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**HJ**

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

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Replacing GB/T 7482-1987

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**Water quality. Determination of fluoride. Visual colorimetry with  
zirconium alizarinsulfonate**

水质 氟化物的测定 茜素磺酸锆目视比色法

**(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 developed for the monitoring of fluoride in health and water and wastewater.

This standard specifies the visual colorimetric method for the determination of fluoride in drinking water, surface water, groundwater and industrial wastewater.

This standard amends the "Determination of Fluoride in Water Quality by Zirconium Sulfonic Acid Visual Colorimetric Method" (GB 7482-87), this standard First published in 1987, the original standard drafting unit was China Environmental Monitoring Station. This is the first revision.

The main revisions are as follows.

- Modified the preparation method of the zirconium sulfonate solution;
- Changed the amount of zirconium sulfonate acidic solution added.

The national environmental protection standard "Water quality approved and issued by the former National Environmental Protection Agency on March 14, 1987 since the implementation of this standard.

Determination of Fluoride The Zirconium Sulfate Sulfate Visual Colorimetric Method (GB 7482-87) was abolished.

Appendix A of this standard is a normative appendix.

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

This standard is mainly drafted by. China Environmental Monitoring Center, Liaoning Provincial Environmental Monitoring Center Station.

This standard was approved by the Ministry of Environmental Protection on September 27, 2009.

This standard has been implemented since November 1, 2009.

This standard is explained by the Ministry of Environmental Protection.

Water quality - Determination of fluoride - Zirconium sulphonate

## **1 Scope of application**

This standard specifies the visual colorimetric determination of zirconium sulfonate of fluoride in drinking water, surface water, groundwater and industrial wastewater.

This standard applies to the determination of fluoride in drinking water, surface water, groundwater and industrial wastewater.

When taking 50 ml sample and directly measuring the concentration of fluoride, the detection limit of this method is 0.1 mg/L, and the lower limit of determination is 0.4 mg/L.

The upper limit is 1.5 mg/L (high samples can be analyzed after dilution).

## **2 Principle of the method**

In an acidic solution, sodium limonate sulfonate and zirconium salt form a red complex, which can capture zirconium in the complex when fluoride ions are present in the sample.

Ions, producing colorless zirconium fluoride ions, releasing yellow sodium bismuth sulfonate, depending on the color of the solution from red to yellow Colorimetric

quantification.

### 3 Reagents and materials

The reagents used in this standard are analytically pure reagents that meet the national standards unless otherwise stated. The experimental water is newly prepared.

Ionic water or distilled water.

#### 3.1

Perchloric acid ( $\text{HClO}_4$ ). 70% to 72%.

#### 3.2

Hydrochloric acid ( $\text{HCl}$ ).  $\rho_{20} = 1.19 \text{ g/ml}$ .

#### 3.3

Sulfuric acid ( $\text{H}_2\text{SO}_4$ ).  $\rho = 1.84 \text{ g/ml}$ .

#### 3.4

Sodium arsenite ( $\text{NaAsO}_2$ ).  $\rho = 5 \text{ g/L}$ , weigh 0.5 g of sodium arsenite, dissolve in a small amount of water, and dilute to 100 ml.

**Note:** Sodium arsenite is highly toxic and prevents entry into the mouth.

#### 3.5

Fluoride standard stock solution.  $\rho = 100.0 \text{ } \mu\text{g/ml}$ , take sodium fluoride at  $105^\circ \text{C}$  for 2 h, after cooling in a desiccator, accurately Take 0.221 0 g, dissolve in water, transfer to a 1 000 ml volumetric flask, and dilute to the mark with water.

#### 3.6

Fluoride standard solution.  $\rho = 10.00 \text{ } \mu\text{g/ml}$ , take 10.00 ml of fluoride standard solution (3.5), in a 100 ml volumetric flask, with water Dilute to the mark.

### 3.7 Zirconium sulfonate acidic solution

#### 3.7.1

Zirconium silicate sulfonate solution. Weigh 0.3 g of zirconium oxychloride ( $\text{ZrOCl}_2 \cdot 8\text{H}_2\text{O}$ ) in a 100 ml beaker and dissolve it in 50 ml of water.

Move into a 1 000 ml volumetric flask. Another 0.7 g sodium decanoate ( $\text{C}_{14}\text{H}_{27}\text{O}_2\text{Na} \cdot \text{H}_2\text{O}$ ) is dissolved in 50 ml of water and shaken continuously.

Next, slowly inject the zirconium oxychloride solution. After shaking fully, place it for clarification.

### 3.7.2

Mixed acid solution. Measure 101 ml of hydrochloric acid (3.2) and dilute to 400 ml with water, and measure 33.3 ml of sulfuric acid (3.3).

With stirring, slowly add to 400 ml of water and, after cooling, combine the two acids.

### 3.7.3

Zirconium sulfonate acidic solution. Pour the mixed acid (3.7.2) into a volumetric flask containing zirconium sulfonate solution (3.7.1), with water Dilute to the mark and shake well. This solution can be used from red to yellow after about 2 hours. This solution is stored in the dark and stable for 6 months.

## 4 Instruments

4.1 50 ml with colorimetric tube.

### 4.2

Distillation unit, see A.5.1.

## 5 Interference and elimination

Pre-distillation is required when the interfering ions in the sample exceed the following concentrations. total alkalinity ( $\text{CaCO}_3$ ) 400 mg/L, chloride 500 mg/L, Sulfate.200 mg/L, aluminum 0.1 mg/L, phosphate 1.0 mg/L, iron 2.0 mg/L, turbidity 25 degrees, chroma 25 degrees.

### 6.1 Acquisition and preservation

The water sample of the fluoride was measured, and a polyethylene bottle was used for collecting and storing the sample.

### 6.2 Preparation of samples

- (1) If the water sample contains residual chlorine, add one drop (0.05 ml) of sodium arsenite solution per 0.1 mg of chlorine and mix to remove it.
- (2) If there are many interfering substances in water, and can not be directly measured by colorimetry, pre-distillation treatment can be carried out according to Appendix A.

### 7.1 Preparation of standard colorimetric series

Pipette 0.00, 0.50, 1.00, 2.00, 2.50, 4.00, 5.00, and 7.50 ml of fluoride standard solution (3.6) into 50 ml.

In the colorimetric tube, make up to volume with pure water. The fluoride content of this standard color column is. 0.00, 5.00, 10.00, 20.00, 25.00, 40.00, 50.00 and 75.00  $\mu\text{g}$ . Add