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HJ

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

HJ 1078-2019

**Stationary source emission--Determination of 8 organosulfur
compounds including methanethiol--Bag sampling and
preconcentration/gas chromatography mass spectrometry**

固定污染源废气 甲硫醇等8种含硫有机化合物的测定 气袋采样-预浓缩气相色谱-
质谱法

(English Translation)

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Foreword

To implement the Environmental Protection Law of the People 's Republic of China and the Air Pollution Prevention and Control Law of the People 's Republic of this standard is formulated to ensure human health, regulate the determination of sulfur-containing organic compounds in exhaust gas from stationary pollution sources.

This standard stipulates the sampling and pre-concentration of airbags for the determination of 8 sulfur-containing organic compounds such as methyl mercaptan in waste gas from stationary sources.

Gas chromatography-mass spectrometry.

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

This standard is issued for the first time.

This standard is organized and formulated by the Ecological Environment Monitoring Department and the Regulations and Standards Department of the Ministry of Ecology and Environment.

1 Scope of application

This standard stipulates the sampling and pre-concentration of airbags for the determination of 8 sulfur-containing organic compounds such as methyl mercaptan in waste gas from stationary sources.

Gas chromatography-mass spectrometry.

This standard applies to methyl mercaptan, ethyl mercaptan, methyl sulfide, methyl ethyl sulfide, carbon disulfide, ethyl sulfide in waste gas from stationary sources Determination of 8 sulfur-containing organic compounds including ether, dimethyl disulfide and thiophene.

When the sampling volume is 50.0 ml, the detection limit of the method for sulfur-containing organic compounds determined by this standard is 0.01 ~ 0.02 mg/m³, the lower limit of determination is 0.04 to 0.08 mg/m³, see Appendix A for details.

2 Normative references

This standard refers to the following documents or their clauses. For the cited documents without date, the valid version applies to this standard.

GB/T 16157 Sampling methods for particulate matter and gaseous pollutants in exhaust from stationary sources HJ 732 Sampling airbag method for volatile organic compounds in waste gas from stationary sources HJ/T 373 Technical Specifications for Quality Assurance and Quality Control of Fixed Pollution Source Monitoring (Trial) HJ/T 397 Fixed source

exhaust gas monitoring technical specification

3 Method principle

Collect waste gas from stationary pollution sources with an air bag, after cold trap concentration and thermal analysis, enter gas chromatography separation and detect by mass spectrometry Device for detection. Qualitative comparison with the standard substance mass spectrum and retention time, internal standard method for quantification.

4 Interference and elimination

When the concentration of other types of volatile organic compounds affects the measurement of sulfur-containing organic compounds, you can reduce the Release the sample or use a highly selective detector for sulfur compounds for analysis.

5 Reagents and materials

5.1

Standard gas. methyl mercaptan, ethyl mercaptan, methyl sulfide, methyl ethyl sulfide, carbon disulfide, ethyl sulfide, dimethyl disulfide, thiophene the concentration of 2 is 2 $\mu\text{mol/mol}$. High-pressure steel cylinders are stored, the pressure of the steel cylinder is not less than 1.0 MPa, please refer to the standard gas certificate for the storage period
Related instructions.

Note: The concentration of standard gas is for reference only, and other concentration standard gas can be purchased on demand if the method performance parameters are met.

5.2

Standard gas. use a gas diluter (6.7) to dilute the standard gas (5.1) with nitrogen (5.8) to 20 nmol/mol, Stored in the tank (6.6), now ready for use.

5.3

Internal standard gas. the concentration is 1 $\mu\text{mol/mol}$. Internal standard is 1,4-difluorobenzene, stored in high-pressure steel cylinders, the pressure of the steel cylinders is not low at 1.0 MPa, the storage period refers to the relevant description of the standard gas certificate. Before meeting the method requirements without interfering with the target compound Mentioned, other substances can also be used as internal standards.

5.4

Internal standard gas. use gas diluter (6.7) to dilute internal standard gas (5.3) with nitrogen (5.8) to the concentration is 100 nmol/mol and it can be stored in the tank (6.6) for 20 days.

5.5 4-bromofluorobenzene standard gas. the concentration is 1 $\mu\text{mol/mol}$, stored in a high-pressure steel cylinder, the pressure of the cylinder is not less than 1.0 MPa, stored

Please refer to the relevant description of the standard gas certificate.

5.6 4-bromofluorobenzene standard gas. use a gas diluter (6.7) to use 4-bromofluorobenzene standard gas (5.5) with nitrogen (5.8) Dilute to a concentration of 100 nmol/mol and store in a tank (6.6) for 20 days.

5.7

High purity helium. purity $\geq 99.999\%$.

5.8

High purity nitrogen. purity $\geq 99.999\%$.

5.9

Liquid nitrogen.

6 Instruments and equipment

6.1

Sampling device. The requirements of the air bag sampling device shall comply with the relevant regulations of HJ 732.

6.2

Gas Chromatography-Mass Spectrometer. The gas phase has an electronic flow controller, and the column temperature box has a program temperature rise function.

Equipped with column thermostat cooling device. The mass spectrometer part has an electron bombardment (EI) ion source, full scan/selective ion scan, Dynamic/manual tuning, library search and other functions.

6.3

Capillary chromatography column. 60 m x 0.32 mm x 1.04 μm (100% dimethyl polysiloxane fixative), or other Effective capillary chromatography column.

6.4

Gas cold trap concentrator. It has the functions of automatic quantitative sampling and automatic addition of standard gas and internal standard. Should have three levels the cold trap can introduce liquid nitrogen to be cooled to a minimum of $-180\text{ }^{\circ}\text{C}$, complete three-stage trapping and remove interferences such as H_2O and CO_2 . Concentrator the internal pipeline and the pipeline connected with the gas chromatography-mass spectrometer are all made of inert materials, and can be heated in the range of 50 to $150\text{ }^{\circ}\text{C}$.

6.5

Automatic sampler of concentrator. It can carry out automatic sampling of air bag and