

CCS Z 16

HJ

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

HJ 977-2018

**Water quality - Determination of alkylmercury - Purge and Trap /
gas chromatography cold vapor atomic fluorescence
spectrometry**

水质 烷基汞的测定吹扫捕集气相色谱-冷原子荧光光谱法

(English Translation)

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Foreword

Protecting the ecology 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 Prevention the environment, the human health, and the method for determining the determination of alkyl mercury in water, and the development of this standard.

This standard specifies the extraction and capture of alkyl mercury in surface water, groundwater, domestic sewage, industrial wastewater and seawater.

Gas chromatography - cold atomic fluorescence spectrometry.

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, the Department of Regulation and Standards of the Ministry of Ecology and Environment.

This standard was drafted. South China Environmental Science Research Institute of the Ministry of Environmental Protection, Qingyuan Environmental Monitoring Station and Lianzhou Environmental Supervision Station.

The standard verification unit. China Academy of Environmental Sciences, Guangdong Environmental Monitoring Center, Sichuan Environmental Monitoring Station, Shenzhen Environmental Monitoring Center Station, Jiangmen Environmental Monitoring Center Station and Shaoguan Environmental Monitoring Center Station.

This standard is approved by the Ministry of Ecology and Environment on November 13, 2018.

This standard has been implemented since March 1, 2019.

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

1 Determination of water quality alkyl mercury - Purge and trap/gas chromatography - cold atomic fluorescence spectrometry Warning. Derivatization reagents and reference materials used in the experiments are toxic compounds, reagent preparation and sample preparation the process should be carried out in a fume hood; protective equipment should be worn as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the purge and trap/gas chromatography-cold atomic fluorescence spectrometry for the determination of alkylmercury in water.

This standard applies to surface water, groundwater, domestic sewage, industrial wastewater and alkyl mercaptan (methylmercury, ethylmercury) in seawater.

Determination.

When the sampling volume is 45 ml, the detection limits of methylmercury and ethylmercury are both 0.02 ng/L, and the lower limit of determination is 0.08 ng/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 442 Code for Environmental Monitoring of Coastal Waters HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring

3 Principle of the method

After the sample is distilled, the alkyl mercury in the distillate is derivatized with sodium tetrapropylborate to form volatile methylpropylmercury and B.

Mercaptopropylmeride, after being separated by purging, trapping, thermal desorption and gas chromatography, pyrolyzed into mercury vapor and measured by cold atomic fluorescence Mercury meter detection. According to the retention time qualitative, the external standard method is quantitative.

4 Interference and elimination

4.1

Sulfide, water-soluble organic matter (DOM) and chloride ions have negative interference with the determination of alkyl mercury, steaming the sample Distillation can remove or reduce interference.

4.2

Some small organic molecules in samples (such as oil-contaminated samples) will be adsorbed and adsorbed together with alkyl mercury derivatives.

The trap tube, after heating and desorption, will have a quenching effect on the atomic fluorescence signal, and should be diluted before distillation and analysis.

4.3

When the concentration of Hg_2 in the sample is higher than 440 ng/L, it will cause positive interference to the determination of alkylmercury. It should be diluted and then distilled and divided.

Analysis.

Note: It is not advisable to separate alkyl mercury and Hg_2 by distillation for water bodies with severe mercury contamination.

25 Reagents and materials

Unless otherwise stated, analytically pure reagents that meet national standards are used for analysis, and laboratory water is not included in the target combination.

Pure water of matter.

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

5.2 Nitric acid. $\rho(HNO_3) = 1.42 \text{ g/ml}$, excellent grade pure.

5.3 Glacial acetic acid. $\rho(CH_3COOH) = 1.05 \text{ g/ml}$, excellent grade pure.

5.4 Potassium hydroxide (KOH).

5.5 Sodium acetate (CH_3COONa).

5.6 Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$).

5.7 Sodium tetrapropylborate [$NaB(C_3H_7)_4$]. purity $\geq 98\%$, sealed at room temperature and protected from light.

5.8 Hydrochloric acid solution. 1 1.

5.9 Nitric acid solution. 19.

5.10 Potassium hydroxide solution. $\rho(KOH) = 20 \text{ g/L}$.

Weigh 2.0 g of potassium hydroxide (5.4), dissolve in 100 ml of water, mix and store in a plastic reagent bottle with a screw.

5.11 Sodium tetrapropylborate solution. $\rho[\text{NaB}(\text{C}_3\text{H}_7)_4]=10 \text{ g/L}$.

Weigh 1.0 g of sodium tetrapropylborate (5.7) and dissolve in 100 ml of potassium hydroxide solution (5.10) pre-cooled to $0^\circ\text{C} \sim 4^\circ\text{C}$.

Shake well and quickly dispense into a 1.5 ml screw-top glass bottle with a gasket and freeze at $-18^\circ\text{C} \pm 2^\circ\text{C}$ to save 6 Months. When used, take a small bottle of reagent and use it when the ice in the bottle melts about half. Melted tetrapropylborate solution Unstable, one-time use.

5.12 Acetic acid-sodium acetate buffer solution. $c = 4 \text{ mol/L}$, $\text{pH} \sim 6.0$.

Weigh 32.8 g of sodium acetate (5.5), dissolve in 80 ml of water, add 2 ml of glacial acetic acid (5.3), dilute to 100 ml with water.

Stored in plastic reagent bottles, ready for use.

5.13 Saturated copper sulfate solution.

Weigh 50 g of copper sulfate pentahydrate (5.6), dissolve in 100 ml of water, mix and store in plastic reagent bottle.

Take the upper layer solution.

5.14 Standard stock solution of methylmercury. hydrazine (CH_3Hg) = 1.00 mg/L.

Purchase a commercially available certified standard solution with methanol as solvent. After opening, refrigerate, protect from light and seal at 4°C .

Months.

5.15 Ethyl mercury standard stock solution. ($\text{C}_2\text{H}_5\text{Hg}$) = 1.00 mg/L.

Purchase a commercially available certified standard solution with methanol as solvent. After opening, refrigerate, protect from light and seal at 4°C .

Months.

5.16 Mixed standard intermediate solution. = 10.0 $\mu\text{g/L}$.

Transfer 500 μl of methylmercury standard stock solution (5.14) and ethylmercury standard stock solution (5.15) to 50 ml capacity respectively.

Add 250 μl glacial acetic acid (5.3) and 100 μl hydrochloric acid (5.1) to the bottle and dilute to the mark with water. This solution is lined with tape the brown reagent bottle or fluoropolymer bottle of Teflon screw cap can be stored in the dark for less than 4°C for 8 months.