

# HJ

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

### HJ 677-2013

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**Water quality. Digestion of total metals. Nitric acid digestion  
method**

水质 金属总量的消解 硝酸消解法

**(English Translation)**

**Issued on: January 1, 1970**

**Implemented on: January 1, 1970**

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**Issued by: Ministry of Ecology and Environment**

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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 this standard is formulated to protect human health and standardize the pretreatment method of total metal in water.

This standard specifies the nitric acid digestion pretreatment method for the total amount of metal in water.

This standard is the first release.

Appendix A of this standard is an informative annex.

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

This standard is mainly drafted by. Ningbo Environmental Monitoring Center.

The standard verification unit. Shanghai Environmental Monitoring Center, Hangzhou Environmental Monitoring Center Station, Shaoxing Environmental Monitoring Center Station, Taizhou Environmental Monitoring Center Station, Ningbo Entry-Exit Inspection and Quarantine Bureau and Ningbo Environmental Monitoring Center.

This standard was approved by the Ministry of Environmental Protection on November 21, 2013.

This standard has been implemented since February 1, 2014.

This standard is explained by the Ministry of Environmental Protection.

Digestion of the total amount of water metal Warning. The digestion operation must be carried out in a fume hood. Wear protective gloves and other protective equipment as required to avoid contact with the skin.

Skin and clothes.

## 1 Scope of application

This standard specifies the nitric acid digestion pretreatment method for the total amount of metal in water.

This standard applies to the total amount of 20 metal elements in surface water, groundwater, domestic sewage and industrial wastewater.

Treatment, including silver (Ag), aluminum (Al), arsenic (As), beryllium (Be), barium (Ba), calcium (Ca), cadmium (Cd), cobalt (Co), Chromium (Cr), copper (Cu), iron (Fe), potassium (K), magnesium (Mg), manganese (Mn), molybdenum (Mo), nickel (Ni), lead (Pb), T (Ti), vanadium (V), zinc (Zn). Other metal elements are also valid for this method after verification.

## 2 Normative references

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

In this standard.

HJ/T 91 Surface Water and Wastewater Monitoring Technical Specifications HJ/T 164 Technical Specifications for Groundwater Environmental Monitoring HJ 493 Technical Regulations for Preservation and Management of Water Quality Samples

### 3 Terms and definitions

The following terms and definitions apply to this standard.

Total metal

Refers to the metal content determined after digestion of the unfiltered water sample.

### 4 Principle of the method

At  $95 \pm 5$  ° C, the metal element in the water sample is converted to a single high valence state by oxidation of nitric acid and hydrogen peroxide or it is converted into an inorganic compound that is easy to separate, the organic matter in the sample is destroyed, and the sample is uniformly clear.

### 5 Reagents and materials

Unless otherwise stated, the analysis uses the superior purification reagents that meet the national standards, and the experimental water is the second preparation of the new preparation.

Deionized water or sub-boiling distilled water, resistivity  $\geq 18 \text{ Mohm} \cdot \text{cm}$  (25 ° C).

#### 5.1

Nitric acid.  $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$ .

#### 5.2

Nitric acid.  $\rho(\text{HNO}_3) = 1.42 \text{ g/ml}$ , analytically pure.

#### 5.3

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

#### 5.4

Hydrogen peroxide.  $\omega(\text{H}_2\text{O}_2) = 30\%$ .

#### 5.5

Nitric acid solution. 1 1, prepared with concentrated nitric acid (5.2).

#### 5.6

Hydrochloric acid solution. 1 1 Prepared with concentrated hydrochloric acid (5.3).

5.7 0.45  $\mu\text{m}$  cellulose acetate filter.

Using the edge of the stainless steel tweezers coated with polyethylene film, immersed vertically into the hydrochloric acid solution (5.6) one by one, at least Soak for 12h. Rinse with distilled water until neutral, dry and seal for use.

## 6 Instruments and equipment

### 6.1

Temperature-controlled heating equipment. various heating devices capable of controlling the solution temperature in the range of  $95\pm 5^{\circ}\text{C}$

### 6.2

Filtering device.

### 6.3

Centrifugal separator. The speed can reach more than 3000rpm.

### 6.4

Common instruments and equipment used in general laboratories.

## 7.1 Sample collection and preservation

Water samples were collected and stored according to HJ/T 91, HJ/T 164, and HJ 493.

## 8 Digestion steps

Measure 50 ml of the mixed water sample in a 150ml beaker or conical flask, add 5 ml of concentrated nitric acid (5.1), and place it in temperature control.

On the heating equipment (6.1), cover the surface dish or small funnel, keep the temperature of the solution at  $95\pm 5^{\circ}\text{C}$ , and heat it for 30 minutes without boiling.

Remove the watch glass and evaporate until the solution is about 5 ml to stop heating. After cooling, add 5 ml concentrated nitric acid (5.1) and cover.

The upper watch glass is heated to reflux. If brown smoke is formed, repeat this step (add 5 ml of concentrated nitric acid each time), straight Until there is no more brown smoke, the solution is evaporated to about 5 ml.

After the solution is cooled, slowly add 3 ml of hydrogen peroxide (5.4), continue to cover the watch glass, and keep the solution temperature.

$95\pm 5^{\circ}\text{C}$ , heating until no more bubbles are generated. After the solution is cooled, continue to add hydrogen peroxide (5.4), each time 1 ml, Until only the fine bubbles or the general appearance does not change, remove the watch glass and continue heating until the solution volume evaporates to about 5ml.

After the solution is cooled, rinse the inner wall with an appropriate amount of water for at least 3 times, transfer to a 50 ml volumetric flask and dilute to volume.

**Note:** If there are particles in the sample, use a suction filter (6.2) 0.45 $\mu\text{m}$  acetate filter (5.7) for