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

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**Water quality. Determination of butyl xanthate. Purge and
trap/gas chromatography-mass spectrometry**

水质 丁基黄原酸的测定 吹扫捕集气相色谱-质谱法

(English Translation)

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Foreword

In order to carry out "Law of the People's Republic of China on Environmental Protection" and "Water Pollution Prevention and Control Law of the People's Republic of China", protect the environment and protect people Body health, regulate the determination of butyl xanthic acid in water, the development of this standard.

This standard specifies the determination of butyl xanthate in water by purge trap/gas chromatography - mass spectrometry.

This standard is released for the first time.

This standard by the Environmental Protection Department of Environmental Monitoring Division and Science and Technology Standards Division to develop.

This standard was drafted. China Environmental Monitoring Station.

This standard verification unit. Beijing Environmental Protection Monitoring Center,

Environmental Monitoring Center of Hebei Province, Zhejiang Province Environmental Monitoring Center, heavy Qing City Ecological Environment Monitoring Center, Ningbo City Environmental Monitoring Center and Yangzhou City Environmental Monitoring Center Station.

This standard MEP approved on December 21, 2017.

This standard since February 1, 2018 implementation.

This standard is interpreted by the MEP.

Water quality - Determination of Butyl Xanthic Acid - Purge and Trap/Gas Chromatography - Mass Spectrometry

1 Scope of application

This standard specifies the determination of butyl xanthate in water by purge trap/gas chromatography - mass spectrometry.

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

This standard does not apply to salts containing more than 25 g/L of high salt industrial wastewater and the presence of dexamethasone (manganese) pesticides or diethyl disulfide Substitution of carbamates (eg copper reagents) and other substances in water samples.

If the water samples were detected butyl xanthic acid, if necessary, can be used to further confirm other analytical methods.

When the sample volume was 5 ml, the detection limit of butyl xanthogen was 0.04 g/L, and the lower limit of determination was 0.16 g/L.

2 Normative references

This standard references the following documents in the terms. For undated references, the effective version applies to this standard.

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

3 Method principle

Butyl xanthogen in water decomposes under acidic conditions to produce carbon disulfide (CS₂), which is indirectly determined by the determination of CS₂ Acid concentration. CS₂ is purged with high-purity helium (or nitrogen) and adsorbed into the trap. The trap is quickly heated and purged with high-purity helium (or nitrogen Gas) backflushing, the CS₂ that is

thermally desorbed is separated by gas chromatography and detected with a mass spectrometer. Through the standard solution with butyl xanthogen the retention time and mass spectra of liquid reaction generated CS₂ were compared qualitatively with internal standard method.

4 Interference and eliminate

The presence of CS₂ in the water sample can interfere with the assay and can be subtracted from the difference between the measured values before and after the addition of the acid.

The presence of oxidizing substances in the water sample will make the determination result is low, may be appropriate to add 5 ~ 20 mg sodium thiosulfate removal.

5 Reagents and materials

Unless otherwise specified, the analysis of the use of analytical standards in line with national standards of chemical reagents, experimental water for the target does not contain pure water.

5.1

methanol (CH₃OH). Chromatography pure.

5.2

concentrated phosphoric acid (H₃PO₄). = 1.69 g/ml.

5.3

Sodium hydroxide (NaOH).

5.4

Sodium thiosulfate (Na₂S₂O₃ · 5H₂O).

25.5 Potassium butylxanthate (C₄H₉OCSSK). Purity ≥95%.

5.6

phosphoric acid solution. 1 6.

5.7

Sodium hydroxide solution. (NaOH) = 400 g/L.

Weigh 10 g of sodium hydroxide (5.3) and dissolve to 25 ml with water.

5.8

Sodium hydroxide solution. (NaOH) = 40 g/L.

The sodium hydroxide solution is diluted with water (5.7).

5.9

Sodium hydroxide solution. (NaOH) = 0.08 g/L.

Take 200 L sodium hydroxide solution (5.7) was added to 1L of water.

5.10

butyl xanthic acid standard stock solution. (C₄H₉OCSSH) = 100 g/ml.

Accurately weigh 0.0330 g potassium butylxanthate (5.5), dissolve in a small amount of water and add 100 L sodium hydroxide solution (5.7), and dilute with water to 250 ml, about 4 °C refrigerated, stable for two weeks.

5.11

butyl xanthic acid standard solution. (C₄H₉OCSSH) = 10.0 g/ml.

With sodium hydroxide solution (5.9) accurate dilution of butyl xanthic acid standard stock solution (5.10), with the current service.

5.12

butyl xanthate standard solution. (C₄H₉OCSSH) = 1.00 g/ml.

With sodium hydroxide solution (5.9) accurate dilution of butyl xanthic acid standard solution (5.11), with the current available.

5.13

Internal standard solution. fluorobenzene, = 100 g/ml, the solvent is methanol, a certified standard solution on the market.

5.14

Internal standard solution. fluorobenzene, = 10.0 g/ml.

Use methanol (5.1) to accurately dilute the internal standard solution (5.13).

5.15

Helium. Purity ≥99.999%.

5.16

Nitrogen. Purity ≥99.999%.

6 Instruments and equipment

6.1

Gas Chromatography - Mass Spectrometer. The gas chromatograph section has a split/splitless inlet that can be programmed to temperature and the mass spectrometer EI source.