

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

HJ 809-2016

**Water quality - Determination of nitrosamine compounds - Gas
chromatography**

水质 亚硝胺类化合物的测定 气相色谱法（发布稿）

(English Translation)

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Table of Contents

Preface	.ii
1 Scope of application	
2 Normative reference documents	
3 Principle of the method	
4 Reagents and materials	
5 Instruments and equipment	
6 Sample	
7 Analysis steps	
8 Results are calculated and expressed	
9 Precision and accuracy	
10 Quality assurance and quality control	
11 Waste treatment	
Appendix A (informative) method of precision and accuracy	
Appendix B (informative) Reference conditions for auxiliary qualitative columns	

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, Protection of human health, regulate the determination of nitrosamines in water method, the development of this standard.

This standard specifies the determination of surface water, groundwater, industrial wastewater and domestic sewage nitrosamines in gas chromatography law.

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. Gansu Province Environmental Monitoring Center Station.

The standard verification unit. Fujian Province Environmental Monitoring Center Station, Shaanxi Province Environmental Monitoring Center Station, Qinghai Province Environmental Monitoring Heart station, Lanzhou City Environmental Monitoring Station,

Zhangye City Environmental Monitoring Station and China Petroleum Lanzhou Petrochemical Branch Environmental Monitoring Station.

The environmental protection department of this standard approved on July 26, 2016.

This standard has been implemented since October 1, 2016.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of nitrosamines - Gas chromatographic method Warning. nitrosamines are carcinogens, their standard substances and standard stock solution in use, to avoid contact Skin, eyes, etc .; should be in a well-ventilated indoor fume hood to operate; use of dichloromethane, ether, pentane and other tests , Should wear protective equipment, avoid inhalation or contact with skin and clothing.

1 Scope of application

This standard specifies the determination of nitrosamines in water by gas chromatography.

This standard applies to surface water, groundwater, industrial wastewater and domestic sewage N-nitrosodimethylamine, N-nitroso Determination of diethylamine, N-nitroso-n-propylamine and N-nitrosodiphenylamine.

When the sampling volume of 250 ml, the standard method of detection limit were. N-nitrosodimethylamine 0.6 µg/L, N-nitrosodiamine 0.5 µg/L, N-nitrosodi-n-propylamine 0.5 µg/L, N-nitrosodiphenylamine 0.4 µg/L;

The lower limits are. N-nitrosodimethylamine 2.4 µg/L, N-nitrosodiethylamine 2.0 µg/L, N-nitrosodi-n-propylamine 2.0 Mg/L, N-nitrosodiphenylamine 1.6 µg/L.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Any annotated file that does not specify the date, its valid version Applicable to this standard.

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

3 Principle of the method

Extraction of nitrosamines from the sample with dichloromethane, dehydration and concentration, florisil column or alkaline oxidation Aluminum column after purification capacity. Separation by capillary column, hydrogen flame ionization detector (FID) determination. To retain the time qualitative, External standard method.

4 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents are used in accordance with national standards. The experimental water is freshly prepared Deionized water or distilled water.

4.1

dichloromethane (CH_2Cl_2). pesticide residues.

4.2

Methanol (CH_3OH). Pesticide level.

4.3

Acetone ($\text{C}_3\text{H}_6\text{O}$). pesticide residues.

4.4

pentane (C_5H_{12}). pesticide residues.

4.5

ether [$(\text{C}_2\text{H}_5)_2\text{O}$]. pesticide residues.

4.6

sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$). excellent grade pure.

4.7

anhydrous sodium sulfate (Na_2SO_4). excellent grade pure.

Before use, should be in the muffle furnace at $450\text{ }^\circ\text{C}$ for 4 h, after cooling into the grinding glass bottle sealed, placed in the dry Save the device.

4.8

Sodium chloride (NaCl). excellent grade pure.

Before use, should be in the muffle furnace at $350\text{ }^\circ\text{C}$ for 4 h, after cooling into the grinding glass bottle sealed, placed in the dry Save the device.

4.9

sulfuric acid. $\rho(\text{H}_2\text{SO}_4) = 1.84\text{ g/ml}$, excellent grade pure.

4.10

sulfuric acid solution. 1 4 (v/v).

Prepared with sulfuric acid (4.9).

4.11

sodium hydroxide (NaOH). excellent grade pure.

4.12

Sodium hydroxide solution. $c(\text{NaOH}) = 2 \text{ mol/L}$.

Weigh 8.0 g of sodium hydroxide (4.11) in the appropriate amount of water, to be cooled to room temperature and then diluted to 100 ml, mixed uniform. Transferred to the plastic reagent bottle to save.

4.13

Florisil. 60 mesh to 100 mesh.

In the muffle furnace at 650 °C for 5 h, after cooling stored in a glass plug or lined with aluminum foil screw cap glass Inside the container. Before use, take the appropriate amount of calcined silica, in a lined aluminum foil screw cap glass container at 130 °C Activate for 16 h, add 2 ml of water, shake for 10 min, and allow to stand for at least 2 h.

4.14

Alumina. 100 mesh to 200 mesh.

Weigh 100.0 g Alumina in 500 ml glass flasks with glass stoppers or aluminum foil screw caps, add 2 ml Water, shake fully mixed for 10 min, standing for at least 2 h after use.

4.15

Mixed standard stock solution. $\rho = 2000 \text{ mg/L}$.

Respectively, weighed 0.200 g (accurate to 0.0001 g) N-nitrosodimethylamine, N-nitrosodiethylamine, N-nitroso N-propylamine and N-nitrosodiphenylamine were dissolved in an appropriate amount of dichloromethane (4.1) and transferred to a 10 ml volumetric flask.

Methane (4.1) constant volume to the mark, mix, at 4 °C below the cold, dark and sealed can be stored for 6 months. Can also be direct Purchase commercially available standard solution.

4.16

Mixing standard liquid. $\rho = 200 \text{ mg/L}$.

Accurate removal of 1.00 ml of mixed standard stock solution (4.15) in a 10 ml volumetric flask with dichloromethane (4.1) to the mark line, mix, at 4 °C below the cold, dark and sealed can be stored for 2 months.

4.17

Glass wool. commercially available silanized glass wool.