

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Ambient air Emergency determination of volatile organic
compounds - Portable gas chromatography-mass spectrometry**

环境空气 挥发性有机物的应急测定 便携式气相色谱-质谱法

(English Translation)

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1 Scope of application

This standard specifies a portable gas chromatography-mass spectrometry method for the determination of volatile organic compounds in ambient air.

This standard is applicable to the on-site emergency determination of 52 volatile organic compounds such as toluene in the ambient air around the site of an environmental emergency.

This method can also be used for qualitative analysis and concentration estimation of volatile organic compounds.

In full scan mode, when the injection volume is 600 μl , the split ratio is 50:1, or the injection volume is 100 μl , splitless, the method detection limit is 3 $\mu\text{g}/\text{m}^3$ ~9 $\mu\text{g}/\text{m}^3$, and the lower limit of determination is 12 $\mu\text{g}/\text{m}^3$ ~36 $\mu\text{g}/\text{m}^3$, 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 589 Technical specification for emergency monitoring of environmental emergencies
HJ 732 Sampling air bag method for volatile organic compounds in waste gas from stationary pollution sources

3 Principles of the method

The sample is directly introduced or adsorbed by an adsorption tube and thermally desorbed, and then enters into the gas chromatography for separation, and is detected by a mass spectrometer. for 52 species Standard compound, qualitative by comparing with standard substance mass spectrum and retention time, internal standard method quantification; for substances other than 52 target compounds by comparing with the mass spectrum of the standard substance qualitatively, according to the ratio of its response value to the response value of the internal standard, its concentration is estimated.

4 Reagents and Materials

4.1

Standard gas. the concentration is not less than 1 $\mu\text{mol/mol}$, the pressure is not less than 1.0 MPa, stored in a steel cylinder, it can be stored for 1 year, or refer to the standard Instructions for the gas certificate. According to the actual work needs, you can purchase a higher concentration of certified standard gas or customize a suitable mixture in a qualified unit.

standard gas.

4.2

Field standard gas. use the gas diluter (5.7) in the laboratory to dilute the standard gas (4.1) with nitrogen gas (4.4) to 10 nmol/mol.

Field standard gas can be stored in stainless steel tank (5.6) for 20 days and in air bag (5.2) for 8 hours.

Note: The static dilution method (see Appendix B) can also be used to prepare liquid standard substances.

4.3

Internal standard gas. 1,3,5-tris(trifluoromethyl)benzene (100 $\mu\text{mol/mol}$), bromopentafluorobenzene (50 $\mu\text{mol/mol}$) or 4-bromo Fluorobenzene (20 $\mu\text{mol/mol}$), stored in a steel cylinder, can be kept for 1 year, or refer to the relevant instructions of the standard gas certificate. meeting the method requirements and Under the premise of not interfering with the target compound, other substances can also be used as internal standards.

4.4

Diluent gas. nitrogen, purity $\geq 99.999\%$.

4.5

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

5 Instruments and equipment

5.1

Vacuum gas sampling box. It is composed of air inlet pipe, vacuum box, valve and air pump.

The components to be tested react.

5.2

Air bag. The material should meet the relevant requirements of HJ 732.

5.3

Portable gas chromatography-mass spectrometer. gas sampling module with temperature control function, adsorption thermal desorption module, gas chromatography module (with temperature programmed function), and quadrupole or ion trap mass spectrometry modules. The mass spectrometry module features a 70 eV electron impact (EI) ion source with full scan Scanning/selected ion scanning, automatic/manual tuning, spectral library search and quantitative analysis, etc. The performance indicators should meet the requirements of GB/T 32210. Place Pipes and fittings through which the sample passes should be inert.

5.4

Capillary column. 15 $\text{m} \times 0.25 \text{ mm} \times 1.0 \text{ m}$ or 10 $\text{m} \times 0.1 \text{ mm} \times 0.4 \text{ m}$, the stationary phase is 100% dimethylpolysiloxane alkane, 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

Stainless steel tank. The inner wall is inertized, and the pressure resistance value is greater

than 241 kPa.

5.7

Gas diluting instrument. the maximum dilution ratio is at least 1000 times, and the pipelines should be inertized.

5.8

Commonly used instruments and equipment.

6 Preparation before monitoring

Check the working status of the portable gas chromatograph-mass spectrometer, and confirm that the on-site standard gas (4.2) and internal standard that meet the work requirements are carried.

gas (4.3) and carrier gas (4.5); confirm carrying batteries, vacuum gas sampling box (5.1), gas bag (5.2), gas sampling module and personal Protective equipment and other necessary spare parts.

7.1 Sample Collection

7.1.1

The requirements for point layout and on-site monitoring shall be implemented in accordance with the relevant regulations of HJ 589.

7.1.2

Select an appropriate method according to the on-site situation, and use the gas sampling module of the portable gas chromatograph-mass spectrometer (5.3) for direct sampling;

When the field conditions are limited or the actual work requires, the vacuum gas sampling box (5.1) can also be used to collect air bag samples.

7.1.3

After sampling, air bag samples should be transported and stored in dark and normal temperature conditions, and should be analyzed as soon as possible.

7.2 Preparation of blank samples

Fill the pre-cleaned air bag (5.2) with nitrogen gas (4.4).

8.1.1 Adsorption thermal desorption reference conditions

Condition 1. Sampling probe temperature. 40 °C; heating line temperature. 70 °C; injection flow rate. 100 ml/min; adsorption tube desorption temperature.

300 °C; pre-desorption time of adsorption tube. 0.1 min; desorption time of adsorption tube.