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HJ

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

HJ 903-2017

**Ambient air. Determination of polychlorinated biphenyls. Gas
chromatography**

环境空气 多氯联苯的测定 气相色谱法

(English Translation)

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1 Ambient air Determination of polychlorinated biphenyls Gas chromatography

Warning. The solvents and reagents used in this method are toxic. The experimental procedure should be carried out in a fume hood.

Wear protective equipment as required and avoid contact with skin and clothing.

1 scope of application this standard specifies the determination of PCBs in ambient air by gas chromatography.

This standard applies to ambient air and particulate matter in the PCB 8, PCB 18, PCB 28, PCB 44, PCB 52, PCB 66, PCB 77, PCB 81, PCB 101, PCB 105, PCB 114, PCB 118, PCB 123, PCB 126, PCB 128, PCB 138, PCB 153, PCB 156, PCB 157, PCB 167, PCB 169, PCB 170, PCB 180, PCB 187, PCB 189, PCB 195 and PCB 206 Determination of a total of 27 kinds of PCBs. See Appendix B for details.

When the sampling volume is 350 m³ (standard state) and the volume of concentrated volume is 1.0 ml, the method measures 27 kinds of polychlorine the detection limit of

biphenyl is between 0.01 ng/m³ and 0.04 ng/m³, and the lower limit of determination is between 0.04 ng/m³ and 0.16 ng/m³. See Appendix A for details.

2 Normative references

This standard references the following documents in the terms. For undated references, the effective version applies this standard.

HJ 194 Manual Air Quality Monitoring Technical Specifications HJ 691 Environmental airborne semi-volatile organic compounds sampling techniques

3 Method principle

A large flow sampler is used to collect the filters and polyurethane foam from the PCBs in ambient air and particulate matter (PUF), extracted with ether-n-hexane mixed solvent, the extract was concentrated, purified, gas chromatographic separation, electron Capture detector detection, according to the retention time qualitative, internal standard method or external standard method.

4 Interference and elimination

Other organic matter in the sample may interfere with the determination, the choice of sulfuric acid, magnesium silicate chromatography column or a composite silica gel column Methods to remove interference, see 7.3.3.

5 Reagents and materials

Unless otherwise specified, analytical grade analytical reagents used in accordance with national standards were used. Experimental water is freshly prepared Pure water.

5.1

acetone (C₃H₆O). pesticide grade.

5.2

n-hexane (C₆H₁₄). pesticide residues.

5.3

ether (C₄H₁₀O). Chromatography.

5.4

Dichloromethane (CH₂Cl₂). pesticide grade.

25.5 Anhydrous Sodium Sulfate (Na₂SO₄). Bake at 400 °C in muffle furnace for 4 hours before use, after cooling, Sealed in the preservation.

5.6

Sodium chloride (NaCl). Bake in a muffle furnace at 400 ° C for 4 h before use, cool and seal in a milled glass jar save.

5.7

sulfuric acid (H₂SO₄). rho = 1.84 g/cm³, excellent grade pure.

5.8

ether - n-hexane mixed solvent. 1 9, Pro use now.

5.9

Diethyl ether - n-hexane mixed solvent. 6 94, Pro use now.

5.10

Sodium chloride solution. rho = 50.0 g/L.

Weigh 50.0 g of sodium chloride (5.6), dissolved in a beaker of water and set to 1000 ml, mix, Pro with the current.

5.11

Substitute stock solution. rho = 500 µg/ml.

Direct purchase of a certified commercial standard solution, 2,4,5,6-tetrachloro-m-xylene (TCX), deca-biphenyl (DCBP) single Solution. Other suitable alternatives may also be used.

5.12

Substitute intermediate solution. rho = 50.0 µg/ml.

Pipette 1.00 ml TCX stock solution (5.11) in a 10 ml volumetric flask with n-hexane (5.2), and mix well.

5.13

Alternative solution. rho = 1.00 µg/ml.

Pipette 1.00 ml each of TCX and DCBP replacement stock (5.11) in a 50 ml volumetric flask with n-hexane (5.2) Set the volume, mix well.

5.14

Internal standard stock solution. rho = 1000 µg/ml.

Direct purchase of a certified commercial standard solution containing 1-bromo-2-nitrobenzene (BNB).

5.15

Internal standard intermediate solution. rho = 100 µg/ml.

Pipette 1.00 ml internal standard stock solution (5.14) in 10 ml volumetric flask, with

n-hexane (5.2) volume, mix.

5.16

Internal standard solution. $\rho = 10.0 \mu\text{g/ml}$.

Pipette 1.00 ml internal standard solution (5.15) in 10 ml volumetric flask, with n-hexane (5.2) volume, mix.

5.17

standard stock solution. $\rho = 10.0 \mu\text{g/ml}$.

Direct purchase of 28 kinds of commercially available certified standard solution, containing components IUPAC number PCB 8, PCB 18, PCB 28, PCB 44, PCB 52, PCB 66, PCB 77, PCB 81, PCB 101, PCB 105, PCB 114, PCB 118, PCB 123, PCB 126, PCB 128, PCB 138, PCB 153, PCB 156, PCB 157, PCB 167, PCB 169, PCB 170, PCB 189, PCB 180, PCB 187, PCB 195, PCB 206 and DCBP, see Appendix B. 4 °C the following sealed Paul Save, or reference standard solution certificate preservation conditions.

5.18

standard solution. $\rho = 1.00 \mu\text{g/ml}$.

Pipette 1.00 ml standard stock solution (5.17) and 200 μl intermediate solution containing 2,4,5,6-tetrachloro-m-xylene (5.12) in 10 ml volumetric flask, with n-hexane (5.2) constant volume, mix well.

Note: All solutions (5.11 ~ 5.18) are transferred to a screw-capped glass bottle with Teflon liner, refrigerated below 4 °C, sealed from light save.

5.19 Composite silica gel column

The bottom of the column filled with glass wool (5.24), from bottom to top were filled with anhydrous sodium sulfate 4 g, silica gel 0.9 g, 2% KOH Silica gel 3 g, silica gel 0.9 g, 44% silica gel 4.5 g, 22% silica gel 6 g, silica gel 0.9 g, 10% silver nitrate silica gel 3 g,

3 The top layer is anhydrous sodium sulfate 6 g. Can also be purchased directly commodity column.

5.20 Magnesium silicate. 150 μm to 250 μm (100 mesh to 60 mesh), activated at 130 °C for at least 18 h before use, After cooling in the desiccator, transfer to a glass bottle and seal it for preservation.

5.21 quartz/glass fiber filter. According to the sampling head to select the appropriate specifications, the filter on the 0.3 μm standard particle retention efficiency not less than 99%. Before use in the muffle furnace 400 °C heating 5 h above, after cooling, stored in the filter box, to ensure that the filter at Sampling before and after not contaminated, and in the sample before the flat state.