

ICS 13.040.20

CCS Z 15

HJ

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

HJ 900-2017

**Ambient air. Determination of organochlorine pesticides. Gas
chromatography/mass spectrometry method**

环境空气 有机氯农药的测定 气相色谱-质谱法

(English Translation)

Issued on: December 14, 2017

Implemented on: March 1, 2018

Issued by: Ministry of Ecology and Environment

Table of Contents

1	Scope of application
2	Normative references
3	Method principle
4	Interference and elimination
5	Reagents and materials
6	Instruments and equipment
6.3	Sampling device
7	Samples
7.1	Sample Collection
7.2	Sample preservation
7.3	Sample Preparation
7.4	Preparation of blank samples
7.8	Analysis steps

1 Scope of application

This standard specifies the determination of organochlorine pesticides in ambient air by gas chromatography - mass spectrometry.

This standard applies to ambient air and particulate matter hexachlorobenzene, alpha-BHC, gamma-BHC, beta-BHC, delta-VI Hexachlorocyclohexane, Aldrin, Epichlorohydrin B, gamma-Chlordane, alpha-Chlordane, Endosulfan I, 4,4'-DDE, Dieldrin, Endrin, 4,4'-DDD, endosulfan II, 2,4'-DDT, 4,4'-DDT, endoddehyde, endosulfan sulfate, methoxy DDT, Moxa total of 23 kinds of organochlorine pesticides determination. See Appendix B for details. If verified, this standard also applies to other organic chlorine Determination of pesticides.

When the sampling volume is 350 m³ (standard state), the volume of concentrated volume is 1.0 ml, and the selective ion scan (SIM) Method, the detection limit of 23 organochlorine pesticides in this method was 0.03 ng/m³ ~ 0.07 ng/m³, the lower limit of determination 0.12 ng/m³ ~ 0.28 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 organochlorine pesticides in the ambient air and particulate matter into the filter and polyurethane foam (PUF), extracted with ether-n-hexane mixed solvent, the extract was concentrated, purified, and separated by gas chromatography-mass spectrometry Detection, retention time and characterization of ion abundance ratio qualitative, internal standard method.

4 Interference and elimination

Other organic matter in the sample may interfere with the determination, choose to use sulfuric acid, magnesium silicate column and other purification methods to remove interference, However, the purification of sulfuric acid can cause the decomposition of some organochlorine pesticides, as detailed in 7.3.3.

5 Reagents and materials

Unless otherwise specified, the analysis of analytical reagents are used in line with national standards of analytical chemistry, experimental water is freshly prepared Pure water.

5.1

acetone (C_3H_6O). pesticide grade.

5.2

n-hexane (C_6H_{14}). pesticide residues.

25.3 Ether ($C_4H_{10}O$). Chromatographic purity.

5.4

Dichloromethane (CH_2Cl_2). pesticide grade.

5.5

Anhydrous sodium sulfate (Na_2SO_4). before use in a muffle furnace 400 °C baking 4 h, after cooling, in a ground glass jar Sealed and saved

5.6

Sodium chloride ($NaCl$). Bake in a muffle furnace at 400 ° C for 4 h before use, then cool and seal in a ground-glass jar Save.

5.7

sulfuric acid (H_2SO_4). $\rho = 1.84 \text{ g/cm}^3$, excellent grade pure.

5.8

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

5.9

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

5.10

ether - n-hexane mixed solvent. 5 5, Pro use now.

5.11

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

5.12

ether - n-hexane mixed solvent. 15 85, Pro use now.

5.13

Sodium chloride solution. $\rho = 50 \text{ g/L}$.

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

5.14

Endrin and 4,4'-DDT standard solution. $\rho = 1.0 \text{ mg/L}$.

Buy a commercially available standard solution directly and dilute with n-hexane (5.2).

5.15

pairs of terphenyl - D14. purity of 98% or more.

Alternatives are analyzed, and other deuterated species or analogues not found in the sample can be used. Can be purchased directly from the market with a permit standard Solution.

5.16

Analysis Alternative stock solution. $\rho = .2000 \text{ }\mu\text{g/ml}$.

Weigh p-terphenyl-D14 (5.15) about 0.1 g, accurate to 0.1 mg, in a 50 ml volumetric flask, with a small amount of dichloromethane After dissolving alkane (5.4), dilute with n-hexane (5.2) and mix well.

5.17

Analysis of intermediate solution. $\rho = 40.0 \text{ }\mu\text{g/ml}$.

Pipette 500 μl of stock solution (5.16) into a 25 ml volumetric flask and dilute with n-hexane (5.2) and mix well.

5.18

Analysis Alternatives Solution. $\rho = 2.00 \mu\text{g/ml}$.

Pipette 100 μl of stock solution (5.16) into 100 ml volumetric flask, dilute to volume with n-hexane (5.2), mix uniform.

5.19

Endosulfan I-D4 or 4,4'-DDT-D8 stock solution. $\rho = 100 \mu\text{g/ml}$.

Sampling alternatives, other isotopic markers may also be used. Direct purchase of certified public standard solution, the solvent is isooctane.

5.20

Sample Alternatives Intermediate solution. $\rho = 20.0 \mu\text{g/ml}$.

Take 1.00 ml aliquots of stock solution (5.19) in a 5 ml volumetric flask and dilute to volume with n-hexane (5.2) and mix well.

5.21

sampling alternative solution. $\rho = 2.00 \mu\text{g/ml}$.

Take 1.00 ml of sample substitute intermediate solution (5.20) in a 10 ml volumetric flask, dilute with n-hexane (5.2) and mix well.

5.22

Internal standard stock solution. $\rho = 750 \mu\text{g/ml}$.

Direct purchase of a certified public standard solution, with Philippine-D10, -D12 or other deuterium-substituted PAHs.

5.23

Internal standard intermediate solution. $\rho = 75.0 \mu\text{g/ml}$.

Take 1.00 ml internal standard stock solution (5.22) in a 10 ml volumetric flask, dilute with n-hexane (5.2) and mix well.

5.24

Internal standard solution. $\rho = 15.0 \mu\text{g/ml}$.

3 Take 2.00 ml internal standard solution (5.23) in a 10 ml volumetric flask and dilute with n-hexane (5.2) and mix well.

5.25

standard stock solution. $\rho = 2000 \mu\text{g/ml}$.

Direct purchase of the market a certified standard solution, including alpha-BHC, gamma-BHC, beta-BHC, delta-BHC, heptachlor, Aldrin, epichlorohydrin B,