

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

HJ 852-2017

**Ambient air - Determination of indicative toxaphene - Gas
chromatography mass spectrometry**

环境空气 指示性毒杀芬的测定 气相色谱-质谱法

(English Translation)

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Foreword

In order to implement the Environmental Protection Law of the People's Republic of China and the Law of the People's Republic of China on the Prevention and Control of Atmospheric Pollution, Protection of human health, regulate the ambient air poisoning fenfen determination method, the development of this standard.

This standard specifies the gas chromatography-mass spectrometry for the determination of three indicative toxaphene in ambient air.

This standard is the first release.

Appendix A and Appendix B of this standard are informative.

This standard is organized by the Environmental Monitoring Department of the Ministry of Environmental Protection and the Department of Science and Technology Standards.

The main drafting unit of this standard. Qingdao Environmental Monitoring Center Station.

The standard verification unit. the National Environmental Analysis and Testing Center, the Ministry of Agriculture Agricultural Product Quality Supervision, Inspection and Testing Center (Green Island), Shandong Entry-Exit Inspection and Quarantine Bureau Inspection and Quarantine Technology Center, Chinese Academy of Sciences Qingdao Bio-Energy

and Process Research Institute, Qingdao sea Ocean Geology Institute and Qingdao University of Technology.

The environmental protection department of this standard approved on August 28, 2017.

This standard has been implemented since November 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Determination of Ambient Air Indicative Toxaphene by Gas Chromatography - Mass Spectrometry WARNING. The organic solvents and standard solutions used in the experiments are toxic substances. The preparation of the solution and the preparation of the sample should Fume hood, the operation should be required to wear protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the gas chromatography-mass spectrometry for the determination of three indicative toxaphene in ambient air.

This standard is applicable to the determination of three indicative toxaphenes (P26, P50, P62) in ambient air.

When the extraction capacity of 500 m³ (standard state), the three kinds of indicative toxaphene P26, P50, P62 method detection limit Do not 4 pg/m³, 4 pg/m³, 8 pg/m³, the lower limit of determination is 16 pg/m³, 16 pg/m³, 32 pg/m³.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Those who do not specify the date of the reference file, the effective version of the appropriate for this standard.

Technical requirements and testing methods for HJ 93 ambient air particulate matter (PM₁₀ and PM_{2.5}) samplers Technical Guidelines for Sampling of Semi - Volatile Organic Compounds for Environmental Air

3 Principle of the method

The poisonous fenfen in ambient air was collected on a quartz fiber filter and a polyurethane foam (PUF) with a large flow sampler, with the appropriate organic solvent extraction, the extract was concentrated, the multi-layer acidic silica gel column and activated silica gel column purification, with gas chromatography Separation of negative chemical source mass spectrometry for detection. The retention time and the characteristic ion abundance ratio were qualitative and the internal standard method was quantified.

4 Reagents and materials

4.1

concentrated sulfuric acid. ρ (H_2SO_4) = 1.84 g/ml, excellent grade pure.

4.2

anhydrous sodium sulfate (Na_2SO_4). analytical grade.

Before use in the muffle furnace at 450 °C for 2 h, after cooling, stored in the grinding glass bottle in the dryer sealed.

4.3

Acetone ($\text{C}_3\text{H}_6\text{O}$). pesticide residues.

4.4

ether ($\text{C}_2\text{H}_6\text{O}$). pesticide residues.

4.5

n-Hexane (C_6H_{14}). Pesticide level.

4.6

Toluene (C_7H_8). Pesticide level.

4.7

Extract. A mixed solution of ether (4.4) and n-hexane (4.5), 5 95.

4.8

Eluent. A mixed solution of n-hexane (4.5) and toluene (4.6), 6535.

4.9

toxaphene standard solution. ρ = 5.0 $\mu\text{g/ml}$, the solvent is isooctane.

Containing the standard solution of poisonous fenfen P26, P50, P62, the direct purchase of certified standard solution.

4.10

Substitutes Standard solution. ρ = 1.0 $\mu\text{g/ml}$, solvent is nonane.

With $^{13}\text{C}_{10}$ -gamma-chlordane as a substitute, the direct purchase of certified standard solution.

4.11

standard stock solution. ρ = 100 $\mu\text{g/ml}$, the solvent is methanol/toluene, 97 3.

With octachlorobenzene (OCN) as the internal standard, direct purchase of certified standard solution.

4.12

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

2 Standardize the internal standard stock solution (4.11) with n-hexane (4.5) to $10.0 \mu\text{g/ml}$.

4.13

Silica gel. for chromatography, 178-150 μm (80-100 mesh).

4.14

Activated silica gel. Silicone gel (4.13) was activated at 180°C for 6 h, cooled in a desiccator, stored in a clean grinding the glass bottle is sealed and stored.

4.15 22% Acid Silica Gel. Activated Silica Gel (4.14) 78 g Sulfuric acid (4.1) 22 g was added dropwise, Stored in a clean grinding glass bottle sealed.

4.16 44% Acid Silica Gel. Activated Silica Gel (4.14) 56 g After adding concentrated sulfuric acid (4.1) 44 g, Stored in a clean wide-mouth glass bottle sealed to save.

4.17

quartz fiber filter. before use in the muffle furnace at 450°C for 4 h, after cooling with aluminum foil wrapped, stored in the filter box, Ensure that the filter is not contaminated before and after sampling and is in flattened before sampling.

The retention efficiency of $0.3 \mu\text{m}$ standard particles was not less than 99%, and when the air velocity was 0.45 m/s , Force is not greater than 3.5 kPa , in this air velocity, the extraction of high efficiency filter purification of air 5 h, per square centimeter filter loss Weighs no more than 0.012 mg .

4.18

Polyurethane foam (PUF). Polyether type, density 0.022 g/cm^3 , cut into cylindrical (diameter according to the sampling head Glass sampling tube specifications to determine).

Before use, boil the hot water with hot, and then placed in warm water repeatedly scrub, dry with acetone (4.3) Soxhlet extraction 16 h, and then extracted with ether/n-hexane (4.7) Soxhlet for 16 h, wrapped in aluminum foil washed with n-hexane (4.5), stored in Dryer is saved. You can also purchase commercially available purified PUF.

4.19

Carrier gas. helium, purity 99.999%.

4.20

Reaction gas. methane gas, purity 99.999%.

Note 1: All of the above standard solutions are kept at or below 4°C or stored in the manufacturer's product description preservation method. Should be restored before use to room temperature, mix well.