

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

HJ 810-2016

**Water quality - Determination of volatile organic compounds -
Headspace / Gas chromatography mass spectrometry**

水质 挥发性有机物的测定 顶空气相色谱-质谱法（发布稿）

(English Translation)

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Foreword

To implement the "Environmental Protection Law of the People's Republic of China" and "Water Pollution Prevention Law of the People's Republic of China" to protect the environment and protect the human body Health, regulate the determination of volatile organic compounds in water, the development of this standard.

This standard specifies headspace/gas chromatography for the determination of volatile organic compounds in surface water, groundwater, domestic sewage, industrial waste water and seawater - mass spectrometry.

This standard is the first release.

Appendix A to this standard is a normative appendix, Appendix B and Appendix C are informative appendices.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting of this standard. Nanjing Environmental Monitoring Center Station.

The standard verification unit. Jiangsu Province Environmental Monitoring Center, Changzhou City Environmental Monitoring Center, Taizhou City Environmental Monitoring Center Station, Zhenjiang City Environmental Monitoring Center Station, Jiangsu Province Physical and Chemical Testing Center and Nanjing Water Group Co., Ltd. Water Quality Monitoring Center.

The environmental protection department of this standard approved on July 26, 2016.

This standard has been implemented since October 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of volatile organic compounds - Headspace/gas chromatography - mass spectrometry Warning. The organic solvents and reference materials used in the experiment are toxic and hazardous substances. The preparation and sample preparation process should be carried out in the fume hood.

Line; operation should be required to wear protective equipment, to avoid direct contact with skin and clothing.

1 Scope of application

This standard specifies the headspace/gas chromatography-mass spectrometry for the determination of volatile organic compounds in water.

This standard applies to the determination of 55 volatile organic compounds in surface water, groundwater, domestic sewage, industrial waste water and seawater.

When the sampling volume was 10.0 ml, the detection limit of the method of the target compound was 2 µg/L to 10 µg/L, the detection limit was 8 µg/L to 40 µg/L. The detection limit of the target compound was 0.4 µg/L ~ 1.7 Mg/L, the lower limit of determination was 1.6 µg/L to 6.8 µg/L. See Appendix A. for details.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. For undated references, the valid version applies to this standard.

Technical specification for surface water and wastewater monitoring Technical specification for groundwater environmental monitoring Specification for ocean monitoring - Part 3. Sample collection, storage and transport

3 Principle of the method

Under certain temperature conditions, the volatile components of the sample in the headspace bottle evaporate to the liquid space, resulting in vapor pressure, in the gas-liquid two-phase to After the dynamic equilibrium of thermodynamics, the volatile organic compounds in the gas phase are separated by gas chromatography and detected by mass spectrometer. By keeping with the reference substance Time and mass spectrum compared to qualitative, internal standard method quantitative.

4 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents are used in accordance with national standards.

4.1

Experimental water. secondary distilled water or water equipment prepared by water.

Before use, subject to a blank test to confirm that there is no interference with the presence or peak of the chromatographic peaks in the retention time interval of the target compound the concentration of the compound is lower than the method detection limit.

Note 1: If the laboratory has a volatile solvent such as dichloromethane, you need to pay special attention to check the quality of experimental water, you can choose to boil, nitrogen purge or Use special volatile organic matter to remove the column, etc., until the requirements are met.

4.2

Methanol (CH₃OH). Chromatographic Purification. Before use, subject to a blank test to confirm that the target compound or target compound concentration is lower than the method the detection limit.

4.3

Sodium chloride (NaCl). Before use, in the muffle furnace at 400 °C for 4 h, placed in a dryer to cool to room temperature, transferred to the grinding glass Preserved in a jar.

4.4

ascorbic acid (C₆H₈O₆).

4.5

hydrochloric acid. ρ (HCl) = 1.19 g/ml, excellent grade pure.

4.6

hydrochloric acid solution. 1 1 (v/v), with hydrochloric acid (4.5) preparation.

4.7

standard stock solution. ρ = 1000 mg/L ~ 5000 mg/L.

Commercially available certified standard solution, according to the instructions required to save.

4.8

standard use of liquid. ρ = 50 mg/L ~.200 mg/L.

The standard stock solution (4.7) was diluted with methanol (4.2). Vinyl chloride standard use of liquid with the current allocation, the remaining standard use of liquid retention period of 30 d.

4.9

internal standard stock solution. ρ = 100 mg/L ~.2000 mg/L.

Should use fluoride benzene, 1,2-dichlorobenzene-d₄ as the internal standard, can be

directly purchased commercially available standard solution. Meet the requirements of this method and do not work. Other internal standards may also be used under the premise of determining the target compound.

4.10

Internal standard liquid I. $\rho = 200 \text{ mg/L}$.

The internal standard stock solution (4.9) was diluted with methanol (4.2).

4.11

Internal standard liquid II. $\rho = 20 \text{ mg/L}$.

The internal standard stock solution (4.9) was diluted with methanol (4.2).

4.12

Mass spectrometry Tuning solution. 4-bromofluorobenzene ($\text{C}_6\text{H}_4\text{BrF}$, BFB), $\rho = 25 \text{ mg/L}$.

Can be directly purchased commercially available standard solution, or with the standard material preparation.

4.13

helium. purity $\geq 99.999\%$.

4.14

Nitrogen. Purity $\geq 99.999\%$.

Note 2: Unless otherwise stated, the solution (4.7 to 4.12) is stored in methanol (4.2) as a solvent and stored in a brown dense flask at temperatures below -10°C .

According to the manufacturer's product description preservation; vinyl chloride stored separately. Before use should be restored to room temperature, mix.

5 Instruments and equipment

5.1

Gas Chromatography/Mass Spectrometer. The gas chromatographic section has a split/splitless inlet and can be programmed to warm up. The mass spectrum portion has 70 eV electrons (EI) ion source with NIST mass spectrometer library with full scan (Scan) and selective ion (SIM) scanning, manual/auto tuning, Data collection, quantitative analysis and library search and other functions.

5.2

automatic headspace sampler. heating temperature control range between room temperature to 120°C ; temperature control accuracy of $\pm 1^\circ \text{C}$.