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

HJ 743-2015

**Soil and sediment. Determination of polychlorinated biphenyls
(PCBs). Gas chromatography mass spectrometry**

土壤和沉积物 多氯联苯的测定 气相色谱-质谱法

(English Translation)

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Table of Contents

Foreword
11 Scope of application
46 Instruments and equipment
78 Analysis steps

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 the determination of PCBs, the development of this standard.

This standard specifies the determination of polychlorinated biphenyls in soil and sediments by gas chromatography-mass spectrometry.

This standard is the first release.

Appendix A of 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. Jiangsu Province Environmental Monitoring Center.

The standard verification unit. Taizhou City Environmental Monitoring Center Station, Henan Province Environmental Monitoring Center, Nanjing Environmental Monitoring Center Station, Dalian Environmental Monitoring Center, Ministry of Environmental Protection Nanjing Environmental Protection Science Research Institute and Nanjing Entry-Exit Inspection and Quarantine Bureau Electronic and Electrical Products Laboratory.

The environmental protection department of this standard approved on May 4, 2015.

This standard has been implemented since July 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

11 Scope of application

This standard specifies the determination of polychlorinated biphenyls in soil and sediments by gas chromatography-mass spectrometry.

This standard applies to the determination of seven indicative PCBs and 12 coplanar

polychlorinated biphenyls in soil and sediments. other Polychlorinated biphenyls can also be determined by this method if verified.

When the sampling amount is 10.0 g, the detection limit of PCB method is 0.4 ~ 0.6 µg/kg when the selected ion scanning mode is adopted, the lower limit of determination is 1.6 ~ 2.4 µg/kg, see Appendix A.

2 normative reference documents the contents of this standard refer to the following documents or their terms. For those who do not mind the date of the reference file, the valid version applies in this standard.

GB 17378.3 Marine Monitoring Specification Part 3 Sample Collection Storage and Transportation GB 17378.5 Marine monitoring regulations Part 5 Sediment analysis Determination of Dry Matter and Moisture in Soil Technical specification for soil environmental monitoring 3 terms and definitions 3.1 Indicative PCBs Soil and sediment - Determination of polychlorinated biphenyls - Gas chromatographic - mass spectrometry WARNING. The solvents and reagents used in the test are of some toxicity and some PCBs are highly carcinogenic, the process should be carried out in a fume hood, operating in accordance with the requirements of wearing protective equipment, to avoid solvent and reagents direct contact with the skin Skin and clothing.

2 refers to PCBs that are used as alternative monitoring of PCB contamination.

Coplanar PCBs

Refers to non-contiguous or mono-ortho-substituted polychlorinated biphenyls in polychlorinated biphenyls, which is similar to dioxins.

4 principle of the method Extraction of polychlorinated biphenyls (PCBs) in soils or sediments by appropriate extraction methods (microwave extraction, ultrasonic extraction, etc.) According to the sample matrix interference, select the appropriate purification method (concentrated sulfuric acid sulfonation, copper powder desulfurization, Florisil column, silica gel column and other gel permeation purification column), the extract purification, concentration, constant volume, with gas chromatography - mass spectrometry separation, detection, within Quantitative method.

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

5.1 Toluene (C₇H₈). Chromatographic pure 5.2 n-hexane (C₆H₁₄). pure chromatography.

5.3 Acetone (CH₃COCH₃). Chromatographic pure.

5.4 anhydrous sodium sulfate (Na₂SO₄). excellent grade pure.

In the muffle furnace at 450 °C baking 4 hours after the cooling, placed in the dryer glass bottle in reserve.

5.5 Potassium carbonate (K_2CO_3). excellent grade pure.

5.6 nitric acid. $\rho(HNO_3) = 1.42 \text{ g/ml}$.

5.7 Nitric acid solution. 1 9.

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

5.9 N-Hexane - Acetone Mixed Solvents. 1 1.

Mixed with n-hexane (5.2) and acetone (5.3) in a 1. 1 by volume ratio.

5.10 N-Hexane - Acetone Mixed Solvent. 9 1.

Mixed with n-hexane (5.2) and acetone (5.3) at a volume ratio of 9. 1.

