# GB

NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

ICS 71.060.30

G 17

# GB/T 1626-2008

Replacing GB/T 1626-1988

# Oxalic acid for industrial use

工业用草酸

**Issued on: June 04, 2008** **Implemented on: December 01, 2008**

**Issued by: General Administration of Quality Supervision, Inspection and Quarantine of PRC;**

**Standardization Administration of PRC.**

# Table of Contents

# Oxalic acid for industrial use

## 1 Scope

This standard specifies the technical requirements, test methods, inspection rules, marking, packaging, transportation and storage of oxalic acid for industrial use.

This standard is applicable to the production, inspection and sales of industrial oxalic acid produced by the synthesis of producer gas and sodium hydroxide (hereinafter referred to as the synthesis method) or the oxidation of glucose with nitric acid (hereinafter referred to as the oxidation method).

Molecular formula: $H_2C_2O_4 \cdot 2H_2O$

Relative molecular mass: 126.07 (according to the international relative atomic mass in 2005)

## 2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 601-2002 Chemical reagent - Preparations of standard volumetric solutions

GB/T 602-2002 Chemical reagent - Preparations of standard solutions for impurity (ISO 6353-1:1982, NEQ)

GB/T 603-2002 Chemical reagent - Preparations of reagent solution for use in test method (ISO 6353-1:1982, NEQ)

GB/T 1250 Rules for expression and judgement of limiting values

GB/T 3049-2006 Chemical products for industrial use - General method for determination of iron content - 1,10-Phenanthroline spectrophotometric method (ISO 6685: 1982, IDT)

GB/T 6678-2003 General principles for sampling chemical products

# 6 Test method

Unless otherwise specified, only use reagents confirmed to be analytically pure and grade 3 water that meets the requirements of GB/T 6682-2008 in the analysis.

The standard titration solutions, preparations and products used in the analysis shall be prepared in accordance with GB/T 601-2002, GB/T 602-2002, GB/T 603-2002, when other requirements are not specified.

## 6.1 Determination of oxalic acid content

### 6.1.1 Method summary

Acid-base titration. Use phenolphthalein as an indicator; use sodium hydroxide standard titration solution to make titration; calculate the oxalic acid content.

### 6.1.2 Reagents

**6.1.2.1** Sodium hydroxide standard titration solution: c(NaOH) = 0.5 mol/L;

**6.1.2.2** Phenolphthalein indicator solution: 10 g/L.

### 6.1.3 Analytical procedures

Weigh 1 g of specimen, accurate to 0.002 g; place it in a 250 mL conical flask; add 30 mL of carbon dioxide-free water to dissolve the specimen; add 2 ~ 3 drops of phenolphthalein indicator solution; use sodium hydroxide standard titration solution to titrate, until a pale pink color appears; when this color does not fade in 30 s, it is the end point.

### 6.1.4 Result calculation

The mass fraction of oxalic acid (calculated as $H_2C_2O_4 \cdot 2H_2O$) is $w_1$; the value is expressed in %, calculated according to formula (1):

$$w_1 = \frac{V_1 \cdot c \cdot M}{m \times 1\,000} \times 100 \qquad \cdots\cdots (1)$$

Where:

$V_1$ - The volume value of the standard titration solution (6.1.2.1) of sodium hydroxide consumed by the sample, in milliliters (mL);

c - The exact value of the concentration of the sodium hydroxide standard titration solution, in moles per liter (mol/L);

a high-temperature furnace at 850 °C for 5 min. After cooling, add 10 mL of water and 2 mL of hydrogen peroxide to the residue. After a short time of boiling, add 1 mL of hydrochloric acid solution; then evaporate to dryness on a water bath. Add a small amount of water and 0.5 mL of hydrochloric acid solution to dissolve the residue; use water to wash it into a 50 mL colorimetric tube; add water to 25 mL (if the solution is turbid, filter it); use it as a specimen solution.

**6.2.4.2 Preparation of standard turbidity solution of sulfate radical**

Add 0.5, 0.7, 1.0, 1.5, 2.0, 3.0, 4.0, ... mL of sulfate standard solution to each evaporating dish; then add 0.5 mL of sodium carbonate solution, 10 mL of water, 2 mL of hydrogen peroxide, 1 mL of hydrochloric acid solution, respectively; evaporate it to dryness on a water bath; add a small amount of water and 0.5 mL of hydrochloric acid solution into each residue; dissolve the residue; use an appropriate amount of water to rinse it into a 50 mL colorimetric tube; add water to 25 mL, as the standard turbidity solution of sulfate radical.

**6.2.4.3 Determination**

Add 10 mL of barium chloride ethanol glycerol solution to the specimen solution and the sulfate standard turbidity solution; shake well; place for 30 minutes. In natural light or daylight, compare axially the specimen solution and the sulfate standard turbidity solution. Take the turbidity closest to the standard turbidity solution of sulfate as the test result. If the turbidity of the specimen solution is between two standard turbidity sulfate solutions, the turbidity of the standard turbidity sulfate solution with high turbidity is used as the test result.

