application

This standard specifies high performance liquid chromatography for the determination of polycyclic aromatic hydrocarbons in soil and sediments.

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

When the sampling volume is 10.0 g and the volume of volume is 1.0 ml, the method of 16 kinds of polycyclic aromatic hydrocarbons is determined by UV detector.

And the limit of determination was 12 µg/kg ~ 20 µg/kg. The 16 kinds of polycyclic aromatic compounds the detection limit of the method is 0.3 µg/kg ~ 0.5 µg/kg, and the limit of determination is 1.2 µg/kg ~ 2.0 µg/kg. See Appendix A. for details.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Any unspecified 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 Technical specification for soil environmental monitoring

3 Principle of the method

The polycyclic aromatic hydrocarbons (PAHs) in soil and sediment samples were extracted with a suitable extraction method (Soxhlet extraction, pressurized fluid extraction, etc.) According to the sample matrix interference to take appropriate purification methods (silica gel column, silica gel or magnesium silicate solid phase extraction column, etc.) Extraction solution for purification, concentration, constant volume, equipped with UV/fluorescence detector high-performance liquid chromatography separation test to protect Time qualitative, external standard method.

4 Reagents and materials

Unless otherwise stated, analytical reagents and experimental water are used in accordance with national standards.

4.1

acetonitrile (CH_3CN). HPLC grade.

4.2

n-hexane (C_6H_{14}). HPLC grade.

4.3

dichloromethane (CH_2Cl_2). HPLC grade.

4.4

Acetone (CH_3COCH_3). HPLC grade.

4.5

Acetone-n-hexane mixed solution. 1 1.

Mix with acetone (4.4) and n-hexane (4.2) in a 1. 1 by volume ratio.

4.6

Dichloromethane-n-hexane mixed solution. 2 3.

Mixed with dichloromethane (4.3) and n-hexane (4.2) at a volume ratio of 2. 3.

4.7

Dichloromethane-n-hexane mixed solution. 1 1.

2 was mixed with methylene chloride (4.3) and n-hexane (4.2) in a 1. 1 by volume ratio.

4.8

Polycyclic aromatic hydrocarbon standard stock solution. $\rho = 100 \text{ mg/L} \sim 2000 \text{ mg/L}$.

Purchase a commercially available standard solution, store it at 4°C , store it in a dark place, or store it with reference to a standard solution certificate. Make Use should be restored to room temperature and shake.

4.9

Polycyclic aromatic hydrocarbons standard use of liquid. $\rho = 10.0 \text{ mg/L} \sim 200 \text{ mg/L}$.

Remove the 1.0 ml polycyclic aromatic hydrocarbon standard stock solution (4.8) in a 10 ml brown flask, diluted with acetonitrile (4.1) and set to the scale, shake, transferred to the dense bottle at 4°C in the cold, dark preservation.

4.10

Diphenyl biphenyl (C₁₂F₁₀). 99% purity.

Alternatives, other analogues may also be used.

4.11

Decafluorobenzene stock solution. $\rho = 1000 \text{ mg/L}$.

Weigh decafluorobenzene (4.10) 0.025 g (exact to 0.001 g), dissolve with acetonitrile (4.1) and set to 25 ml Color volume bottle, shake, transfer to a dense bottle in the 4 °C cold storage, dark preservation. Or purchase a commercially available standard solution.

4.12

Use of decafluorobenzene. $\rho = 40 \text{ }\mu\text{g/ml}$.

Remove the 1.0 ml solution of tetradecafluorobenzene (4.11) in a 25 ml brown volumetric flask, dilute with acetonitrile (4.1) and set the volume to the scale, shake, transferred to the dense bottle at 4 °C in the cold, dark to save.

4.13 Desiccant. anhydrous sodium sulfate (Na₂SO₄) or granular diatomaceous earth

Placed in the muffle furnace 400 °C bake 4 h, after cooling placed in the grinding glass bottle sealed preservation.

4.14 Silica gel. particle size 75 μm ~ 150 μm (200 mesh ~ 100 mesh).

Before use, should be placed in a flat bottom tray, with aluminum foil loosened, 130 ° C activated at least 16 h.

4.15 glass column. diameter of about 20 mm, length 10 cm ~ 20 cm, with PTFE piston.

4.16 silica gel solid phase extraction column. 1000 mg/6 ml.

4.17 magnesium silicate solid phase extraction column. 1000 mg/6 ml.

4.18 quartz sand. particle size 150 μm ~ 830 μm (100 mesh ~ 20 mesh), before use to be tested to confirm that no interference.

4.19 glass wool or glass fiber filter. muffle furnace in 400 °C baking 1 h, after cooling placed in the grinding glass bottle sealed.

4.20 Nitrogen. Purity $\geq 99.999\%$.

5 Instruments and equipment