

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

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**Ambient air and waste gas - Determination of pyridine - Gas
chromatography method**

环境空气和废气 吡啶的测定 气相色谱法

(English Translation)

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1 Scope of application

This standard specifies a gas chromatography method for the determination of pyridine in ambient air and exhaust gas.

This standard is applicable to the determination of pyridine in ambient air, the air of fugitive emission monitoring points and the organic emission of fixed pollution sources.

When collecting ambient air and fugitive emission monitoring point air, the sampling volume is 30 L, and the constant volume of absorption liquid is 10 ml, the method is checked.

The output limit is 0.02 mg/m³, and the lower limit of determination is 0.08 mg/m³; when collecting the organized waste gas from fixed pollution sources, the sampling volume is 30 L.

The detection limit of the method was 0.09 mg/m³ and the lower limit of determination was 0.36 mg/m³ when the volume of the collected liquid was 50 ml.

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 16157 Sampling method for particulate matter and gaseous pollutants in exhaust gas from stationary pollution sources HJ/T 55 Technical guidelines for monitoring fugitive emissions of air pollutants HJ 194 Technical Specification for Manual Monitoring of Ambient Air Quality HJ/T 397 Technical Specification for Stationary Source Waste Gas Monitoring

3 Principles of the method

The pyridine in ambient air and waste gas is absorbed by the acid absorption liquid, and the absorption liquid is placed in a sealed headspace bottle. Under alkaline conditions, the headspace the pyridine in the sample in the bottle volatilizes into the head space to generate vapor pressure, and the thermodynamic dynamic equilibrium is achieved in the gas-liquid two-phase. Quantitative extraction of gas phase fraction Separation by gas chromatography, hydrogen flame ionization detector detection. Qualitatively based on retention time, quantified by external standard method of working curve.

4 Interference and cancellation

Under the experimental conditions specified in this method, some of the volatile organic compounds in the sample absorbed by the sulfuric acid absorption solution may interfere with the determination.

Interferences were removed by separation using columns of different polarities.

5 Reagents and Materials

Unless otherwise stated, analytical reagents that meet national standards are used in the analysis, and the experimental water is newly prepared water without organic matter.

5.1

Pyridine (C₅H₅N). The purity should be $\geq 99.5\%$, and it should be stored in a refrigerator away from light.

5.2

Sulfuric acid. $\rho = 1.84$ g/ml, excellent grade.

5.3

Sodium hydroxide (NaOH). flake, excellent grade pure.

5.4

Sodium chloride (NaCl). heated at 400 °C for 4 h, and then transferred to a ground glass bottle after being slightly cooled, and stored in a desiccator.

5.5

Sulfuric acid absorption solution. $c(\text{H}_2\text{SO}_4)=0.1 \text{ mol/L}$.

Pipette 5.4 ml of sulfuric acid (5.2) into 1 L of water and mix well. Ready to use.

5.6

Standard stock solution of pyridine. $\rho(\text{C}_5\text{H}_5\text{N})=10.0 \text{ mg/ml}$.

Accurately weigh 0.250 g (accurate to $\pm 0.1 \text{ mg}$) of pyridine (5.1), transfer it to a 25 ml brown volumetric flask with a small amount of water, and determine with water.

Make up to mark, shake well. Refrigerated below 4°C , sealed and protected from light, it can be stored for 2 months. You can also directly purchase certified standard solutions, refer to the standard certificate to save.

5.7

Pyridine standard solution. $\rho(\text{C}_5\text{H}_5\text{N})=1.00 \text{ mg/ml}$.

Accurately pipette 1.00 ml of pyridine standard stock solution (5.6) into a 10 ml brown volumetric flask, dilute with water to the mark, and prepare as pyridine standard solution, refrigerated below 4°C , sealed and protected from light, it can be stored for 7 d.

5.8

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

5.9

Combustion gas. hydrogen, purity $\geq 99.99\%$.

5.10

Supporting gas. air, dehydrated by silica gel, dehydrated by activated carbon.

6 Instruments and equipment

6.1

Air sampler. the sampling flow range is $0.1 \text{ L/min} \sim 1.0 \text{ L/min}$, other performance and technical indicators should comply with the provisions of HJ 194.

6.2

Flue gas sampler. the sampling flow range is $0.1 \text{ L/min} \sim 1.0 \text{ L/min}$, the sampling tube is made of hard glass or fluororesin, and it should have heating and heat preservation function, the heating temperature of the sampling tube is not lower than 120°C , and other performance and technical indicators should comply with the provisions of HJ/T 397.

6.3

Connecting pipe. Teflon hose or silicone rubber pipe lined with Teflon film.

6.4

Cooling device. ice water bath.

6.5

Small multi-well glass plate brown absorption bottle. 25 ml. The absorption bottle should be tight and airtight, the foaming of the porous glass plate absorption bottle should be uniform, and other properties and technical indicators should comply with the provisions of HJ 194.

6.6

Large multiwell glass plate brown absorption bottle. 75 ml or larger volume multiwell glass plate brown absorption bottle. The absorption bottle should be tight and airtight, porous the foaming of the glass plate absorption bottle should be uniform, and other performance and technical indicators should comply with the provisions of HJ/T 397.

6.7

Gas chromatograph. with capillary column split/splitless injection port, with constant flow or constant pressure function, programmable temperature, with hydrogen flame ionization Chemical Detector (FID) and Workstation.

6.8

Chromatographic column. quartz capillary column, the stationary phase is cross-linked polyethylene glycol, 30 m x 0.25 mm x 0.5 m; or other equivalent capillary column.

6.9

Automatic headspace sampler. temperature control accuracy is ± 1 °C, with headspace vial, sealing gasket (teflon/siloxane or teflon) /butyl rubber), caps (screw caps or disposable glands).

6.10

Analytical balance. the actual division value is 0.1 mg.

6.11

General laboratory instruments and equipment.

7.1.1 Ambient air and fugitive emission monitoring point air samples

The air sampling of ambient air and fugitive emission monitoring points shall be carried out in accordance with the relevant regulations in HJ 194 and HJ/T 55. Air sampler (6.1) Airtightness check and flow calibration should be performed prior to use. Connect a 9 ml sulphuric acid absorbent (5.5) to the air sampler (6.1) 25 ml small multi-well glass plate brown absorption bottle (6.5), sample at a flow rate of about 0.5 L/min, and the sampling