

Chemical Professional Standard of the People's Republic of China

HG/T 5725-2020

High purity reagent— Cupric sulfate pentahydrate

高纯试剂 五水合硫酸铜（硫酸铜）

(English Translation)

(Manuscript for approval)

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Foreword

SAC/TC63/SC3 is in charge of this English translation. In case of any doubt about the contents of English translation, the Chinese original shall be considered authoritative.

This standard was drafted according to the rules given in the GB/T 1.1-2009.

This standard was proposed by China Petroleum and Chemical Industry Federation.

This standard was prepared by the Chemical Reagent Sub-technical Committee of National Chemical Standardization Technical Committee (SAC/TC63/SC3).

High purity reagent—Cupric sulfate pentahydrate

1 Scope

This standard specifies the description, specifications, test, inspection regulations, packaging and sign of high purity reagent—cupric sulfate pentahydrate.

This standard is applicable to the test of high purity reagent—cupric sulfate pentahydrate.

Molecular formula: $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$

Relative molecular weight: 249.68 (According to the international relative atomic mass in 2018)

CAS number: 7758-99-8

2 Normative References

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 601 *Chemical reagent—Preparations of reference titration solutions*

GB/T 602 *Chemical reagent—Preparations of standard solutions for impurity*

GB/T 603 *Chemical reagent—Preparations of reagent solutions for use in test methods*

GB/T 609 *Chemical reagent—General method for the determination of total nitrogen*

GB/T 6682 *Water for analytical laboratory use—Specification and test methods*

GB/T 9723-2007 *Chemical reagent—General rule for flame atomic absorption spectrometric analysis*

GB/T 9724 *Chemical reagent—General rule for the determination of pH*

GB/T 9738 *Chemical reagent—General method for the determination of water insoluble matter*

GB 15346 *Chemical reagent—Packaging and marking*

GB/T 23942-2009 *Chemical reagent—General rules for inductively coupled plasma atomic emission spectrometry*

HG/T 3921 *Chemical reagent—Regulations for sampling and acceptance*

JJG 694 *Atomic Absorption Spectrophotometers*

3 Description

This reagent is blue crystal, weathered in dry air, and soluble in water.

4 Specifications

The specifications of cupric sulfate pentahydrate are shown in Table 1.

Table 1 The specifications of cupric sulfate pentahydrate

Items	Specifications
Assay ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), w/%	≥ 99.5
pH (50 g/L, 25 °C)	3.5~4.5
Water insoluble matter, w/%	≤ 0.005
Chloride (Cl), w/%	≤ 0.0005
Total nitrogen (N), w/%	≤ 0.001
Arsenic (As), w/%	≤ 0.0003
Sodium (Na), w/%	≤ 0.001
Magnesium (Mg), w/%	≤ 0.0005
Aluminum (Al), w/%	≤ 0.0005
Potassium (K), w/%	≤ 0.0005
Calcium (Ca), w/%	≤ 0.0005
Chromium (Cr), w/%	≤ 0.0002
Manganese (Mn), w/%	≤ 0.0001
Iron (Fe), w/%	≤ 0.001
Cobalt (Co), w/%	≤ 0.0002
Nickel (Ni), w/%	≤ 0.0005
Zinc (Zn), w/%	≤ 0.0005
Cadmium (Cd), w/%	≤ 0.0001
Lead (Pb), w/%	≤ 0.0005

5 Tests

5.1 Warning

Some reagents used in this test method are toxic or corrosive. In some testing procedures the operator should take appropriate safety and health measures, if not, it can lead to hazardous situations.

5.2 General provisions

Unless otherwise specified in this clause, the reference titration solutions, standard solutions, reagent solutions used in this clause are prepared according to GB/T 601, GB/T 602, and GB/T 603. Unless otherwise specified in this clause, water for analytical laboratory use shall comply with grade 3 water specification in GB/T 6682. Unless otherwise specified in this clause, samples are weighed accurate to 0.01 g. If the concentration of solutions is expressed in “%”, it refers to mass fraction.

5.3 Assay

Weigh, to the nearest 0.0001 g, 0.8 g of sample. Transfer it to an iodine flask, and dissolve

it in 60 mL of water. Add 5 mL of sulfuric acid solution (20%) and 3 g of potassium iodide, mix well, place in the dark for 10 min. Titrate with sodium thiosulfate reference titration solution [$c(\text{Na}_2\text{S}_2\text{O}_3)=0.1 \text{ mol/L}$]. Add 3 mL of starch indicator solution (10 g/L) as the endpoint is approached and continue titrating until the blue color of the solution disappears. Perform a blank test in parallel with the sample determination.

