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HJ

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

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**Soil and sediment--Determination of polychlorinated
biphenyls--Gas chromatography**

土壤和沉积物 多氯联苯的测定 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 PCBs, the development of this standard.

This standard specifies the gas chromatography determination of 18 kinds of PCBs in soil and sediment.

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 Environmental Monitoring Center Station, Henan Province Environmental Monitoring Center, Nanjing Environmental Monitoring Center Station, Dalian City Environmental Monitoring Center, Nanjing Environmental Protection Research Institute and the Nanjing Entry-Exit Inspection and Quarantine Electronic and Electrical Products 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 sediment - Determination of PCBs - Gas chromatography Warning. The solvents and reagents used in the experiment have some toxicity, some PCBs are carcinogenic, the standard Solution preparation and sample preparation process should be carried out in a fume hood; operation should be worn protective equipment, avoid direct contact with the skin and clothing.

1 Scope of application

This standard specifies the determination of PCBs in soil and sediment by gas chromatography.

This standard applies to the determination of 7 indicative PCBs and 12 coplanar polychlorinated biphenyls in soils and sediments. other PCBs, if verified, can also be measured using this standard.

When the sample volume is 10.0 g, the detection limit of PCBs is 0.03 µg/kg ~ 0.07 µg/kg, and the lower limit of determination 0.12 µg/kg to 0.28 µg/kg. See Appendix A for details.

2 Normative references

This standard references the following documents or the terms of them. 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

3.1 Indicative PCBs PCBs

Refers to PCBs that are used as alternative monitors for the contamination of PCBs, including 2,4,4'-trichlorobiphenyl (PCB28), 2,2', 5,5'-tetrachlorobiphenyl (PCB52), 2,2', 4,5,5'-pentachlorobiphenyl (PCB101), 2,3', 4,4', 5-pentachlorophenol (PCB118), 2,2',

3,4,4', 5'-hexachlorobiphenyl (PCB138), 2,2', 4,4', 5,5'-hexachlorobiphenyl (PCB153), 2,2', 3,4,4', 5,5'-heptachlorobiphenyl (PCB180) and other seven compounds.

Co-planar PCBs coplanar PCBs Refers to polychlorinated biphenyls that are not ortho or ortho-ortho substituted in PCBs and are similar in their toxicity to dioxins, including 3,3', 4,4'-tetrachlorobiphenyl (PCB77), 3,4,4', 5-tetrachlorobiphenyl (PCB81), 2,3,3', 4,4'-Benzene (PCB105), 2,3,4,4', 5-Pentachlorobenzene (PCB114), 2,3', 4,4', 5-Pentachlorobenzene (PCB118), 2', 3,4,4', 5 - Pentachlorobenzene (PCB123), 3,3', 4,4', 5-Pentachlorobenzene (PCB126), 2,3,3', 4,4', 5-Hexachlorobiphenyl (PCB156), 2,3,3', 4,4', 5'-hexachlorobiphenyl (PCB 157), 2,3', 4,4', 5,5'-hexachlorobiphenyl (PCB 167), 3,3', 4,4', 5,5'-hexachlorobiphenyl 2,3,3', 4,4', 5,5'-heptachlorobiphenyl (PCB189) and other 12 kinds of compounds. Among them 2,3', 4,4', 5-Pentachlorobenzene (PCB118) also is indicative of PCBs.

4 Method principle

PCBs in soils or sediments are extracted, purified, concentrated and fixed. Electron capture detectors Gas chromatography. According to the retention time of qualitative, external standard method.

5 Interference and elimination

Other organic impurities in the sample, interference PCBs determination, the choice of sulfuric acid, magnesium silicate column, such as appropriate net Method to remove. After purification, there will still be other compounds and the target compound at the same time peak, interference determination, through another Different polarity of the column auxiliary qualitative. If required, confirm that the sensitivity meets the requirements by gas chromatography-mass spectrometry.

6 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.

6.1

n-Hexane (C₆H₁₄). Chromatographic purity.

6.2

Acetone (CH₃COCH₃). Chromatography pure.

6.3

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.

6.4

Potassium carbonate (K_2CO_3). excellent grade pure.

6.5

Sulfuric acid. $\rho(H_2SO_4) = 1.84 \text{ g/ml}$.

6.6

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

Mix with acetone (6.2) and n-hexane (6.1) at a volume ratio of 1.1.

6.7

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

Mix 1.9 volumes with acetone (6.2) and n-hexane (6.1).

6.8

Potassium carbonate solution. $\rho = 0.1 \text{ g/ml}$.

Weigh 100 g of potassium carbonate (6.4) dissolved in water, set to 1000 ml.

6.9

Polychlorinated biphenyls standard stock solution. $\rho = 10.0 \text{ mg/L}$ to 100 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.

6.10

PCB standard solution. $\rho = 1.0 \text{ mg/L}$ (reference concentration).

Dilute PCB stock standard solution with n-hexane (6.1) (6.9). Avoid light at 4°C closed refrigeration, storage period of six months.

6.11

Magnesium silicate solid phase extraction column. commercially available, 1000 mg/6 ml. Or according to the impurity content to choose other capacity of magnesium silicate SPE column.

6.12

quartz sand. $270 \mu\text{m} \sim 830 \mu\text{m}$ (50 mesh $\sim$ 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.