

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 716-2014

**Water quality. Determination of nitroaromatics. Gas
chromatography mass spectrometry**

水质 硝基苯类化合物的测定 气相色谱-质谱法

(English Translation)

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Foreword

To implement the "People's Republic of China Environmental Protection Law" and "People's Republic of China Water Pollution Prevention Law," protecting the environment, safeguarding human Healthy, standardized measurement method nitrobenzene compounds in water, the development of this standard.

This standard specifies the determination of surface water, groundwater, industrial wastewater, sewage and seawater nitrobenzene compounds by GC -Mass spectrometry.

This standard is the first release.

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

This standard is developed by the Ministry of Environmental Protection Science, Technology organization.

Drafting of this standard. Tianjin Environmental Monitoring Center.

The standard verification unit. State Key Lab of odor pollution control environmental protection, scientific research and monitoring by the Ministry of Agriculture, Environmental Protection, Tianjin Hexi District Environmental Monitoring Station, Tianjin Tanggu District Environmental Monitoring Station, Tianjin Dongli District Environmental Monitoring Station

and Tianjin Dagang District Environmental Monitoring Station.

This standard is approved by the Ministry of Environmental Protection November 27, 2014.

This standard since January 1, 2015 implementation.

The standard explanation by the Ministry of Environmental Protection.

Water quality measurement 1 nitrobenzene compounds Gas chromatography - mass spectrometry Warning. Reagents and standard solutions used in this method has some toxicity, potentially harmful to health, and this should be avoided Direct contact with these chemicals. Operation should be required to required to wear protective equipment, sample pre-treatment process should be carried out in a fume hood.

1 Scope

This standard specifies the liquid-liquid extraction Determination of nitrobenzene compounds and solid phase extraction/gas chromatography - mass spectrometry.

This standard applies to surface water, groundwater, measurement of industrial wastewater, sewage and seawater 15 kinds of nitrobenzene compounds.

When the sample volume is 1L, the method detection limit of the target compound 0.04 µg/L ~ 0.05 µg/L, detection limit of 0.16 µg/L ~ 0.20 µg/L. See Appendix A.

2 Normative references

This standard refers to the contents of the following documents or the terms. For undated references, the effective version applies to this standard quasi.

GB 17378 specification for marine monitoring Part 3 sample collection, storage and transport HJ/T 91 surface water and wastewater monitoring technical specifications HJ/T 164 groundwater environmental monitoring technical specifications

3 PRINCIPLE OF THE METHOD

Liquid-liquid extraction or solid phase extraction methods nitrobenzene compounds in the sample, extract was dehydrated, concentrated, purified and constant volume Separation by gas chromatography, mass spectroscopy. Based on retention time and mass spectra qualitative, quantitative internal standard.

4 and elimination of interference High concentration samples with low concentration samples alternans cause interference when analyzing a high concentration samples should be analyzed after a blank sample or Reagent blank in order to prevent cross-contamination. If a target compound contained in the sample before the next sample also appeared, the analyst must shall not be proved because the remaining result.

5 Reagents and materials

Unless otherwise indicated, the use of line with national standards for analytical chemistry reagents and distilled water analysis.

5.1

acetone ($\text{C}_3\text{H}_6\text{O}$). pesticide grade.

5.2

methanol (CH_3OH). pesticide grade.

5.3

toluene (C_7H_8). pesticide grade.

5.4

dichloromethane (CH_2Cl_2). pesticide grade.

5.5

n-hexane (C_6H_{14}). pesticide grade.

5.6

sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$).

25.7 anhydrous sodium sulfate (Na_2SO_4).

Calcined at 400 °C for 4h, cooled charged grinding mouth glass bottles, placed in a desiccator save.

5.8

hydrochloric acid (HCl). (HCl) = 1.19 g/ml.

5.9

Sodium hydroxide (NaOH).

5.10

nitrobenzene stock solution. = 10 mg/ml, the solvent is methanol, commercially available.

5.11

internal standard stock solution. = 10 mg/ml, the solvent is methanol, commercially.

5.12

alternatives stock solution. = 10 mg/ml, the solvent is methanol, commercially.

5.13

tuning stock standard solution. = 2.5 mg/ml, the solvent is methanol, commercially

available.

5.14

nitrobenzene using standard solution. = 200 µg/ml.

Take 0.50 ml nitrobenzene stock solution (5.10) added to a solution containing an appropriate amount of methylene chloride (5.4) of 10.0 ml brown volumetric flask, Diluted with dichloromethane (5.4) to the mark, frozen.

5.15

internal standard solution is used. = 200 µg/ml.

Take 0.50 ml of internal standard stock solution (5.11), containing an appropriate amount was added to methylene chloride (5.4) in 10.0 ml brown flask, with Dichloromethane (5.4) diluted to the mark, frozen.

5.16

alternatives using standard solution. = 200 µg/ml.

Take 0.50 ml substitute stock solution (5.12) added to a solution containing an appropriate amount of methylene chloride (5.4) of 10.0 ml brown volumetric flask, Diluted with dichloromethane (5.4) to the mark, frozen.

5.17

tuning using standard solution. = 50 µg/ml.

Take 0.20 ml tuning stock standard solution (5.13) added to a solution containing an appropriate amount of methylene chloride (5.4) of 10.0 ml brown volumetric flask Dichloromethane (5.4) diluted to the mark, frozen.

5.18

hydrochloric acid solution. 11.

Sodium hydroxide solution 5.19. C (NaOH) = 0.1 mol/L.

5.20

dichloromethane - n-hexane. 91.

5.21

SPE column. C18 or equivalent type of filler or combination of fillers type filler, 1000 mg/6.0 ml, commercially available, or solid phase extraction disk, etc.

Extraction performance with equivalent items.

5.22

Florisil Column. 1000 mg/6.0 ml, particle size of 40 µm, commercially available.