

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 768-2015

**Solid waste. Determination of organic phosphorous pesticides.
Gas chromatography**

固体废物 有机磷农药的测定 气相色谱法

(English Translation)

Issued on: November 20, 2015

Implemented on: December 15, 2015

Issued by: Ministry of Ecology and Environment

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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 Environmental Pollution by Solid Wastes, Protection of human health, regulate solid waste and its leaching solution of organophosphorus pesticide determination method, the development of this standard.

This standard specifies the determination of solid waste and its leaching solution of organophosphorus pesticide gas chromatography.

This standard is the first release.

Appendix A and B of this standard are normative appendices, Appendix C, Appendix D are informative appendices.

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

The main drafting unit of this standard. Shanghai Environmental Monitoring Center.

The standard verification unit. Suzhou City Environmental Monitoring Center Station, Shanghai Huangpu District Environmental Monitoring Station, Shanghai Textile Energy Conservation and Environmental Protection Center, on City Putuo District Environmental Monitoring Station, Shanghai Qingpu District Environmental Monitoring Station, Shanghai Songjiang District Environmental Monitoring Station.

The Environmental Protection Department of this standard approves on November 20, 2015.

This standard is implemented as of December 15, 2015.

This standard is explained by the Ministry of Environmental Protection.

Solid waste - Determination of organophosphorus pesticides - Gas chromatographic method Warning. The organic solvent used in the test, the standard substances are toxic substances, the solution preparation, pre-treatment process should be in the fume hood to carry out the operation; according to the provisions of the requirements of wearing protective equipment, to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the determination of solid waste and its leaching solution of organophosphorus pesticide gas chromatography.

This standard is applicable to the determination of 12 organophosphorus pesticides in solid waste and its leachate, including propenbromide, phorate, dimethoate, diazepine Agriculture, B, mixed with phosphorus, different rice blast net, methyl parathion, malathion, chlorpyrifos, parathion, rice feng and ethionin. If verified, this standard can also be applied to the determination of other organophosphorus pesticides.

Determination of solid waste, when the sampling volume of 10.0 g, 12 kinds of organophosphorus pesticide method detection limit of 0.6 µg/kg ~ 1.2 µg/kg, under the determination of Limited to 2.4 µg/kg to 4.8 µg/kg. Determination of solid waste leachate, when the sampling volume of 100 ml, 12 kinds of organophosphorus pesticide method detection Limited to 0.2 µg/L to 0.3 µg/L, the determination of the lower limit of 0.8 µg/L ~ 1.2 µg/L. See Appendix A. for details.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard.

Technical specification for SJ sample preparation for industrial solid waste Technical Specification for Hazardous Waste Identification HJ/T 299 Solid waste leaching toxicity

leaching method sulfuric acid nitric acid method HJ/T 300 solid waste leaching toxicity
leaching method acetic acid buffer solution method

3 Principle of the method

Solid waste or solid waste leaching solution of organic phosphorus pesticides extracted by organic solvents, the extract was concentrated by volume and then by gas chromatography from the flame photometric detector to determine the time to qualitative, external standard method.

4 Interference and elimination

When the extract contains interferences, the appropriate cleaning method can be used to remove the interference, recommended purification method see Appendix B. Can also be used Auxiliary column or gas chromatography-mass spectrometry (GC/MS) is further characterized. The characteristics of the auxiliary characterization column and the chromatographic reference are described in Appendix C.

5 Reagents and materials

Unless otherwise stated, analytical reagents conforming to the relevant standards are used for the analysis, and the test water is a new preparation for the absence of the target Sub-water.

5.1

n-hexane (C₆H₁₄). chromatographic purity.

5.2

Acetone (CH₃COCH₃). Chromatographic pure.

5.3

dichloromethane (CH₂Cl₂). chromatographic purity.

5.4

Sodium chloride (NaCl).

5.5

n-Hexane - Acetone Mixed Solvent. 9 1 (v/v).

Mixed with n-hexane (5.1) and acetone (5.2) at a volume ratio of 9. 1.

5.6

Standard stock solution. rho =.200 mg/L.

Can be directly purchased commercially available standard solution; can also be prepared

with standard materials, preparation of the solvent is n-hexane (5.1). Including propyl bromide, Phorate, dimethoate, dibenzoyl, phiophosphate, different rice blast net, methyl parathion, malathion, chlorpyrifos, parathion, rice feng and ethidium phosphorus.

5.7

standard use of liquid. $\rho = 5.00 \text{ mg/L}$.

Add 3 900 μl of n-hexane (5.1) to a 4 ml brown bottle and add 100 μl of standard stock solution (5.6) to mix.

5.8

anhydrous sodium sulfate (Na_2SO_4). baked at 400°C for 4 h, placed in a dryer to cool to room temperature, transferred to the grinding glass bottle, in the dryer to save.

5.9

Diatomite. 100 mesh to 400 mesh, baked at 400°C for 4 h, placed in a dryer to cool to room temperature, transferred to the grinding glass bottle, in the Dryer in the preservation.

5.10

Quartz sand. 20 mesh to 50 mesh, baked at 400°C for 4 h, placed in a dryer to cool to room temperature, transferred to the grinding glass bottle, in the dry Cooler to save.

5.11

filter paper. the speed of qualitative filter paper.

5.12

paper sleeve. with Soxhlet extraction device.

5.13

nitrogen (N_2). purity $\geq 99.999\%$.

5.14

Hydrogen (H_2). purity $\geq 99.999\%$.

6 Instruments and equipment

6.1

Gas Chromatograph. with split/splitless inlet with flame photometric detector (FPD).

6.2

Column. 30 m x 0.25 mm x 0.25 μm , ultra low loss (5% phenyl) -methylpolysiloxane capillary column; or 30 m x 0.32 mm x 0.25 μm , (14% cyano-phenyl) -methylpolysiloxane capillary column, or other equivalent capillary column.