

HJ

INDUSTRY STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

HJ 648-2013

Replacing GB/T 13194-1991

Water quality. Determination of nitroaromatics by gas chromatography

水质 硝基苯类化合物的测定 液液萃取/固相萃取-气相色谱法

(English Translation)

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Foreword

To protect the environment and protect the human body 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 Water Pollution Prevention and Control this standard is established for the determination of healthy, standardized nitrobenzene compounds in

water.

This standard specifies the liquid-liquid extraction and solidification of nitrobenzene compounds in surface water, groundwater, industrial wastewater, domestic sewage and seawater.

Phase extraction gas chromatography.

This standard is for the determination of water nitrobenzene, nitrotoluene, nitrochlorobenzene, dinitrotoluene by gas chromatography (GB 13194-91) Revision.

This standard was first published in 1991. The original drafting unit was the Wuhan Environmental Monitoring Center Station. This is the first revision. This revision the main content is.

- Amend the standard name to "Determination of water-nitrobenzene compounds by liquid-liquid extraction/solid phase extraction - gas chromatography".
- Increased the type of measurement of nitrobenzene compounds.
- Expanded the scope of application of the method and increased the determination of domestic sewage and seawater.
- Added sample preparation methods for solid phase extraction.
- The analytical column was changed from packed column to capillary column, and the chromatographic conditions were changed accordingly.
- Liquid-liquid extraction solvent changed from benzene to toluene.
- Modified the quantitative method for nitrobenzene compounds.
- Modified the method detection limit.
- Added quality assurance and quality control provisions.

Gas chromatographic method for the determination of nitrobenzene, nitrotoluene, nitrochlorobenzene and dinitrotoluene from the date of implementation of this standard (GB 13194-91) abolished.

Appendix A of this standard is a normative appendix, and Appendix B is an informative appendix.

This standard was formulated by the Science and Technology Standards Department of the Ministry of Environmental Protection.

This standard was drafted. Tianjin Environmental Monitoring Center, Environmental Standards Institute of the Ministry of Environmental Protection.

This standard is verified by the National Key Laboratory of Environmental Pollution Control, Environmental Monitoring Center of Shenyang, and Environmental Protection of the

Ministry of Agriculture.

Environmental Protection Monitoring Institute, Tianjin Tanggu District Environmental Protection Monitoring Station, Tianjin Dongli District Environmental Protection Monitoring Station and Tianjin Dagang District Environmental Protection Monitoring station.

This standard was approved by the Ministry of Environmental Protection on June 3, 2013.

This standard has been implemented since September 1, 2013.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of nitrobenzene compounds - Liquid-liquid extraction/solid phase extraction - Gas chromatography

1 Scope of application

This standard specifies liquid-liquid extraction and solid phase extraction gas chromatography for the determination of 15 nitrobenzene compounds in water. 15 nitrobenzenes the compounds include nitrobenzene, p-nitrotoluene, m-nitrotoluene, o-nitrotoluene, p-nitrochlorobenzene, m-nitrochlorobenzene, o-nitrochloride.

Benzene, p-dinitrobenzene, m-dinitrobenzene, o-dinitrobenzene, 2,4-dinitrotoluene, 2,6-dinitrotoluene, 3,4-dinitrotoluene, 2,4-Dinitrochlorobenzene, 2,4,6-trinitrotoluene.

This standard applies to the determination of nitrobenzene compounds in surface water, groundwater, industrial wastewater, domestic sewage and seawater.

The sampling amount of liquid-liquid extraction method is 200ml, the detection limit of the method is 0.017µg/L~0.22µg/L; when the sampling amount of solid phase extraction is 1.0L, the detection limit of the method was 0.0032µg/L~0.048µg/L. See Appendix A for details.

2 Normative references

The contents of this standard refer to the terms in the following documents. For undated references, the valid version applies to this standard.

GB 17378 Ocean Monitoring Code HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications

3 Principle of the method

Liquid-liquid extraction. The nitrobenzene compound in the water is extracted with a certain amount of toluene, and the extract is subjected to dehydration, purification, and chromatographic analysis.

Solid phase extraction. adsorption of nitrobenzene compounds in water by solid phase extraction column or extraction disk, elution with n-hexane/acetone, eluent After dehydration and constant volume, chromatographic analysis was carried out.

The extract is injected into a gas chromatograph, and the target compound is separated by a quartz capillary column and determined by an electron capture detector.

Qualitative, external standard method quantitative.

4 Interference and elimination

4.1

Organic compounds such as organochlorine pesticides (hexachlorocyclohexane, DDT), halogenated hydrocarbons, chlorobenzene, etc., which may coexist in the water sample, are on the electron capture detector.

Although there is a response, there is no significant interference with the method in the method due to the difference in retention time.

4.2

Other substances in the water that may be co-existing with halogen or nitrogen in the electron capture detector may interfere with the determination, and the polarity is poor.

If the two larger capillary columns are separated and measured separately, the qualitative error can be greatly reduced.

4.3

For samples with complex background interference, qualitative determination can also be carried out using gas chromatography mass spectrometry.

5 Reagents and materials

Analytical purification reagents that meet national standards are used for analysis, unless otherwise stated. The test water is freshly prepared deionized water or steamed.

Distilled water.

5.1

n-Hexane (C₆H₁₄). Chromatographically pure.

5.2

Acetone (C₃H₆O). chromatographically pure.

5.3

Methanol (CH₄O). Chromatographically pure.