The mass fraction w of cupric sulfate pentahydrate is calculated according to Formula (1):

$$w = \frac{(V - V_0)cM}{m \times 1000} \times 100\% \quad \dots\dots\dots(1)$$

Where:

V — the volume of the sodium thiosulfate reference titration solution consumed by sample, in milliliters (mL);

V_0 — the volume of sodium thiosulfate reference titration solution consumed by blank test, in milliliters (mL);

c — the concentration of sodium thiosulfate reference titration solution, in moles per liter (mol/L);

M — the molar mass of cupric sulfate pentahydrate, in grams per mole (g/mol) [$M(\text{CuSO}_4 \cdot 5\text{H}_2\text{O})=249.68 \text{ g/mol}$];

m — the mass of sample, in grams (g).

5.4 pH

Determined as specified in GB/T 9724.

5.5 Water insoluble matter

Weigh 30 g of sample, dissolve it in 100 mL of boiling water, cool to room temperature, and determined as specified in GB/T 9738.

5.6 Chloride

5.6.1 Preparation of chloride-free cupric sulfate pentahydrate solution

Weigh 8 g of sample, dissolve it in appropriate amount of water, add 8 mL of nitric acid solution (25%) and 4 mL of silver nitrate solution (17 g/L), dilute to 100 mL, mix well, place in the dark for 4 h, and filter after clarification.

5.6.2 Test method

Weigh 2 g of sample, dissolve it in 25 mL of water (filter if necessary), add 2 mL of nitric acid solution (25%), 2.5 mL of glycerol solution (1+4), mix well, add 1 mL of silver nitrate solution (17 g/L), mix well and stand for 15 min. Any turbidity in the test solution shall not exceed that of the standard turbidity solution.

The preparation of the standard turbidity solution: take 25 mL of chloride-free cupric sulfate pentahydrate solution and standard solution containing 0.01 mg of chloride (Cl), add 3 mL of water, 2.5 mL of glycerol solution (1+4), and mix well. Place with the same volume of test solution for 15 min at the same time, and then compare the turbidity.

5.7 Total nitrogen

Weigh 2 g of sample, transfer it to a Kjeldahl flask, add 140 mL of water to dissolve it, add 7 mL of sodium hydroxide solution (320 g/L), 1.0 g of Devarda's alloy, stand for 1 h, and determined as specified in GB/T 609. Any color produced in the test solution shall not exceed that of the standard colorimetric solution.

The preparation of the standard colorimetric solution: take a standard solution containing 0.02 mg of nitrogen (N), dilute to 142 mL, add 5 mL of sodium hydroxide solution (320 g/L), and process the same with the same volume of test solution.

5.8 Arsenic

Weigh 0.5 g of sample, dissolve it in 5 mL of water, add 3 mL of acetic acid (glacial acetic acid) and 2 g of potassium iodide, cover with a watch-glass, and stand for 5 min. Add 0.2 g of ascorbic acid to dissolve, dilute to 40 mL, add 6 mL of hydrochloric acid, and mix well. Add stannous chloride solution (400 g/L) dropwise until the solution turns colorless, mix well, and stand for 10 min. Add 2.5 g of arsenic-free zinc, immediately plug the arsenic absorption tube which pre-installed with lead acetate cotton and mercury bromide test paper, and keep it at 25 °C~30 °C in the dark for 1 h~1.5 h. Any brown-yellow color of the mercury bromide test paper shall not exceed that of the standard colorimetric test paper.

The standard colorimetric test paper is to take a standard solution containing 0.0015 mg of arsenic (As), and process the same as the sample at the same time.

5.9 Sodium

5.9.1 Reagents, materials and apparatus

In accordance with GB/T 9723-2007, Clause 5 and Clause 6.

5.9.2 Apparatus conditions

Light source: sodium hollow cathode lamp.

Wavelength: 589.0 nm.

Flame: acetylene-air.

5.9.3 Test method

Weigh 10 g of sample, dissolve it in water, add 5 mL of hydrochloric acid solution (20%), and dilute to 100 mL. Take four 20 mL portions, determine according to the provisions in GB/T 9723-2007, 7.2.2, and the results are calculated according to the provisions in GB/T 9723-2007, 7.2.3.

5.10 Magnesium, Aluminum, Calcium, Chromium, Manganese, Iron, Cobalt, Nickel, Zinc, Cadmium

5.10.1 Reagents, materials and apparatus

In accordance with GB/T 23942-2009, Clause 5 and Clause 6.

