

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

HJ 1002-2018

**Water quality - Determination of butyl xanthate - Liquid
chromatography/triple quadrupole tandem mass spectrometry**

水质 丁基黄原酸的测定 液相色谱-三重四极杆串联质谱法

(English Translation)

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Foreword

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 Water Pollution, to protect the ecological environment, to ensure human health, standardize the determination method of butylxanthine in water, and formulate this standard.

This standard specifies liquid chromatography-triple quadrupole tandem mass spectrometry for the determination of butylxanthogen in surface water, groundwater, domestic sewage and industrial wastewater.

Appendix A of this standard is an informative annex.

This standard is the first release.

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

This standard was drafted. Dalian Environmental Monitoring Center.

This standard is verified by. Liaoning Provincial Environmental Monitoring Experimental Center, Chengdu Environmental Monitoring Center Station, Petrochemical Park Monitoring Station, Sichuan Environmental Monitoring Station, Chongqing Ecological Environment Monitoring Center, Liaoning Marine Fisheries Research Institute and Jiangsu Environmental Monitoring Center.

This standard is approved by the Ministry of Ecology and Environment on December 26, 2018.

This standard has been implemented since June 1, 2019.

This standard is explained by the Ministry of Ecology and Environment. 1 Determination of butyl xanthate in water - liquid chromatography - triple quadrupole mass spectrometry
Warning. The acetonitrile and butyl xanthate used in the experiment are toxic and harmful compounds.

Avoid contact with skin and clothing during the experiment. The solution preparation process should be carried out in a fume hood.

1 Scope of application

This standard specifies liquid chromatography-triple quadrupole tandem mass spectrometry for the determination of butyl xanthogen in water.

This standard applies to the determination of butyl xanthogen in surface water, groundwater, domestic sewage and industrial wastewater.

The target of this standard is butyl xanthogen, excluding other xanthogens.

When the injection volume is 10.0 μL , the detection limit of butyl xanthogen measured in this standard is 0.2 $\mu\text{g/L}$, and the lower limit of determination is 0.8 $\mu\text{g/L}$.

2 Normative references

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

HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 164
Technical Specifications for Groundwater Environmental Monitoring

3 Principle of the method

The sample is directly injected after filtration, separated by liquid chromatography column, and examined by mass spectrometry under multiple reaction monitoring (MRM) conditions.

Butyl xanthic acid was measured and quantified by internal standard method according to retention time and characteristic ion characterization.

4 Reagents and materials

Analytically pure reagents in accordance with national standards were used for analysis, unless otherwise stated. The experimental water was pure water containing no target.

4.1

Acetonitrile (CH_3CN). Pure by liquid chromatography.

4.2

Ammonia ($\text{NH}_3 \cdot \text{H}_2\text{O}$). Excellent grade.

4.3

Butyl xanthate. Potassium butyl xanthate ($\text{C}_5\text{H}_9\text{OS}_2\text{K}$) or sodium butylxanthate ($\text{C}_5\text{H}_9\text{OS}_2\text{Na}$), purity $\geq 95\%$.

4.4

Formic acid (HCOOH). Excellent grade.

4.5

Sodium hydroxide (NaOH).

4.6

Sodium hydroxide solution. $\rho(\text{NaOH}) = 0.4 \text{ g/ml}$.

Weigh 4 g of sodium hydroxide (4.5) dissolved in water and dilute to 10 ml.

4.7

Ammonia solution 1. 1 1.

4.8 Ammonia solution 2. 2

Take a certain amount of ammonia (4.2) dissolved in water, adjust the pH ~ 9.5, use within 8 h.

4.9 Formic acid solution. 1 1.

4.10 Standard stock solution of butylxanthogen. $\rho(\text{C}_4\text{H}_9\text{OCSSH}) = 100 \text{ mg/L}$.

Weigh butyl xanthate (0.0330 g potassium butyl xanthate or 0.0302 g butyl xanthate) (4.3), add a small amount Dissolve in water, transfer to a 250 ml brown volumetric flask (5.4), add water to the standard line, and add 3 drops of sodium hydroxide solution (4.6) Shake well, make the pH 9~10, and add water to make up. Transfer the solution to a brown glass reagent bottle, and store the stock solution at 4 ° C or less.

Stored in the dark, it can be stored for 20 days. Commercially available certified standard solutions can also be purchased directly.

4.11 Standard use solution of butyl xanthogen. $\rho(\text{C}_4\text{H}_9\text{OCSSH}) = 1.00 \text{ mg/L}$.

Pipette 1.00 ml of butyl xanthogen standard stock solution (4.10) into a 100 ml brown volumetric flask with ammonia solution 2 (4.8) Constant volume. Available now.

4.12 Internal standard stock solution. $\rho(\text{C}_8\text{H}_6\text{Cl}_2\text{O}_3\text{-}^{13}\text{C}_6) = 100 \text{ mg/L}$.

Directly purchase certified 2,4-dichlorophenoxyacetic acid- $^{13}\text{C}_6$ standard solution. The internal standard stock solution is stored in the refrigerator at 4 ° C or less and stored in the dark.

Can be saved for one year. Or save it by referring to the manufacturer's product description.

4.13 Internal standard use solution. $\rho = 1.0 \text{ mg/L}$.

Dilute the internal standard stock solution (4.12) to 1.0 mg/L with water. Now available.

4.14 Membrane. 0.22 μm pore size, hydrophilic polypropylene, glass fiber, hydrophilic polytetrafluoroethylene or other equivalent materials.

4.15 Nitrogen. Purity $\geq 99.9\%$.

5 Instruments and equipment

5.1

High Performance Liquid Chromatography-Triple Quadrupole Mass Spectrometer. High performance liquid chromatography with gradient elution and mass spectrometer equipped with electrospray ionization source (ESI).