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HJ

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

HJ 1048-2019

**Water quality - Determination of 17 aniline compounds - Liquid
chromatography-triple quadrupole mass spectrometry**

水质 17 种苯胺类化合物的测定 液相色谱-三重四极杆质谱法

(English Translation)

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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 Water Pollution Control", protect the ecology Environment, protect human health, standardize the determination method of aniline compounds in water, and formulate this standard.

This standard specifies the liquid phase of 17 aniline compounds in surface water, groundwater, domestic sewage and industrial wastewater.

Chromatography-Triple Quadrupole Mass Spectrometry.

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

This standard is issued for the first time.

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

This standard was drafted. Sichuan Ecological Environment Monitoring General Station.

Verification units of this standard. Chongqing Ecological Environment Monitoring Center, Guangyuan Environmental Monitoring Center Station, Panzhihua Environmental Monitoring Center Monitoring Center Station, Shengzhou Environmental Monitoring Center Station, Yibin Environmental Monitoring Center Station and Nanchong Environmental Monitoring Center Station.

This standard was approved by the Ministry of Ecology and Environment on October 24, 2019.

This standard will be implemented from April 24, 2020.

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

Determination of 17 aniline compounds in water quality Liquid chromatography-triple quadrupole mass spectrometry Warning. The standard materials used in the experiment have high toxicity or carcinogenicity. The reagent preparation and sample preparation process should be Do it in a fume hood; wear protective equipment as required to avoid contact with skin and clothing.

1 Scope

This standard specifies liquid chromatography-triple quadrupole mass spectrometry for the determination of 17 aniline compounds in water.

This standard applies to o-phenylenediamine, aniline, benzidine, p-formylamine in surface water, groundwater, domestic sewage and industrial wastewater.

Aniline, o-methoxyaniline, o-toluidine, 4-nitroaniline, 2,4-dimethylaniline, 3-nitroaniline, 4-chloroaniline, 2-Nitroaniline, 3-chloroaniline, 2-naphthylamine, 2,6-dimethylaniline, 2-methyl-6-ethylaniline, 3,3'-dichlorobenzidine, and 2,6-Determination of 17 aniline compounds such as diethylaniline.

When the direct injection method was used and the injection volume was 10 µl, the method detection limit of 17 aniline compounds was 0.1 µg/L ~ 3 µg/L, the lower limit of determination is 0.4 µg/L ~ 12 µg/L; when the solid phase extraction method is used, the sampling volume is 100 ml (enriched 50 Times), when the injection volume is 10 µl, the method detection limit of 16 aniline compounds is 0.007 µg/L ~ 0.1 µg/L.

The lower limit is 0.028 µg/L to 0.4 µg/L. See Appendix A for details.

2 Normative references

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

HJ/T 91 Technical specifications for surface water and sewage monitoring HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring

3 Method principle

Samples were injected directly after filtration or sampled after being enriched and purified by a cation exchange solid phase extraction column.

Separation and detection of aniline compounds by heavy quadrupole mass spectrometry. Qualitative analysis based on retention time and characteristic ions, quantification by internal standard method.

4 Interference and cancellation

4.1

When matrix interferences exist in the sample, you can optimize the chromatographic conditions, dilute the sample, reduce the injection volume, and Products are reduced or eliminated by pretreatment. When using solid phase extraction, you can also reduce the sample volume or increase the sample the dilution factor reduces matrix interference.

4.2

When there is interference of isomers in the sample, you can improve the resolution by changing the chromatographic conditions or choose a different the secondary mass ion eliminates interference.

4.3

When oxidizing substances such as residual chlorine are present in the sample, sodium thiosulfate (5.8) can be added during sample collection and storage (7.1) Eliminate interference.

5 Reagents and materials

Unless otherwise stated, all analytical reagents that meet national standards are used for analysis.

Standard compound of pure water.

5.1

Methanol (CH_3OH). pure by liquid chromatography.

5.2

Ethanol ($\text{C}_2\text{H}_5\text{OH}$). excellent grade pure.

5.3

Formic acid (HCOOH). pure liquid chromatography.

5.4

Acetic acid (CH_3COOH). pure liquid chromatography.

5.5

Hydrochloric acid. $\rho(\text{HCl}) = 1.19 \text{ g/ml}$.

5.6

Ammonia. $\rho(\text{NH}_3 \cdot \text{H}_2\text{O}) = 0.91 \text{ g/ml}$, excellent grade pure.

5.7

Sodium hydroxide (NaOH).

5.8

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

5.9

Hydrochloric acid solution. 1 1.

5.10

Formic acid solution. $\phi(\text{HCOOH}) = 0.005\%$.

Accurately transfer $50.0 \mu\text{l}$ of formic acid (5.3) into a 1 L volumetric flask previously filled with an appropriate amount of water.

Mix well.

5.11

Acetic acid solution. 3 97.

Mix with acetic acid (5.4) and water in a volume ratio of 3.97.

5.12

methanol solution. 1 9.

Mix with methanol (5.1) and water in a volume ratio of 1. 9.

5.13

Sodium hydroxide solution. $c(\text{NaOH}) = 1 \text{ mol/L}$.

Weigh 4 g of sodium hydroxide (5.7) in water and dilute to 100 ml with water.

5.14

Ammonia methanol solution I. 5 95.

Mix ammonia (5.6) and methanol (5.1) in a volume ratio of 5.95.