5.10.2 Apparatus conditions

Recommended wavelengths: magnesium 279.553 nm, aluminum 396.152 nm, calcium 393.366 nm,

chromium 267.716 nm, manganese 257.610 nm, iron 238.204 nm, cobalt 228.616 nm, nickel 231.604 nm, zinc 206.200 nm, cadmium 214.438 nm.

Incident power: 1100 W.

Observation height: 12 mm.

Argon flow: 19 L/min of carrier gas, 0.2 L/min of auxiliary gas.

Solution extraction rate: 1.2 mL/min.

Analysis time: washing time 15 s, exposure time 10 s.

5.10.3 Test method

Weigh 10 g of sample, dissolve it in water, add 10 mL of hydrochloric acid solution (20%), transfer it to a 100 mL volumetric flask, dilute to the mark, and mix well. To four 100 mL volumetric flask, each add 20 mL the above test solution. Determine according to the provisions in GB/T 23942-2009, 7.3.3, and the results are calculated according to the provisions in GB/T 23942-2009, 7.3.4.

5.11 Potassium

5.11.1 Reagents, materials and apparatus

In accordance with GB/T 9723-2007, Clause 5 and Clause 6.

5.11.2 Apparatus conditions

Light source: potassium hollow cathode lamp.

Wavelength: 766.5 nm.

Flame: acetylene-air.

5.11.3 Test method

As specified in 5.9.3.

5.12 Lead

5.12.1 Graphite furnace atomic absorption spectrometry (arbitration method)

5.12.1.1 Reagents, materials and apparatus

Water for analytical laboratory use shall comply with grade 2 water specification in GB/T 6682. Hydrochloric acid and nitric acid shall be high-purity reagents, or be obtained by sub-boiling distillation with analytical reagents.

Apparatus: Graphite furnace atomic absorption spectrophotometer, which as specified in JJG 694.

5.12.1.2 Apparatus conditions

Light source: lead hollow cathode lamp.

Wavelength: 283.3 nm.

Spectral passband: 0.5 nm.

Lamp current: 9 mA.

Drying temperature and time: 120 °C, 30 s.

Ashing temperature and time: 500 °C, 20 s.

Atomization temperature and time: 1400 °C, 5 s.

Injection volume: 20 μL.

5.12.1.3 Preparation of lead standard solution (1 μg/mL)

Take 1.00 mL of lead standard solution (0.1 mg/mL), place in a 100 mL volumetric flask, add 10 mL of nitric acid solution (0.1 mol/L), dilute to the mark with water, and mix well. Prepare before use.

5.12.1.4 Test method

Weigh 2 g of sample, dissolve it in water, add 2 mL of hydrochloric acid solution (20%), and dilute to 100 mL. Take four 10 mL portions, one without standard, add 1 μg, 2 μg and 4 μg lead standard solution to the other three portions respectively, dilute with water to 100 mL, and mix well. Prepare a blank test solution in parallel with the sample determination. The standard curve of standard addition method is drawn with the concentration of standard solution as the abscissa and the absorbance of corresponding lead elements to be measured as the ordinate. The absolute value of the intersection of the inverse extension of the curve and the abscissa is the concentration of lead in the sample solution.

5.12.2 Flame atomic absorption spectrometry

5.12.2.1 Reagents, materials and apparatus

In accordance with GB/T 9723-2007, Clause 5 and Clause 6.

5.12.2.2 Apparatus conditions

Light source: lead hollow cathode lamp.

Wavelength: 283.3 nm.

Flame: acetylene-air.

5.12.2.3 Test method

Weigh 25 g of sample, dissolve it in water, add 5 mL of hydrochloric acid solution (20%), and dilute to 100 mL. Take four 20 mL portions, determine according to the provisions in GB/T 9723-2007, 7.2.2, and the results are calculated according to the provisions in GB/T 9723-2007, 7.2.3.

6 Inspection regulations

Sampling and acceptance shall be carried out as specified in HG/T 3921.

7 Packaging and sign

Pack, store and transport as specified in GB 15346, and give signs, of which:

— Packaging unit: Category 4;

— Inner packaging forms: NB-4, NBY-4, NB-5, NBY-5, NB-7, NB-8, NB-10, NB-11, NB-13, NB-15;

—— Isolation materials: GC-2, GC-3, GC-4;

—— Outer packaging forms: WB-1, WB-2, WB-3.

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