

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Water quality Determination of 28 organophosphorus
pesticides Gas chromatography-mass spectrometry**

水质 28种有机磷农药的测定 气相色谱-质谱法

(English Translation)

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1 Scope of application

This standard specifies the gas chromatography-mass spectrometry method for the determination of organophosphorus pesticides in water.

This standard is applicable to dichlorvos, rapid phosphorus elimination, systemic phosphorus absorption, linear phosphorus elimination, Phosphos, Phorate, Terbution, Diazinon, Difenthion, Isobath, Dimethoate, Chlorazophos, Chlorpyrifos Methyl, Phosphatamine, Methyl Parathion, Chlorpyrifos, Phosphos, Malathion, Parathion, Bromthion, Methyl isofosphos, Aminophos, Daofengsan, Profenofos, Determination of 28 kinds of organophosphorus pesticides, such as fenamiphos, triazophos, fenamiphos, and trichlorfon.

When the sampling volume of surface water, groundwater and seawater is 1 L and the constant volume is 1.0 ml, the detection limits of 28 organophosphorus pesticides are 0.3 µg/L~0.6 µg/L, the lower limit of determination is 1.2 µg/L~2.4 µg/L; when the sampling volume of domestic sewage and industrial wastewater is 100 ml, the constant volume when the concentration was 1.0 ml, the detection limits of the 28 organophosphorus pesticides

were 4 µg/L~7 µg/L, and the lower determination limits were 16 µg/L~28 µg/L.

Please refer to Appendix A for the detection limit and lower limit of determination of this standard for the determination of target compounds.

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.

GB 17378.3 Marine Monitoring Specification Part 3: Sample Collection, Storage and Transportation HJ/T 91 Technical Specification for Surface Water and Sewage Monitoring HJ 91.1 Technical Specification for Wastewater Monitoring HJ 164 Technical Specification for Groundwater Environmental Monitoring HJ 442.3 Technical Specifications for Environmental Monitoring in Offshore Seas Part III Water Quality Monitoring in Offshore Seas

3 Principles of the method

The organophosphorus pesticides in the water samples were extracted with chloroform, concentrated and fixed to volume, then separated by gas chromatography and detected by mass spectrometry. by retention time and Characteristic ion abundance ratios were used for quantification and internal standard method for quantification. Trichlorfon in water samples was determined after being converted into dichlorvos by alkaline hydrolysis.

4 Reagents and Materials

Unless otherwise stated, analytical reagents that meet national standards were used in the analysis, and the experimental water was newly prepared pure water without target substances.

4.1

Trichloromethane (CHCl₃). chromatographically pure.

4.2

Acetone (CH₃COCH₃). chromatographically pure.

4.3

n-hexane (C₆H₁₄). chromatographically pure.

4.4

Concentrated sulfuric acid (H₂SO₄). =1.84 g/ml.

4.5

Sodium Hydroxide (NaOH).

4.6

Sodium chloride (NaCl). After burning at 450 °C for 4 h, put it in a desiccator to cool to room temperature, then put it in a reagent bottle and seal it for storage.

4.7

Anhydrous sodium sulfate (Na₂SO₄). calcined at 450 °C for 4 h, placed in a desiccator and cooled to room temperature, then placed in a reagent bottle and sealed for storage.

4.8

Sulfuric acid solution. phi=50%.

Mix concentrated sulfuric acid (4.4) and water in a volume ratio of 1.1.

4.9

Sulfuric acid solution. phi=9.1%.

Mix concentrated sulfuric acid (4.4) and water in a volume ratio of 1.10.

4.10

Acetone-n-hexane mixed solution. phi=50%.

Mix acetone (4.2) and n-hexane (4.3) in a 1.1 volume ratio.

4.11

Sodium hydroxide solution. (NaOH)=10 g/L.

Weigh 1.0 g of sodium hydroxide (4.5), dissolve it in 100 ml of water, mix well, and store it in a plastic reagent bottle with a screw mouth.

4.12

Sodium hydroxide solution. (NaOH) =1.0 g/L.

Weigh 1.0 g of sodium hydroxide (4.5), dissolve it in 1 L of water, mix well, and store it in a plastic reagent bottle with a screw mouth.

4.13

Organophosphorus pesticide standard stock solution (without trichlorfon). =2000 g/ml.

Purchase commercially available certified standard solutions directly, or prepare them in acetone (4.2). See Appendix A for the components of organophosphorus pesticides (excluding trichlorfon insect). Commercially available certified reference materials should be

stored in accordance with the instructions; self-prepared standard stock solutions should be stored frozen at -18 °C, which can be stable for 6 months.

Note: The sum of the concentrations of (E)-Furfenac and (Z)-Framfenox is 2000 g/ml; the sum of the concentrations of Systemic Phosphorus-S and Systemic Phosphorus-O is 2000 g/ml; (E)-the sum of the concentrations of phosphatamine and (Z)-phosphatamine is 2000 g/ml.

4.14

Standard stock solution of trichlorfon. =2000 g/ml.

Purchase commercially available certified standard solutions directly, or prepare them in acetone (4.2). Commercially available certified reference materials are prepared according to the instructions the standard stock solution prepared by yourself can be stored at -18 °C, and it can be stored stably for 6 months.

Note: Trichlorfon standard stock solution is only used when spiking samples.

4.15

Organophosphorus pesticide standard solution (without trichlorfon). =50.0 g/ml.

Accurately pipette 25 µl of the organophosphorus pesticide standard stock solution (4.13) into 975 µl of the acetone-n-hexane mixed solution (4.10).

match.

4.16

Internal standard stock solution. =2000 g/ml.

Naphthalene-d8, acenaphthene-d10, phenanthrene-d10 and Qi-d12 should be selected as internal standards. Commercially available certified standard solutions can be purchased directly, or prepared by themselves. Commercially available the certified reference materials are stored in accordance with the requirements of the instructions; the internal standard stock solution prepared by yourself is frozen and stored at -18 °C, and 12 samples can be stored stably.

moon.

4.17

Substitute standard stock solution. =1000 g/ml.

It is advisable to use tributyl phosphate-d27 as a substitute. Commercially available certified standard solutions can be purchased directly, or prepared by themselves. Commercially available certified standards the quality of the product is stored in accordance with the requirements of the instructions; the standard stock solution of the self-prepared substitute is stored frozen at -18 °C, and it can be stored stably for 6 months.