

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

HJ 976-2018

**Solid waste - Determination of benzene and its analogies -
Headspace/gas chromatography-mass spectrometry**

固体废物 苯系物的测定 顶空气相色谱-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 Law of the People's Republic of China on the Prevention and Control of Environmental Pollution by Solid Waste this standard is formulated to protect the ecological environment, protect human health, and regulate the determination of benzene in solid waste and its leachate.

This standard specifies the headspace/gas chromatography-mass spectrometry for the determination of benzene in solid waste and its leachate.

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

This standard is the first release.

This standard is formulated by the Department of Eco-Environmental Monitoring, the Department of Regulation and Standards of the Ministry of Ecology and Environment.

This standard was drafted. Anshan City Environmental Monitoring Center Station.

This standard is verified by. Liaoning Provincial Environmental Monitoring Experimental Center, Shenyang Environmental Monitoring Center Station, Dalian Environmental Monitoring Center, Fushun Environmental Monitoring Center Station, Liaoyang Environmental Monitoring Station and Jinzhou Environmental Monitoring Center Station.

This standard is approved by the Ministry of Ecology and Environment on November 13, 2018.

This standard has been implemented since March 1, 2019.

This standard is explained by the Ministry of Ecology and Environment.

1 Determination of benzene series in solid wastes by headspace/gas chromatography-mass spectrometry Warning. The organic solvents and standard solutions used in the experiment are volatile and toxic substances. The reagent preparation and pretreatment process should be performed in a fume hood; wear protective equipment as required to avoid contact with skin and clothing.

1 Scope of application

This standard specifies the headspace/gas chromatography-mass spectrometry for the determination of benzene in solid waste and its leachate.

This standard applies to solid waste and its leachate in benzene, toluene, ethylbenzene, p-xylene, m-xylene, cumene, Determination of nine benzene compounds such as o-xylene, n-propylbenzene and styrene. If other benzene compounds are verified, this standard can also be used.

Method determination.

When the solid waste sample volume is 2 g, the detection limit of the 9 targets is 3~5 µg/kg, and the lower limit is 12~20.

Gg/kg. When the sample volume of solid waste leachate is 10 ml, the detection limit of the nine targets is 0.6~ 1 µg/L.

The lower limit is 2.4~4 µg/L. See Appendix A for details.

2 Normative references

This standard refers to the following documents or their terms. For undated references, the valid version applies to this standard.

HJ/T 20 Technical Specifications for Sampling and Sample Preparation of Industrial Solid Waste HJ/T 298 Hazardous Waste Identification Technical Specification HJ/T 299 solid waste leaching toxicity leaching method sulfuric acid nitric acid method HJ/T 300 solid waste leaching toxicity leaching method acetic acid buffer solution method

3 Principle of the method

At a certain temperature, the benzene series in the sample in the headspace bottle volatilizes into the upper space, generating vapor pressure and achieving thermodynamic motion.

State balance. The benzene series in the gas phase was separated by gas chromatography and detected by a mass spectrometer. Passing with standard material retention time Qualitative comparison with standard mass spectra and internal standard method.

4 Reagents and materials

Unless otherwise stated, analytically pure reagents that meet national standards were used for the analysis.

4.1

Experimental water. double distilled water or water prepared by pure water equipment. Need to pass a blank test before use, the target is thick the degree is below the method detection limit.

4.2

Methanol (CH₃OH). pesticide residue level or equivalent. Confirm the retention time interval of the target by blank test There is no interference peak appearing inside.

4.3

Sodium chloride (NaCl).

Bake in a muffle furnace at 400 ° C for 4 h, cool and transfer to a ground glass bottle and store in a desiccator.

24.4 Phosphoric acid (H₃PO₄). rho=1.69 g/ml, excellent grade.

4.5

Saturated sodium chloride solution.

Measure 500 ml of water (4.1), add phosphoric acid (4.4) to adjust pH ≤ 2, add 180 g of sodium chloride (4.3), dissolve and Mix and store at 4 ° C or below.

4.6

Standard stock solution. rho = 1000 mg/L, the solvent is methanol. Can directly purchase certified standard solutions, or standard materials Formulated.

4.7

Standard use solution 1. rho = 10 mg/L, the solvent is methanol.

4.8

Standard use solution 2. rho = 100 mg/L, the solvent is methanol.

4.9

Internal standard solution. rho=250 mg/L, the solvent is methanol.

Fluorobenzene was selected as an internal standard. A certified standard solution can be purchased directly.

4.10

Alternative standard solution. $\rho = 2000 \text{ mg/L}$, the solvent is methanol.

Toluene- d_8 or 4-bromofluorobenzene was used as a substitute.

4.11

Alternative use solution 1. $\rho = 10 \text{ mg/L}$, the solvent is methanol.

4.12

Alternative use solution 2. $\rho = 100 \text{ mg/L}$, the solvent is methanol.

4.13 4-bromofluorobenzene (BFB) solution. $\rho = 25 \text{ mg/L}$, the solvent is methanol.

The certified standard solution can be purchased directly or can be prepared with a high concentration standard solution.

4.14

Quartz sand. $0.30\sim 0.85 \text{ mm}$ (50~20 mesh).

It was fired in a muffle furnace at 400°C for 4 h, cooled and transferred to a ground glass bottle and sealed for storage.

4.15

Carrier gas. high purity helium (purity $\geq 99.999\%$).

5 Instruments and equipment

5.1

Gas Chromatograph. Capillary split/splitless inlet for temperature programming.

5.2

Mass Spectrometer. Electron Bombardment (EI) Ionization Source with 70 eV, NIST Mass Spectrometry Library, Manual/Automatic Tuning, Number According to acquisition, quantitative analysis and library search and other functions.

5.3

Column. Quartz capillary column, $30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \text{ m}$ (polyethylene glycol 20M), can also be used He is equivalent to a capillary column.

5.4

Headspace sampler. The temperature control range of the furnace is between room temperature and 120°C ; the temperature control accuracy is $\pm 1^\circ \text{C}$.

5.5

Reciprocating Oscillator. The oscillation frequency is 50~250 times/min, and the oscillation