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**HJ**

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

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**Soil and sediment. Determination of polychlorinated biphenyl  
mixtures. Gas chromatography**

土壤和沉积物 多氯联苯混合物的测定 气相色谱法

**(English Translation)**

**Issued on: December 17, 2017**

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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, regulate soil and sediment Determination of PCBs in industrial mixtures, the development of this standard.

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

This standard Appendix A, Appendix B and Appendix C are informative appendix.

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. National Environmental Analysis Test Center.

This standard verification unit. Hunan Provincial Environmental Monitoring Center, Xiangtan City Environmental Monitoring Station, Wuxi City Environmental Monitoring Center, the Soviet Union State Environmental Monitoring Center, Shandong Provincial Analysis and Testing Center and Beijing Jinxiu earth technology testing and analysis center.

This standard MEP approved on December 17, 2017.

This standard since February 1, 2018 implementation.

This standard is interpreted by the MEP.

### 1 Soil and sediment polychlorinated biphenyls Determination of gas chromatography

Warning. The organic solvents and reagents used in this method have some toxicity. The experimental operation should be carried out in a fume hood and press Provisions on

wearing protective equipment, to avoid contact with skin and clothing.

1 scope of application this standard specifies the determination of soil and sediment in the PCB by gas chromatography.

This standard applies to soil and sediment PCB1221, PCB1242, PCB1248, PCB1254 and PCB1260 A total of 5 kinds of PCBs determination of industrial products, other PCBs industrial products through the verification can also be measured using this method.

When the sample volume is 5 g and the volume of constant volume is 1.0 ml, the detection limits of the 5 PCBs industrial products by this method are 5 µg/kg, the lower limit of determination of 20 µg/kg.

## 2 Normative references

This standard references the following documents or the terms. For undated references, the effective version applies to this book 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 166 Soil Environmental Monitoring Technical Specifications

## 3 Terms and definitions

The following terms and definitions apply to this standard.

PCBs congeners

Polychlorinated biphenyls (PCBs), also known as chlorinated biphenyl, ring is a hydrogen atom is replaced by a class of chlorine atoms formed Synthetic organic compounds, from a chlorinated biphenyl to 10 chlorinated biphenyl, a total of 209 kinds of compounds, although the molecular formula of these compounds are different, but there is a similar chemical structure, known as homologues.

Feature recognition peak diagnostic peak each polychlorinated biphenyl industrial product has between 70 and 80 identifiable peaks in the GC/ECD spectrum. Some of these chromatograms Peak abundance larger, better separation, and the relative position and intensity of the surrounding peaks have obvious characteristics, easy to identify, known as special Sign recognition peak.

## 24 Method principle

PCBs in soils and sediment samples were extracted with organic solvents and the extract was concentrated with concentrated sulfuric acid and purified on a silica gel column.

After volume determination by gas chromatography, electron capture detector detection. Retention time and peak shape of the sample peak passed the standard Qualitative comparison of products, select 5 to 10 characteristics of identification peaks, with external standard quantitative.

5 Interference and elimination Soil and sediments may exist in the BHC, organophosphorus pesticides, oxygenated compounds will not interfere with the determination of PCBs.

DDE, DDD and DDE at concentrations greater than 5 µg/kg interfere with quantification and should be avoided in the selection of characterization peaks the peak time of the thing.

6 Reagents and materials Unless otherwise stated, analytical grade reagents that meet national standards are used.

6.1 Experimental water in a 1 L separatory funnel by adding 500 ml of deionized or distilled water and 100 ml of n-hexane, shaking 10 min standing After the layer, the aqueous phase is released and stored in brown glass bottles for later use. Hexane can be reused 3 times.

6.2 n-Hexane (C<sub>6</sub>H<sub>14</sub>). pesticide residues, or PCBs were not detected after 100-fold concentration.

6.3 Dichloromethane (C<sub>2</sub>H<sub>2</sub>Cl<sub>2</sub>). pesticide grade, or 50 times concentrated PCBs were not detected.

6.4 acetone (C<sub>3</sub>H<sub>6</sub>O). pesticide residues, or PCBs were not detected.

6.5 Absolute ethanol (C<sub>2</sub>H<sub>5</sub>OH). HPLC grade, PCBs were not detected after 100 times concentration.

6.6 Nitric acid. rho (HNO<sub>3</sub>) = 1.42 g/ml.

6.7 Sulfuric acid. rho (H<sub>2</sub>SO<sub>4</sub>) = 1.84 g/ml.

6.8 Potassium hydroxide (KOH). Purity 85%.

6.9 Sodium bicarbonate (NaHCO<sub>3</sub>).

6.10 Anhydrous sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>).

Muffle furnace 450 °C burning 4 h, remove and put in a clean dryer standby.

6.11 Dichloromethane - n-hexane mixed solution. 1 1.

Mix with methylene chloride (6.3) and n-hexane (6.2) at a volume ratio of 1. 1.

6.12 nitric acid solution. rho (HNO<sub>3</sub>) = 1 mol/L.

Take 6.9 ml of nitric acid (6.6), add 100 ml of water and mix well.

6.13 sulfuric acid solution. 1 9.

Take 10 ml of sulfuric acid (6.7), add 90 ml of water and mix.

6.14 Potassium hydroxide solution.  $\rho(\text{KOH}) = 0.05 \text{ g/ml}$ .

Take 59 g potassium hydroxide (6.8) dissolved in a small amount of water, diluted to 1 L.

6.15 Potassium hydroxide - ethanol solution. 1 mol/L.

Weigh 33 g of potassium hydroxide (6.8) added to 500 ml of anhydrous ethanol (6.5), dissolved and mixed.

6.16 Sodium bicarbonate solution.  $\rho(\text{NaHCO}_3) = 0.02 \text{ g/ml}$ .

3 Weigh 2 g of sodium bicarbonate (6.9), dissolve in 100 ml of water and mix well.

6.17 PCB standards

PCB1221, PCB1242, PCB1248, PCB1254, PCB1260 and other commercial PCB standards.

**Note:** Some of the commercially available PCB series of standard samples of methanol solvent, methanol and n-hexane, petroleum ether and other non-polar solvents can not be any ratio Dilution ratio should be greater than 15. 1.

6.18 PCB stock standard solution.  $\rho = 5.00 \text{ mg/L}$ .

Dilute PCB standards with n-hexane (6.2) (6.17). Dark sealed in a standard sample bottle cold, at least Save for 6 months.

6.19 copper wire (beads or powder).

Before use soaked in nitric acid solution (6.12) to remove the surface oxide layer, washed with water until neutral and then with acetone (6.4) and n-hexane (6.2) three times.

6.20 Silica.  $75 \mu\text{m} \sim 150 \mu\text{m}$  (200 mesh  $\sim$  100 mesh).

130 °C activated 16 h, placed in a clean dryer standby.

6.21 alkaline silica gel.

Take silica gel (6.20) 98 g, add potassium hydroxide solution (6.14) 40 ml, shake well, into a powder, after bottling Store in the dryer.

6.22 acidic silica gel.

Take silica gel (6.20) 56 g, add sulfuric acid (6.7) 44 g, shake well, into a powder, stored in a dry bottle in the device.

6.23 Compound Silica Column. The following methods can be used to fill, you can also buy commercially available products.

Place a small glass wool (6.27) on the bottom of the column (7.6), add 40 ml of n-hexane (6.2) and fill in with water 1 g of sodium sulfate (6.10), 1 g of silica gel (6.20), 3 g of basic silica gel (6.21), 1 g of silica gel (6.20) 8 g, silica gel (6.20) 1 g, anhydrous sodium sulfate