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# HJ

## INDUSTRY STANDARD OF THE

## PEOPLE'S REPUBLIC OF CHINA

# HJ 959-2018

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## Water quality-Determination of tetraethyl lead - Headspace/gas chromatography-mass spectrometry

水质四乙基铅的测定顶空_气相色谱-质谱法

**(English Translation)**

**Issued on: July 29, 2018** **Implemented on: January 1, 2019**

**Issued by: Ministry of Ecology and Environment**

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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 Water Pollution Prevention and Control, to protect the environment, this standard is formulated to ensure human health and to regulate the determination method of tetraethyl lead in water.



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This standard specifies the headspace/gas chromatography for the determination of tetraethyl lead in surface water, groundwater, domestic sewage and industrial wastewater.

Mass spectrometry.

This standard is the first release.

This standard is formulated by the Environmental Monitoring Department and the Science and Technology Standards Department.

This standard was drafted. China Environmental Monitoring Station, Luoyang City Environmental Monitoring Station.

The standard verification unit. Beijing Environmental Protection Monitoring Center, Tianjin Environmental Monitoring Center, Jiangsu Province Environmental Monitoring Heart, Zhejiang Environmental Monitoring Center, Guangxi Zhuang Autonomous Region Environmental Monitoring Center Station, Ningbo Environmental Monitoring Center and Xuzhou City Environmental Monitoring Center Station.

This standard is approved by the Ministry of Ecology and Environment on July 29, 2018.

This standard has been implemented since January 1, 2019.

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

1 Determination of tetraethyl lead in water quality by headspace/gas chromatography-mass spectrometry Warning. The reagents and standard solutions used in this method are volatile toxic compounds. The preparation process should be in the fume hood.

In the middle; protective equipment should be worn as required to avoid contact with skin and clothing.

## 1 Scope of application

This standard specifies the headspace/gas chromatography-mass spectrometry for the determination of tetraethyl lead in water.

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

When the water sample volume is 10.0 ml and the headspace injection volume is 1.0 ml, the detection limit of tetraethyl lead is 0.02 μg/L.

The lower limit of determination is 0.08 μg/L.

## 2 Normative references

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



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

## 3 Principle of the method

Under certain temperature conditions, the tetraethyl lead in the water sample in the headspace bottle volatilizes into the liquid space and reaches the heat in the gas-liquid two phase.

After the mechanical dynamic equilibrium, the tetraethyl lead in the gas phase was separated by gas chromatography and detected by a mass spectrometer. Pass and standard the material retention time was compared with the relative abundance of key ions, and the internal standard method was used for quantification.

## 4 Reagents and materials

Analytically pure reagents in accordance with national standards were used for analysis, unless otherwise stated.

Pure water.

**4.1**
Methanol (CH3OH). chromatographically pure.

**4.2**
Tetraethyl lead ((CH3CH2)4Pb) standard stock solution. rho =.200 mg/L, the solvent is methanol.

Commercially available certified standard solutions are stored in accordance with the instructions.

**4.3**
Tetraethyl lead standard use solution I. rho = 2.00 mg/L.

Prepare with tetraethyl lead standard stock solution (4.2) diluted with methanol (4.1) and transfer to a spiral bottle with Teflon liner in the brown reagent bottle, it can be stored in 0~4C for one month after being stored in the dark.

**4.4**
Tetraethyl lead standard use solution II. rho = 0.20 mg/L.

Prepared by diluting tetraethyl lead intermediate solution (4.3) with methanol (4.1) and transferring to a screw cap with a Teflon liner.

Store in brown reagent bottles at 0~4C for one month.



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**4.5**
Internal standard stock solution. rho=2000 mg/L, the solvent is methanol.

2 1,2-dichlorobenzene-d4 should be selected as the internal standard, and the commercially available certified standard solution can be directly purchased.

**4.6**
Internal standard use solution. rho=2.00 mg/L.

Prepare by diluting the internal standard stock solution (4.5) with methanol (4.1).

**4.7**
Helium, purity >= 99.999%.

## 5 Instruments and equipment

**5.1**
Gas Chromatography-Mass Spectrometer. The chromatographic section has a split/splitless inlet for temperature programming. Mass spectrometry with electron bombardment (EI) ionization source with manual/automatic tuning, data acquisition, quantitative analysis and library search.

**5.2**
Automatic Headspace Sampler. The temperature control range is adjustable from room temperature to 100 °C.

**5.3**
Column. quartz capillary column, 30 m x 0.25 mm, film thickness 0.25 m, stationary phase 5% phenyl 95% A Polysiloxane; or other equivalent capillary column.

**5.4**
Sampling bottle. 40 ml brown screw glass bottle, silicone rubber pad lined with PTFE, or other similar Sampling bottle.

**5.5**
Headspace vial. 22 ml screw or jaw headspace bottle with Teflon silicone rubber gasket.

**5.6**
Microinjectors. 5 µl, 10 µl, 50 µl, 100 µl and 250 µl.

**5.7**
Common instruments and equipment used in general laboratories.



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