**6.2.5 Result calculation**

For the mass fraction of sulphate (calculated as $SO_4$) $w_2$, the value is expressed in % and calculated according to formula (2):

$$w_2 = \frac{V_2 \times (c/1\ 000)}{m/10} \times 100 \qquad \cdots\cdots (2)$$

Where:

$V_2$ - The value of the volume of the sulfate standard solution in the sulfate standard turbidity solution, in milliliters (mL);

m - The value of the mass of the sample, in grams (g);

c - The value of the concentration of the sulfate standard solution, in milligrams per milliliter (mg/mL) [c = 0.1].

Take the arithmetic mean of the two parallel determination results as the determination result. The absolute difference between the two parallel

**6.5.1.2.4** Iron (Fe) standard solution: 0.1 mg/mL.

**6.5.1.3 Apparatus**

**6.5.1.3.1** Flame atomic absorption spectrometer: It is equipped with iron hollow cathode lamp and flame atomizer, which meets the requirements of GB/T 9723;

**6.5.1.3.2** Electric furnace: temperature adjustable.

**6.5.1.4 Instrument operating conditions**

The operating conditions of the instrument recommended by this standard: wavelength 248.3 nm, slit width 0.2 nm, lamp current 2 mA.

**6.5.1.5 Analytical procedures**

**6.5.1.5.1 Drawing of working curve**

Take 0, 1.0, 2.0, 3.0, 4.0, 5.0 mL of the iron standard solution, respectively, in six 100 mL volumetric flasks, respectively; add 5 mL of hydrochloric acid; use water to dilute it to the mark; shake well, as an iron standard working solution. Each millimeter of solution contains 0, 1, 2, 3, 4, 5 μg of iron.

Adjust to the best condition according to the performance of the instrument. Under the given instrument operating conditions, determine the absorbance of the iron standard working solution; take the absorbance as the ordinate and the corresponding iron content (μg/mL) in the iron standard working solution as the abscissa, to draw the working curve. The linear range of the working curve reaches 5 μg/mL.

**6.5.1.5.2 Determination of samples**

Weigh 25 g of the specimen, accurate to 0.01 g; place it in a 100 mL quartz beaker; use a watch glass to cover it; place it on an electric furnace and heat it at low temperature, until the oxalic acid is completely decomposed and the gas escapes. Remove and cool to room temperature; add 1.25 mL of hydrochloric acid; use water to rinse it into a 25 mL volumetric flask; dilute to the mark; shake well, as a specimen solution.

Measure the absorbance of the specimen solution according to the operating conditions for drawing the working curve; find out the iron content in the specimen on the working curve.

**6.5.1.6 Result calculation**

The mass fraction of iron (calculated as Fe), $w_3$, is expressed in % and calculated according to formula (3):

m - The value of the mass of the sample, in grams (g).

Take the arithmetic mean of the two parallel determination results as the determination result.

When the mass fraction of iron is less than 0.003%, the absolute difference between the two parallel determination results is not more than 20% of the arithmetic mean of the two determination values.

When the mass fraction of iron is 0.003 ~ 0.01%, the absolute difference between the two parallel determination results is not more than 10% of the arithmetic mean of the two determination values.

### 6.6 Determination of chloride

#### 6.6.1 Method summary

In an acidic solution, chloride and silver nitrate form a white precipitate of silver chloride, whose turbidity is compared with the standard turbidity solution.

#### 6.6.2 Reagents

**6.6.2.1** Nitric acid solution: 1 + 2;

**6.6.2.2** Silver nitrate solution: 17 g/L;

**6.6.2.3** Chloride (Cl) standard solution: 0.1 mg/mL.

#### 6.6.3 Analytical procedures

##### 6.6.3.1 Preparation of chloride standard turbidity solution

According to different levels of chloride content index values, draw 0.05, 0.02, 0.40, 1.00 mL of chloride standard solutions, respectively, into a 50 mL colorimetric tube; then add 5 mL of nitric acid solution; add water to 25 mL. This solution is used as the standard turbidity solution of chloride.

##### 6.6.3.2 Determination

Weigh 1 g of the specimen, accurate to 0.001 g; place it in a 50 mL colorimetric tube; use a small amount of water to dissolve it; then add 5 mL of nitric acid solution; add water to 25 mL. This solution is used as the specimen solution.

Add 1 mL of silver nitrate solution to the colorimetric tube containing the specimen solution and the chloride standard turbidity solution; mix well; place it for 15 minutes; compare the sample solution and the chloride standard turbidity solution axially. The turbidity of the specimen solution shall not be deeper than the turbidity of the chloride standard turbidity solution.

**This is an excerpt of the PDF (Some pages are marked off intentionally)**



**------ The End ------**