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

Weigh 1.0 g of potassium carbonate (5.5) in water and set to 10.0 ml.

5.12 Copper powder (Cu). 99.5%.

3 before use with nitric acid solution (5.7) to remove the copper oxide surface of the oxide, with distilled water to remove the residual acid, and then acetone cleaning, and drying the copper powder under a nitrogen stream so that the copper powder has a bright surface.
Pre-treatment.

5.13 Polychlorinated biphenyl standard stock solution. $\rho = 10 \sim 100 \text{ mg/L}$.

Prepared with n-hexane dilution of pure standard material, the standard solution at 4°C under the light sealed cold storage, can be stored for six months. Can also be straight (See Polychlorinated Biphenyl Mixed Standard Solution or Single Component Polychlorinated Biphenyl Standard Solution), Storage Time See also A description of the standard solution certificate.

5.14 Standard use of polychlorinated biphenyls. $\rho = 1.0 \text{ mg/L}$ (reference concentration).

The standard stock solution (5.13) was diluted with n-hexane (5.2).

5.15 internal standard stock solution. $\rho = 1000 \sim 5000 \text{ mg/L}$.

Select 2,2', 4,4',5,5'-hexabromobiphenyl or o-nitrobromobenzene as internal standard; when decachlor biphenyl is a non-test compound, Use of decachlor as an internal standard. Can also be purchased directly to prove the standard solution.

5.16 Internal standard liquid. $\rho = 10 \text{ mg/L}$ (reference concentration).

The internal standard stock solution (5.15) was diluted with n-hexane (5.2).

5.17 Substitute stock solution. $\rho = 1000 \sim 5000 \text{ mg/L}$.

When 2,2', 4,4',5,5'-hexabromobiphenyl or tetrachloro-m-xylene is selected as a substitute, when the decabenium is a non-test compound, Also available as a substitute for decachlor.

Can also be purchased directly to prove the standard solution.

5.18 Substitute liquid. $\rho = 5.0 \text{ mg/L}$ (reference concentration).

The stock solution (5.17) was diluted with acetone (5.3).

5.19 decafluorotriphenylphosphine (DFTPP) solution. $\rho = 1000 \text{ mg/L}$, the solvent is methanol
5.20 ten fluorotriphenylphosphine use of liquid. $\rho = 50.0 \text{ mg/L}$ Transfer to $500 \mu\text{l}$ of decafluorotriphenylphosphine (DFTPP) solution (5.19) to a 10 ml volumetric flask with n-hexane (5.2) Capacity to mark, mix.

5.21 Florisil Column. 1000 mg, 6 ml.

5.22 Silica gel column. 1 000 mg, 6 ml.

5.23 Graphite carbon column. 1000 mg, 6 ml.

5.24 quartz sand. 20 ~ 50 mesh in the muffle furnace at 450°C baking 4 hours after the cooling, placed in a glass bottle in the dryer to save.

5.25 diatomaceous earth (100 to 400 mesh) in the muffle furnace at 450°C baking 4 hours after the cooling, placed in a glass bottle in the dryer to save.

46 Instruments and equipment

6.1 Gas Chromatography-Mass Spectrometer. Capillary Split/Splitless Inlet with Constant or Constant Pressure Function; Column Thermostat Programmable Temperature; with EI source.

6.2 Column. quartz capillary column, length 30 m, inner diameter 0.25 mm, film thickness $0.25 \mu\text{m}$, stationary phase 5% - phenyl-methyl Polysiloxane, or equivalent column.

6.3 Extraction device. microwave extraction device, Soxhlet extraction device, probe type ultrasonic extraction device or a device with considerable function.

Need to be used before and in the use of blank test, all interfaces are strictly prohibited the use of grease lubricant.

6.4 Concentration device. nitrogen blowing concentrator, rotary evaporator, KD concentrator or equipment with considerable function.

6.5 Sampling vials. wide mouth brown glass bottle or polytetrafluoroethylene liner screw glass bottle.

6.6 Common laboratory equipment and equipment.

7 samples

7.1 Collection and storage of samples Soil samples were collected and stored in accordance with the relevant requirements of HJ/T 166, and the sediment samples were in