

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 1227-2021

**Water quality - Emergency determination of volatile organic
compounds - Portable headspace/gas chromatography-mass**

水质 挥发性有机物的应急测定 便携式顶空气相色谱-质谱法

(English Translation)

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1 Scope of application

The standard specifies a portable headspace/gas chromatography-mass spectrometry method for the determination of volatile organic compounds in water.

This standard is applicable to 56 kinds of volatile toluene in surface water, groundwater, domestic sewage, industrial wastewater and seawater in sudden environmental events.

On-site emergency determination of organic matter. This method can also be used for qualitative and concentration estimation of other volatile organic compounds.

In full scan mode, when the sample volume in the headspace vial is 10.0 ml, the gas injection volume is 8 ml, splitless injection, or Method detection of 56 target compounds with a sample volume of 20.0 ml in an empty vial, a gas injection volume of 100 ml, and a split ratio of 40:1 the limit of determination is 1 µg/L~3 µg/L, and the lower limit of

determination is 4 µg/L~12 µg/L, see Appendix A for details.

2 Normative references

This standard refers to the following documents or clauses thereof. For dated references, only the dated version applies to this standard.

For undated references, the latest edition (including all amendments) applies to this standard.

GB/T 32210 Portable gas chromatography-mass spectrometer technical requirements and test methods
HJ 442.9 Technical Specifications for Environmental Monitoring in Offshore Sea Areas Part IX Emergency and Special Monitoring in Offshore Sea Areas
HJ 589 Technical specification for emergency monitoring of environmental emergencies

3 Principles of the method

Under certain temperature conditions, the volatile organic compounds in the sample in the headspace sampling bottle volatilize to the headspace and are carried into the portable by the carrier gas.

It is enriched in the built-in adsorption tube of the gas chromatography-mass spectrometer, enters the gas chromatography for separation after thermal desorption, and is detected by a mass spectrometer detector.

Quasi-substance mass spectrum and retention time comparison qualitative, internal standard method for quantification; for substances other than 56 target compounds, by comparing with standard substances the mass spectrum is qualitative, and its concentration is estimated based on the ratio of its response to that of the internal standard.

4 Reagents and Materials

Unless otherwise specified, analytical reagents that meet national standards were used in the analysis.

4.1

Experimental water. double distilled water or water prepared by pure water equipment.

Before use, a blank test should be carried out to confirm that there is no interference peak within the retention time of the target compound or the concentration of the target compound is lower than the formula.

detection limit.

4.2

Ascorbic acid (C₆H₈O₆).

4.3

Methanol (CH₃OH). pure by liquid chromatography.

4.4

Hydrochloric acid. $\rho=1.19$ g/ml, excellent grade.

4.5

Hydrochloric acid solution.

Hydrochloric acid (4.4) and water are mixed in a volume ratio of 1.1.

4.6

Standard stock solution of volatile organic compounds. $\rho=2000$ mg/L.

Commercially available certified standard solution, stored in accordance with the instructions.

4.7

Standard solution for volatile organic compounds. $\rho=100$ mg/L.

Dilute the standard stock solution of volatile organic compounds (4.6) with methanol (4.3), and store it for 30 d in the refrigerator below 4°C, protected from light and sealed.

4.8

Internal standard stock solution. $\rho=2000$ mg/L.

Fluorobenzene and 1,4-dichlorobenzene-d₄ were selected as internal standards. The certified standard solution can be purchased directly and stored according to the instructions. satisfying this Other internal standards may also be used as long as the method requires and does not interfere with the determination of the target compound.

4.9

Internal standard solution. $\rho=100$ mg/L.

Dilute the internal standard stock solution (4.8) with methanol (4.3), and store it for 30 d if it is refrigerated below 4 °C and protected from light.

4.10

Internal standard gas. the components are 1,3,5-tris(trifluoromethyl)benzene (100 μ mol/mol), bromopentafluorobenzene (50 μ mol/mol) or 4-Bromofluorobenzene (20 μ mol/mol), stored in a steel cylinder, can be stored for 1 year, or refer to the relevant instructions of the standard gas certificate. for instrument tuning Harmony.

4.11

Carrier gas. nitrogen or helium, purity $\geq 99.999\%$.

5 Instruments and equipment

5.1

Sampling bottle. 40 ml amber glass bottle with silicone rubber-teflon gasketed screw cap.

5.2

Portable gas chromatography-mass spectrometer. with adsorption thermal desorption module, gas chromatography module (with temperature program function), and quadrupole Rod or ion trap mass spectrometry modules. The mass spectrometry module has a 70 eV electron impact (EI) ion source with full scan/selected ion scan, auto/Functions such as manual tuning, spectral library search and quantitative analysis should meet the requirements of GB/T 32210. All samples pass through the tubing and all joints should be inertized.

5.3

Portable headspace sampler. The heating temperature is controlled between room temperature and 80 °C, and the temperature control accuracy is ± 1 °C.

5.4

Capillary column. 15 m $\times$ 0.25 mm $\times$ 1.0 m or 10 m $\times$ 0.1 mm $\times$ 0.4 m, the stationary phase is 100% dimethylpolysilicon oxane, other equivalent capillary columns can also be used.

5.5

Built-in adsorption tube. Fill with adsorbents such as Tenax, Carbopack, silica gel, or other equivalent adsorbents.

5.6

Headspace vial. Use the glass headspace vial that is fitted with the Portable Headspace Sampler (5.3), with a Teflon-coated seal.

5.7

Commonly used instruments and equipment.

6 Preparation before monitoring

Check the working status of the portable gas chromatograph-mass spectrometer, and confirm that the carrier gas (4.11), internal standard gas (4.10), Volatile organic compound standard solution (4.7) and internal standard solution (4.9), batteries, sampling bottles (5.1) and personal protective equipment, etc.

Necessary spare parts.