

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

HJ 822-2017

**Water quality -- Determination of aniline compounds -- Gas
chromatography mass spectrometry**

水质 苯胺类化合物的测定 气相色谱-质谱法

(English Translation)

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Appendix A (normative) The quantitation of the target compound, the auxiliary ion, the detection limit of the method, and the lower limit of determination.

Appendix B (informative) method of precision and accuracy

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 Water Pollution, the protection of human health, regulate the determination of aniline compounds in water, the development of this standard.

This standard specifies the gas of 19 kinds of aniline compounds in surface water, groundwater, seawater, domestic sewage and industrial waste water.

Determination of Chromatography - Mass Spectrometry.

This standard is the first release.

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

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

The main drafting of this standard. Shanghai Pudong New Area Environmental Monitoring

Station.

The standard verification unit. Shanghai Environmental Monitoring Center, Suzhou City Environmental Monitoring Center Station, Shanghai Entry-Exit Inspection and Quarantine Bureau, Shanghai Chemical Environmental Protection Monitoring Station, Shanghai Jiading District Center for Disease Control and Prevention.

The environmental protection department of this standard approved on March 30, 2017.

This standard has been implemented since May 1, 2017.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of aniline compounds - Gas chromatography - mass spectrometry Warning. Dichloromethane, aniline compounds and other reagents and solutions are volatile or semi-volatile toxic substances, the solution Preparation should be carried out in the fume hood, the operation should be required to wear protective equipment, to avoid inhalation or contact with skin and clothing.

1 Scope of application

This standard specifies the determination of 19 aniline compounds in water by gas chromatography-mass spectrometry.

This standard is applicable to the determination of aniline compounds in surface water, groundwater, seawater, domestic sewage and industrial waste water.

19 aniline compounds include aniline, 2-chloroaniline, 3-chloroaniline, 4-chloroaniline, 4-bromoaniline, 2-nitroaniline, 2,4,6-trichloroaniline, 3,4-dichloroaniline, 3-nitroaniline, 2,4,5-trichloroaniline, 4-chloro-2-nitroaniline, 4-Nitroaniline, 2-chloro-4-nitroaniline, 2,6-dichloro-4-nitroaniline, 2-bromo-6-chloro-4-nitroaniline, 2-chloro-Nitroaniline, 2,6-dibromo-4-nitroaniline, 2,4-dinitroaniline, 2-bromo-4,6-dinitroaniline. After verification, Other methods can also be used for other aniline compounds.

When the sampling volume is 1000ml and the concentrated volume is 1.0ml, the detection limit is 0.05µg/L ~ 0.09µg/L, Limited to 0.20µg/L ~ 0.36µg/L, see Appendix A.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. For undated references, the valid version is applicable in this standard.

Specification for ocean monitoring Technical specification for surface water and wastewater monitoring Technical specification for groundwater environmental monitoring

3 Principle of the method

The aniline compounds in the water sample were extracted with dichloromethane under the condition of $\text{pH} \geq 11$. The extract was dehydrated, concentrated and purified, Measured by gas chromatography/mass spectrometry. Depending on the retention time of the target compound and the standard mass spectrum or characteristic ion characterization, Quantitative method.

4 Reagents and materials

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

4.1

dichloromethane (CH_2Cl_2). pesticide residues.

4.2

isopropyl alcohol ($\text{C}_3\text{H}_8\text{O}$). pesticide residues.

4.3

n-Hexane (C_6H_{14}). Pesticide level.

4.4

Methanol (CH_3OH). Pesticide level.

4.5

Sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$).

4.6

sodium chloride (NaCl). 400 °C baking 4h, slightly cold after the sealed glass bottle, cold to room temperature stand-by.

4.7

anhydrous sodium sulfate (Na_2SO_4). 400 °C baking 4h, slightly cold after the sealed glass bottle, cold to room temperature stand-by.

4.8

Sodium hydroxide solution. $c(\text{NaOH}) = 10 \text{ mol/L}$.

Take 40 g of sodium hydroxide in a small amount of water and dilute to 100 ml.

4.9

Sulfuric acid solution. 1 1.

4.10

aniline compounds standard stock solution. $\rho = 1000 \mu\text{g/ml}$, containing 19 kinds of target compounds.

4.11

Substitutes (aniline-d5) Standard stock solution. $\rho = .2000 \mu\text{g/ml}$.

4.12

internal standard (1,2-dichlorobenzene-d4, phenanthrene-d10) stock solution. $\rho = .2000 \mu\text{g/ml}$.

4.13

aniline compounds standard intermediate. $\rho = 100 \mu\text{g/ml}$.

Take 1.00 ml of aniline compound standard stock solution (4.10) into a 10 ml volumetric flask and use methylene chloride (4.1) the volume is transferred to the reagent bottle (5.9) of the attached polytetrafluoroethylene spin cover.

4.14

Substituted (aniline-d5) Intermediate. $\rho = 100 \mu\text{g/ml}$.

Absorb 0.50 ml of the standard stock solution (4.11) to 10 ml volumetric flask, with methylene chloride (4.1) volume, Transfer to the reagent bottle (5.9) of the attached polytetrafluoroethylene spin cover.

4.15

Internal standard 1 (1,2-dichlorobenzene-d4) Intermediate. $\rho = 400 \mu\text{g/ml}$.

Take 2.0ml of 1,2-dichlorobenzene-d4 stock solution (4.12) into a 10ml volumetric flask, mix with methylene chloride (4.1) Transfer to the reagent bottle (5.9) of the attached polytetrafluoroethylene spin cover.

4.16

Internal standard 2 (phenanthrene-d10) Intermediate. $\rho = 40 \mu\text{g/ml}$.

Take.200 μl of phenanthrene-d10 stock solution (4.12) into a 10 ml volumetric flask and equilibrate with dichloromethane (4.1) Attached to the reagent bottle (5.9) of the attached tetrafluoroethylene rotary cover.

4.17

aniline compounds standard use of liquid (including substitutes). $\rho = 10.0 \mu\text{g/ml}$.

Respectively, 1.00 ml of aniline compounds standard intermediate (4.13) and 1.00 ml of the intermediate (4.14) to a 10 ml volumetric flask, transferred to methylene chloride (4.1), transferred to an agglutinated tetrafluoroethylene rotary cap reagent bottle (5.9) in the preservation.