

ICS 13.040.20
CCS Z 15

HJ

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

HJ 739-2015

**Ambient air. Determination of nitroaromatics. Gas
chromatography mass spectrometry**

环境空气 硝基苯类化合物的测定 气相色谱-质谱法

(English Translation)

Issued on: February 7, 2015

Implemented on: April 1, 2015

Issued by: Ministry of Ecology and Environment

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Foreword

In order 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 Atmospheric Pollution, Health, regulate the ambient air nitrobenzene monitoring methods, the development of this standard.

This standard specifies the gas chromatographic-mass spectrometric method for the determination of gaseous nitrobenzenes in ambient air and unorganized emissions.

This standard is the first release.

Appendix A and Appendix B of this standard are informative.

This standard is organized by the Ministry of Environmental Protection Science and Technology Standards Division.

The main drafting unit of this standard. Tianjin Environmental Monitoring Center.

The standard verification unit. National Environmental Protection Odor Pollution Control Key Laboratory, Tianjin Hexi District Environmental Monitoring Station, Ministry of Agriculture Environment Protection of scientific research and testing, Tianjin Tanggu District Environmental Monitoring Station, Tianjin Dongli District Environmental Monitoring Station,

Tianjin Dagang District Environmental Monitoring Station.

This standard is approved by the Ministry of Environmental Protection on February 7, 2015.

This standard is implemented as of April 1, 2015.

This standard is explained by the Ministry of Environmental Protection.

Determination of nitrobenzene compounds in ambient air Gas chromatography - mass spectrometry WARNING. The reagents and standard solutions used in the experiment are volatile, and the process should be carried out in the fume hood.

Line, according to the provisions of the requirements of wearing protective equipment, to avoid contact with the skin.

1 Scope of application

This standard specifies the determination of gaseous nitrobenzene compounds in ambient air by gas chromatography-mass spectrometry.

This standard applies to the determination of nitrobenzene, nitrotoluene and nitrochlorobenzene in ambient air and unorganized emissions.

When the sample volume is 22.5 L, nitrobenzene, p-nitrotoluene, m-nitrotoluene, o-nitrotoluene, p-nitrochlorobenzene, the detection limit of m-nitrochlorobenzene and o-nitrochlorobenzene was 0.001 mg/m³ and the lower limit was 0.004 mg/m³.

2 Normative reference documents

The contents of this standard refer to the terms of the following documents. For those who do not specify a date, the valid version applies to this standard.

Technical Guidelines for Sampling of Semi - Volatile Organic Compounds in Ambient Air
Technical Guidelines for Unorganized Emissions Monitoring of Air Pollutants
Technical specification for manual monitoring of ambient air quality

3 Principle of the method

Using silica gel sampling tube to collect ambient air and non-organized emissions of nitrobenzene compounds in the exhaust, with dichloromethane ultrasonic solution Suction, by gas chromatography - mass spectrometry separation, detection. According to the retention time and mass spectrometry qualitative, internal standard method quantitative.

4 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents and distilled water are used in accordance with national standards.

4.1

dichloromethane (CH_2Cl_2). pesticide residues.

4.2

Benzene (C_6H_6). pesticide residues.

4.3

isooctane (C_8H_{18}). pesticide residues.

4.4

Reference materials and internal standard materials. nitrobenzene ($\text{C}_6\text{H}_5\text{NO}_2$), o-nitrotoluene ($\text{C}_7\text{H}_7\text{NO}_2$), m-nitrotoluene ($\text{C}_7\text{H}_7\text{NO}_2$), P-nitrotoluene ($\text{C}_7\text{H}_7\text{NO}_2$), m-nitrochlorobenzene ($\text{C}_6\text{H}_4\text{ClNO}_2$), p-nitrochlorobenzene ($\text{C}_6\text{H}_4\text{ClNO}_2$), o-nitrochlorobenzene ($\text{C}_6\text{H}_4\text{ClNO}_2$), the standard material is nitrobenzene-d5, the above standard purity should not be less than 98%, dark storage.

4.5

Standard mixed stock solution. $\rho = 2.00 \text{ mg/ml}$.

0.05 g (accurate to 0.0001 g) standard (4.4), respectively, into the 25 ml volumetric flask, add 2-3 (4.2) dissolved, and then Isooctane (4.3) constant volume to the mark, shake, into the dense bottle seal, 4 °C under the closed light Save, save for one year. Can also be purchased directly to prove mixed standard solution.

24.6 Internal standard stock solution. $\rho = 2.00 \text{ mg/ml}$.

Weigh 0.05 g (accurate to 0.0001g) internal standard, into the 25 ml volumetric flask, add 2-3 drops of benzene (4.2) dissolved, and then with isooctane (4.3) constant volume to the mark, shake, into the dense bottle seal, 4 °C sealed dark preservation, the preservation period of one year.

Can also be purchased directly to the standard solution.

4.7

Tuning standard stock solution. $\rho = 50 \text{ } \mu\text{g/ml}$.

The commercially available decaphenylphosphine (DFTPP) was added to a 10.0 ml brown volumetric flask containing an appropriate amount of dichloromethane (4.1) Diluted with dichloromethane (4.1) to 50 $\mu\text{g/ml}$ and stored frozen.

4.8

Standard intermediate solution. $\rho = .200 \text{ mg/L}$.

Take the 1.00 ml nitrobenzene standard mixed stock solution (4.5) in a 10 ml volumetric flask to prepare dichloromethane (4.1) as the solvent mixture standard use of liquid, which nitrobenzene compounds concentration of .200 mg/L, in the refrigerator 4 °C conditions can

be dense Keep away from light for six months.

4.9

internal standard solution. $\rho = 0.200 \text{ mg/L}$.

Take 1.00 ml of nitrobenzene-d5 standard stock solution (4.6) in a 10 ml volumetric flask with methylene chloride (4.1) Scale preparation of the concentration of 0.200 mg/L of the internal standard liquid, 4 °C conditions can be sealed under the protection of light for six months.

4.10

standard working solution. the nitrobenzene standard intermediate solution (4.8) with dichloromethane (4.1) prepared into several different concentrations the standard working solution, the internal standard concentration for the standard working curve of the middle point, is now with the distribution.

4.11

Silica Sampling Tube. Buy commercially available Silica Sampling Tube, Silicone Sampling Tube Size 20cm long, 6mm OD Diameter 4 mm, the tube is equipped with two 40 mesh silica gel, the former 150 mg, the latter 75 mg, the middle with 2 mm glass wool separated, Two layers of silica gel are silted with silanized glass wool and filled at both ends.

5 Instruments and equipment

5.1

Gas Chromatography-Mass Spectrometer. with capillary column shunt/splitless inlet, with constant current or constant voltage function, programmable temperature, with EI source and chemical workstation.

5.2

Atmospheric Sampler. Sampling flow rate can reach 0.1 ~ 1.0 L/min, flow accuracy $\pm 1\%$.

5.3

Ultrasonic cleaner. Power 250 W.

5.4

Analysis of balance. accuracy of 0.0001g.

5.5

Columns. 60 m x 0.32 mm, 1.0 m film thickness (100% dimethylpolysiloxane column), other equivalent or Tested capillary column.