

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 1224-2021

**Ambient air-Determination of organochlorine pesticides-High
resolution gas chromatography/high resolution mass
spectrometry**

环境空气 有机氯农药的测定 高分辨气相色谱-高分辨 质谱法

(English Translation)

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1 Scope of application

This standard specifies the high-resolution gas chromatography-high-resolution mass spectrometry method for the determination of organochlorine pesticides in ambient air.

This standard is applicable to hexachlorobenzene, alpha-hexahexahexanone, gamma-hexahexahexanone, beta-hexahexahexanol, delta-hexachlorobenzene, heptachlor, Aldrin, chlordane oxide, cis-epoxyheptachlor, trans-epoxyheptachlor, trans-chlordane, 2,4'-DDE, trans-nonachlor, cis-chlordane, Endosulfan-I, 4,4'-DDE, dieldrin, 2,4'-DDD, endrin, 2,4'-DDT, cis-nonachloro, 4,4'-DDD, endosulfan -II, 4,4'-DDT Determination of 25 organochlorine pesticides, including mirex. See Appendix A.

When the sampling volume is 30 m³ (standard state) and the concentration volume is 20 µl, the detection limit of HCB is 0.9 pg/m³.

The limit is 3.6 pg/m³; when the sampling volume is 1200 m³ (standard state) and the concentration volume is 20 µl, other organochlorines except hexachlorobenzene the detection limit of pesticides is 0.006 pg/m³~0.03 pg/m³, and the lower limit of determination is 0.024 pg/m³~0.12 pg/m³. See Appendix B for details.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this standard.

HJ 194 Technical Specification for Manual Monitoring of Ambient Air Quality HJ 691
Technical Guidelines for Sampling of Semi-volatile Organic Compounds in Ambient Air HJ
900 Determination of Organochlorine Pesticides in Ambient Air Gas Chromatography-Mass
Spectrometry

3 Principles of the method

This method uses an active sampler to collect ambient air particulate matter and organochlorine pesticides in the gas phase to filter membranes and polyurethane foam (PUF) Add isotope-labeled extraction internal standard to the sampled filter membrane and PUF, extract with n-hexane-dichloromethane mixed solvent, and extract the solution.

After concentration, purification and other operations, an isotope-labeled injection internal standard was added to it, and high-resolution gas chromatography-high-resolution mass spectrometry was used for separation and detection.

According to the retention time and the monitoring ion abundance ratio, the isotope dilution method was used for quantification.

4 Interference and cancellation

Other organic substances in the sample may interfere with the determination. Select a purification column such as Florisil, graphitized carbon black, etc. to remove the interference, see 7.3.4 for details.

5 Reagents and Materials

Unless otherwise stated, superior-grade pure reagents meeting national standards were used in the analysis, and the experimental water was newly prepared pure water.

5.1

Acetone (C₃H₆O). pesticide residue grade.

5.2

n-hexane (C₆H₁₄). pesticide residue grade.

5.3

Dichloromethane (CH₂Cl₂). pesticide residue grade.

5.4

Nonane (C₉H₂₀). pesticide residue grade.

5.5

Toluene (C₇H₈). pesticide residue grade.

5.6

n-hexane-dichloromethane mixed solvent.

n-Hexane (5.2) and dichloromethane (5.3) were mixed in a 1.1 volume ratio.

5.7

Anhydrous sodium sulfate (Na₂SO₄). bake in a muffle furnace at 400 °C for 4 h, cool and seal it in a ground glass bottle with a stopper, and dry it.

saved in the device.

5.8

Internal standard extraction. select isotope-labeled compounds as the internal standard for extraction, see Appendix C.1. Commercially available certified reference materials can be purchased directly (solution).

5.9

Internal standard for injection. Select the isotope-labeled compound as the internal standard for injection, see Appendix C.1. Commercially available certified standards can be purchased directly quality (solution).

5.10

Organochlorine pesticide standard solution series. refers to the mixture of the organochlorine pesticide standard substance and the corresponding internal standard substance prepared with nonane or other solvents.

mixture. The mass concentration of the standard solution is accurately known, and the mass concentration series should cover the quantification of high-resolution gas chromatography-high-resolution mass spectrometry Linear range, including 5 or more mass concentration gradients, see Appendix C.2. Commercially available certified reference

materials (solutions) can be purchased directly.

5.11

Florisil solid phase extraction column. 1 g, 74 μm ~150 μm (200 mesh~100 mesh), the column volume is 6 ml~10 ml.

5.12

Graphitized carbon black solid phase extraction column. 500 mg, 38 μm ~ 125 μm (400 mesh ~ 120 mesh), the column volume is 6 ml ~ 10 ml.

5.13

Quartz/glass fiber membrane. the retention efficiency of 0.3 μm standard particles is not less than 99%. Bake in a muffle furnace at 400 °C before use Bake for 5 h, cool to room temperature, and store in a vacuum drying oven.

5.14

Polyurethane Foam (PUF). Commonly used density is 0.022 g/cm³. Wash it with boiling water before use, then put it in warm water Repeatedly scrub for more than 2 times, drain the water, put it in the oven to remove the water, and then use the following method to extract and clean the PUF (you can also use the processed by other equivalent methods).

Soxhlet extraction and cleaning. The extraction solvent is n-hexane-dichloromethane mixed solvent (5.6). For other extraction conditions, please refer to the relevant information in HJ 900.

content. The cleaned PUF was heated in a vacuum drying oven at 50 °C until the solvent was completely evaporated, and then placed in a vacuum drying oven for vacuum storage.

Pressurized fluid extraction and cleaning. the extraction solvent is n-hexane-dichloromethane mixed solvent (5.6); the extraction temperature is 100 °C; the heating time is 5 min;

The static extraction time was 8 min; the number of cycles was 3; the purge time was 180 s; the rinsing volume was 60%. The cleaned PUF is placed in a vacuum drying oven it was heated under vacuum at 50 °C until the solvent was completely evaporated, and then placed in a vacuum drying oven for vacuum storage.

5.15

Nitrogen. purity $\geq 99.999\%$.

5.16

Helium. purity $\geq 99.999\%$.