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HJ

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

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**Water quality. Determination of total selenium.3,
3'-Diaminobenzidine spectrophotometric method**

水质 总硒的测定 3,3' -二氨基联苯胺分光光度法（发布稿）

(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 the Prevention and Control of Water Pollution, Human health, regulate the determination of total selenium in water, the development of this standard.

This standard specifies the determination of total selenium in surface water, groundwater, domestic sewage and industrial waste water 3,3'-diaminobenzidine spectrophotometry Degree method.

This standard is the first release.

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

The main drafting of this standard. Lanzhou City Environmental Monitoring Station.

The standard verification unit. Gansu Province Environmental Monitoring Center Station, Chengdu Environmental Monitoring Center Station, Jinchang City Environmental Monitoring Station, China Oil Group Company Lanzhou Company Testing Center, Baiyin City Environmental Monitoring Station and Lanzhou University Analysis and Testing Center.

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 total selenium 3,3' - diaminobenzidine spectrophotometric method Warning. nitric acid and perchloric acid with strong corrosive and strong oxidizing, hydrochloric acid with strong volatility and corrosive, should be worn during operation Protective equipment, avoid contact with skin and clothing. The digestion of all samples should be carried out in a fume hood. Toluene and 3,3'-diamino Benzidine is a toxic reagent, the waste generated in the experiment should be collected and treated.

1 Scope of application

This standard specifies the determination of total selenium in water 3,3'-diaminobenzidine spectrophotometric method.

This standard applies to the determination of total selenium in surface water, groundwater, domestic sewage and industrial waste water.

When the sampling volume is 200 ml, when the 30 mm cuvettes are used, the detection limit of this method is 2.0 µg/L and the lower limit of determination is 8.0 Mg/L.

2 Normative reference documents

The contents of this standard refer to the following documents or their terms. Where a reference file is not dated, its valid version applies this standard.

Technical specification for surface water and wastewater monitoring

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1 Total selenium

Samples were digested after the determination of selenium, that is, the sum of organic selenium and inorganic selenium in the sample.

4 Principle of the method

After digestion with the mixed acid, the total selenium in the sample was reduced to tetravalent by hydroxylamine hydrochloride, and under acidic conditions with the color reagent 3,3'-(3,3'-Diaminobenzidine) to form a yellow compound, which was extracted with toluene at 420 nm Measure the absorbance at the wavelength. In a certain concentration range, the total selenium content and absorbance values in line with Lambert - Beer's law.

Chemical reaction side the program is as follows.

5 Interference and elimination

Common ions in water generally do not interfere with the determination of total selenium in this process. When the iron ion concentration is greater than 50 mg/L, (EDTA-2Na) mixed reagent to mask or eliminate the interference with ethylenediamine tetraacetic acid disodium.

6 Reagents and materials

Unless otherwise stated, analytical pure chemical reagents are used in accordance with national standards. Experimental water is a newly prepared deionized Water or distilled water.

6.1

hydrochloric acid. $\rho(\text{HCl}) = 1.19 \text{ g/ml}$, excellent grade pure.

6.2

nitric acid. $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$, excellent grade pure.

6.3

perchloric acid. $\rho(\text{HClO}_4) = 1.67 \text{ g/ml}$, excellent grade pure.

6.4

Toluene. $\rho(\text{C}_7\text{H}_8) = 0.87 \text{ g/ml}$, Pesticide level.

6.5

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

6.6

Sodium hydroxide (NaOH).

6.7

Ethylenediaminetetraacetic acid disodium dihydrate ($\text{C}_{10}\text{H}_{18}\text{N}_2\text{Na}_2\text{O}_{10}$).

6.8

Hydroxylamine hydrochloride ($\text{NH}_2\text{OH} \cdot \text{HCl}$).

6.9

Cresol red ($\text{C}_{21}\text{H}_{18}\text{O}_5\text{S}$).

6.10

selenium powder (grain). mass fraction $\geq 99.9\%$.

6.11 3,3'-diaminobenzidine tetrahydrochloride ($C_{12}H_{14}N_4 \cdot 4HCl$), excellent grade pure.

6.12

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

Prepared with hydrochloric acid (6.1).

6.13

hydrochloric acid solution. $c = 0.1 \text{ mol/L}$.

Prepared with hydrochloric acid (6.1).

6.14

nitric acid solution. 1 1 (v/v).

Prepared with nitric acid (6.2).

6.15 Nitric acid - perchloric acid solution

Mixed with an equal volume of nitric acid (6.2) and perchloric acid (6.3).

6.16 Sodium hydroxide solution. $\rho = 100 \text{ g/L}$.

Weigh 50 g of sodium hydroxide (6.6) in the appropriate amount of water, to be cooled, diluted to 500 ml.

6.17 Cresol red solution. $\rho = 0.2 \text{ g/L}$.

Weigh 20.0 mg of cresol red (6.9) in the appropriate amount of water, add 1 drop of ammonia (6.5), dissolved and transferred to 100 ml capacity Measuring bottle, the water volume to the mark, shake.

6.18 Ethylenediamine tetraacetic acid disodium mixed test solution

Weigh 10.0 g of ethylenediaminetetraacetic acid disodium dihydrate (6.7) dissolved in water, dissolved in heat, cooled and added 10 ml Cresol red solution (6.17), the volume of water to 200 ml, shake. Stored in plastic bottles, refrigerated below 4°C , sealed can be saved Six months.

6.19 Ethylenediamine tetraacetic acid disodium mixed test solution

Ethylenediamine tetraacetic acid disodium mixed test solution (6.18) diluted with water 10 times that is the use of liquid, with the current allocation.

6.20 Hydroxylamine Hydrochloride Solution. $\rho = 20 \text{ g/L}$.

Weigh 2.0 g hydroxylamine hydrochloride (6.8), dissolved in water, water volume to 100 ml, with the current allocation.