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HJ

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

HJ 1070-2019

**Water quality--Determination of 15 chlorinated herbicides--Gas
chromatography**

水质 15种氯代除草剂的测定 气相色谱法

(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 Water Pollution Control", protect the ecology Environment, protect human health, standardize the determination method of 15 chlorinated herbicides in water, and formulate this standard.

This standard specifies the determination of 15 kinds of chlorinated herbicides in surface water, groundwater, seawater, domestic sewage and industrial wastewater.

Phase chromatography.

Appendix A of this standard is a normative appendix, and Appendix B is an informative appendix.

This standard is issued for the first time.

This standard is formulated by the Department of Eco-Environmental Monitoring, Laws and Standards Department of the Ministry of Ecology and Environment.

This standard was drafted. Nanjing Institute of Environmental Sciences, Ministry of Ecology and Environment.

Verification units of this standard. Jiangsu Environmental Monitoring Center, Shanghai Environmental Monitoring Center, Nanjing Environmental Monitoring Center, Jiangsu Province Xin, Changzhou Environmental Monitoring Center of Jiangsu Province, Jiangyin Environmental Monitoring Center of Jiangsu Province and Zhangjiagang Environmental Monitoring Center of Jiangsu Province.

This standard was approved by the Ministry of Ecology and Environment on December 31, 2019.

This standard will be implemented from June 30, 2020.

This standard is explained by the Ministry of Ecology and Environment.

Water quality-Determination of 15 chlorinated herbicides by gas chromatography Warning. The reagents and standard solutions used in the experiment are corrosive, irritating and toxic. Before reagent preparation and sample preparation the treatment process should be carried out in a fume hood; wear protective equipment as required to avoid breathing into the respiratory tract or touching the skin and Clothing.

1 Scope

This standard specifies gas chromatography for the determination of 15 chlorinated herbicides in water.

This standard applies to 2,2-dichloropropionic acid, 3,5-dichlorobenzoic acid, 2-(4-chloro-2-methylphenoxy) propionic acid, and 3,6-dichloro 2-methoxybenzoic acid, 2-methyl-4-chlorophenoxyacetic acid, 2,4-dipropionic acid, 2,4-dichlorophenoxyacetic acid, 2,4,5-trichlorophenoxyethyl Acid, pentachlorophenol, 2,4,5-lanthionic acid, 3-amino-2,5-dichlorobenzoic acid, 2,4-dichlorophenoxybutyric acid, 4-amino-3,5,6- three Determination of 15 kinds of chlorinated herbicides of clopidic acid, triflufenacil and tetrachloroterephthalic acid.

The pretreatment methods of this standard are divided into liquid-liquid extraction and solid-phase extraction. Liquid-liquid extraction is suitable for surface water, groundwater, Domestic sewage, industrial wastewater and seawater; solid phase extraction is suitable for cleaner surface water and groundwater.

When the sampling volume is 500 ml, the constant volume is 10 ml, and the injection volume is 1.0 μ l, the method detection limit is 0.1 ~ 0.2 μ g/L, the lower limit of determination is 0.4 ~ 0.8 μ g/L, see Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses therein. For undated references, the valid version applies to this standard.

GB 17378.3 Marine Monitoring Code Part 3. Sample Collection, Storage and Transportation
HJ 91.1 Technical Specifications for Sewage Monitoring HJ 442 Specifications for
Environmental Monitoring in Coastal Waters HJ/T 91 Technical specifications for surface
water and sewage monitoring

3 HJ/T 164 Groundwater Environmental Monitoring Technical Specification Method Principle

The sample is hydrolyzed under alkaline conditions ($\text{pH} \geq 12$), and then at $\text{pH} \leq 2$ using dichloromethane or a solid-phase extraction column Chlorinated herbicides were extracted from the sample. After the extract was concentrated and the solvent was converted, it was derivatized with pentafluorobenzyl bromide.

It was then separated by gas chromatography and detected by an electron capture detector. Qualitative according to retention time, quantified by external standard method.

4 Reagents and materials

Unless otherwise stated, analytical reagents that meet national standards are used in the analysis. The test water is free of target compounds.

Pure water.

4.1

Phosphoric acid. $\rho(\text{H}_3\text{PO}_4) = 1.69 \text{ g/ml}$, excellent purity.

4.2

Hydrochloric acid. $\rho(\text{HCl}) = 1.19 \text{ g/ml}$.

4.3

Anhydrous sodium sulfate (Na_2SO_4).

Burn at 400°C for 2 h before use. After cooling, place it in a ground glass bottle and store in a desiccator.

4.4

Sodium chloride (NaCl).

Burn at 400°C for 2 h before use. After cooling, place it in a ground glass bottle and store in a desiccator.

4.5

n-hexane (C_6H_{14}). pesticide residue grade.

4.6

Dichloromethane (CH_2Cl_2). pesticide residue level.

4.7

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

4.8

Methanol (CH_4O). pesticide residue level.

4.9

Toluene (C_7H_8). pesticide residue grade.

4.10

Toluene-n-hexane mixed solvent. 16.

4.11

N-hexane-toluene mixed solvent. 1 9.

4.12

Sodium hydroxide solution. $c(\text{NaOH}) = 6 \text{ mol/L}$.

Weigh 24.0 g of sodium hydroxide in water and dilute to 100 ml.

4.13

Hydrochloric acid solution. 1 1.

4.14

Potassium carbonate solution. $\rho(\text{K}_2\text{CO}_3) = 100 \text{ g/L}$.

Weigh 10.0 g of potassium carbonate in water and make up to 100 ml.

4.15

Pentafluorobenzyl bromide ($\text{C}_7\text{H}_2\text{BrF}_5$).

4.16

Pentafluorobenzyl bromide solution. $\rho(\text{C}_7\text{H}_2\text{BrF}_5) = 30 \text{ g/L}$.

Weigh 3.0 g of pentafluorobenzyl bromide (4.15) in acetone and make up to 100 ml.

4.17 Standard stock solution of chlorinated herbicides. $\rho = 100 \text{ mg/L}$

Commercially available certified standard solutions can be purchased directly. The standard solution after opening is sealed and stored under frozen and protected from light, effective 1 year. Alternatively, the standard chlorinated herbicide can be placed in a desiccator containing silica gel for 48 h, then weighed with acetone (4.7) Formulated for solvents.