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HJ

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

HJ 1074-2019

**Water quality--Determination of four organic tin compounds
including tributyltin--Liquid chromatography/inductively
coupled plasma mass spectrometry**

水质 三丁基锡等4种有机锡化合物的测定 液相色谱-电感耦合等离子体质谱法
(English Translation)

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Foreword

In order to implement the "Environmental Protection Law of the People's Republic of China" Republic of the Marine Environmental Protection Law, protects the ecological environment, protects human health, and regulates four kinds of organotinization such as tributyltin in water Method for determination of compounds, formulated this standard.

This standard specifies four kinds of organotinization such as tributyltin in surface water, groundwater, seawater, domestic sewage and industrial wastewater.

Chromatography-inductively coupled plasma mass spectrometry.

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. Dalian Ecological Environment Monitoring Center, Liaoning Province.

Verification units of this standard. National Environmental Analysis and Testing Center, National Marine Environmental Monitoring Center, Shenyang Ecological Environment, Liaoning Province Environmental Monitoring Center, Fushun Environmental Monitoring Center Station, Dalian Food Inspection Institute and Dalian Product Quality Testing Research Institute.

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 4 organotin compounds including tributyltin Liquid chromatography-inductively coupled plasma mass spectrometry Warning. The reagents and standard solutions used in this method are corrosive and toxic. The reagent preparation and sample preparation process it should be carried out in a fume hood, and protective equipment should be worn during operation to avoid contact with skin and clothing.

1 Scope

This standard specifies the liquid chromatography-inductively coupled plasma quality for the determination of four organotin compounds such as tributyltin in water.

Spectrum method.

This standard applies to dibutyltin, tributyltin, dibutyltin in surface water, groundwater, seawater, domestic sewage and industrial wastewater.

Determination of phenyltin and triphenyltin.

When the liquid-liquid extraction method is used, the sampling volume is 1000 ml, the concentration volume is 1.0 ml, and the injection volume is 20.0 μ l, the method the detection limit is 0.004 μ g/L to 0.005 μ g/L, and the lower detection limit is 0.016 μ g/L to 0.020 μ g/L. When the direct injection method is used, when the injection volume is 20.0 μ l, the detection limit of the method is 3 μ g/L to 6 μ g/L, and the lower detection limit is 12 μ g/L to 24 μ g/L. See attached Record A.

2 Normative references

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

HJ 91.1 Technical Specifications for Sewage Monitoring HJ 442 Specifications for Environmental Monitoring in Coastal Waters HJ 493 Technical regulations for the storage and management of water samples HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 164 Technical Specifications for Groundwater Environmental

Monitoring

3 Method principle

The organotin compounds in the samples were enriched by liquid-liquid extraction or directly injected, and then separated by liquid chromatography columns, inductively coupled.

Determination by plasma mass spectrometer. Qualitative according to retention time, quantified by external standard method.

4 Reagents and materials

Unless otherwise specified, analytical reagents that comply with national standards are used in the analysis. The experimental water is freshly prepared pure water or Distilled water.

4.1

Acetonitrile (CH_3CN). pure liquid chromatography.

4.2

Acetic acid (CH_3COOH). excellent grade pure.

4.3

Triethylamine ($\text{C}_6\text{H}_{15}\text{N}$). Excellent grade pure.

4.4

Methanol (CH_3OH). pure by liquid chromatography.

4.5

Acetone (CH_3COCH_3). pure liquid chromatography.

4.6

Dichloromethane (CH_2Cl_2). pesticide residue level.

4.7

Sodium chloride (NaCl). excellent grade pure.

Burn at 400°C for 2 h, and store in a desiccator after cooling.

4.8

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

4.9

Hydrochloric acid solution. 1 1.

Measure 50.0 ml of hydrochloric acid (4.8) and dilute to 100 ml with experimental water.

4.10

Hydrochloric acid solution. 1 19.

Measure 50.0 ml of hydrochloric acid (4.8) and dilute to 1000 ml with experimental water.

4.11

Hydrochloric acid solution. 1 99.

Measure 1.0 ml of hydrochloric acid (4.8) and dilute to 100 ml with experimental water.

4.12

anhydrous sodium sulfate (Na_2SO_4).

Burn at 400 ° C for 4 h, and store in a desiccator after cooling.

4.13

Organic tin (dibutyltin, tributyltin, diphenyltin, and triphenyltin) standard stock solution. $\rho = 1000 \text{ mg/L}$.

Weigh accurately 13.05 mg (accurate to 0.01 mg) of dibutyltin chloride ($\text{C}_8\text{H}_{18}\text{Cl}_2\text{Sn}$) and 11.22 mg of tributyl Tin chloride ($\text{C}_{12}\text{H}_{27}\text{ClSn}$), 12.60 mg of diphenyltin chloride ($\text{C}_{12}\text{H}_{10}\text{Cl}_2\text{Sn}$), 11.01 mg of triphenyltin chloride ($\text{C}_{18}\text{H}_{15}\text{ClSn}$) standard material, dissolved in methanol (4.4) or acetone (4.5), and made up to 10.00 ml. Stock solution per liter it contains 1000 mg of dibutyltin, tributyltin, diphenyltin and triphenyltin. Can be stored frozen at -18 ° C or less 1 year. Certified standard solutions can also be purchased directly.

Note: Other organotin standard materials such as organotin acetate can also be purchased to prepare standard stock solutions.

4.14

Standard solution for organotin (dibutyltin, tributyltin, diphenyltin and triphenyltin). $\rho = 10.0 \text{ mg/L}$.

Measure 1.00 ml of organic tin (dibutyltin, tributyltin, diphenyltin, and triphenyltin) standard stock solution (4.13) Make up to 100 ml with acetonitrile (4.1). It can be stored at -18 °C for 20 days.

4.15

Mobile phase.

Measure 65.0 ml of acetonitrile (4.1), 12.0 ml of acetic acid (4.2), and 0.05 ml of triethylamine (4.3) in 100 ml.

In a brown glass volumetric flask, make up to 100 ml with experimental water.