RESOLUTION OENO 25/2000

INTERNATIONAL OENOLOGICAL CODEX

COPPER SULFATE, PENTAHYDRATE

Copper (II) Sulfate, Pentahydrate

$CuSO_4, 5H_2O$ = 249.68

1. OBJECTIVE, ORIGIN AND SCOPE OF APPLICATION

Copper sulfate is used in processing wines possessing so-called reduction "tastes" due to the presence of hydrogen sulfide or volatile thiols.

The copper sulfides thus formed precipitate and should be removed from wine.

This product must be used in compliance with copper sulfate pentahydrate limiting quantities; furthermore, there are statutory limits restricting the copper content in wines.

2. LABELING

The label should indicate product concentration, even when used in mixtures, as well as its safety and storage conditions.

3. PROPERTIES

Blue crystals which are minimally efflorescent in dry air.

4. COMPOSITION

Minimum of 99 pp 100 CuSO4,$5H_2O$.

5. IDENTIFYING CHARACTERISTICS

Melting point: 110 °C with water loss.

An aqueous solution with ammonium hydroxide (R) produces a dark blue copper tetramine compound. A solution acidified with hydrochloric acid and a barium chloride solution (R) produces a white barium sulfate precipitate.

Certified in conformity Paris, 23th June 2000
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET

© OIV 2000

6. SOLUBILITY

Water at 20 °C	286 g/l
Methanol	15.6 g/l
Insoluble in ethanol	(95% alcohol by volume)

7. TESTS

7.1. Preparing the Solution for Tests

Dissolve 10 g of the substance in water and fill to 50 ml.

7.2. Appearance of the Test Solution

The test solution must be clear.

7.3. Iron

Place 2 ml of the solution prepared for tests under paragraph 7.1 in a decanting glass and add 8 ml of water, 10 ml of 6M hydrochloric acid (R) and 10 ml of 4-methylpentane-2-one. Shake vigorously for 3 minutes. After letting the mixture settle, decant the organic phase in a second decanting glass. Add 10 ml of water then shake vigorously again for 3 minutes. Separate out the aqueous phase and perform the test in the following manner:

Add 2 ml of citric acid solution (20 g of citric acid/100 ml), 0.10 ml of concentrated thioglycolic acid (HS-CH_2-COOH) and a small amount of 6M ammonium hydroxide (10-10.4 g NH_3/100 ml) to the aqueous phase until an alkaline reaction is triggered. Dilute with water until a total volume of 20 ml is reached. After 5 minutes, the sample should not be more intensely colored than the test carried out using the comparison solution described below.

7.3.1. Preparation of the Comparison Solution

Iron (III) and ammonium sulfate solution 1

Dissolve 0.702 g of ammonium sulfate and iron (III) in 1.20 ml of 6M hydrochloric acid

Certified in conformity Paris, 23th June 2000
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET

© OIV 2000

and fill to 100 ml with water.

Iron (III) and ammonium sulfate solution 2

Take 7 ml of ammonium sulfate and iron (III) (Par. 7.3.1.1) and fill to 100 ml with water.

1 ml of solution 2 corresponds to 10 µg of Fe(III).

7.3.2. Test Using the Comparison solution

The comparison solution should be prepared prior to use in the following manner:

Take 1 ml of ammonium sulfate and iron (III) solution (2) and process in the same way as for the substance test.

N.B. : Iron content can also be analyzed by atomic absorption spectometry, using the method described in the Compendium.

The iron content limit is 100 mg/kg.

7.4. Nickel

Add 2 ml of concentrated hydrochloric acid (R) and 1 ml of concentrated nitric acid (R) to the aqueous phase from paragraph 7.3. After evaporating the solution, dissolve the residue in 1 ml of 6M nitric acid (R) and 19 ml of water. Dilute 1 ml of this solution to a total volume of 10 ml. To 2.50 ml of this dilute solution, add 6 ml of water (R), 5 ml bromine solution (R), 7 ml of 6M ammonium hydroxide solution and 3 ml of dimethylgloxime solution in a concentration of 100 g in 100 ml of 96% ethanol by volume. The solution should not exhibit any change after one minute when compared to a "blank" sample.

Nickel content can also be determined using the atomic absorption photometry method described in the Annex.

7.5. Chlorides

Dilute 25 ml of the solution prepared for tests under paragraph 7.1 with 10 ml of water. After adding 8 ml of 6M sodium hydroxide, bring to a boil and heat the mixture in a 100 °C water bath until the precipitate has been completely deposited. After cooling, dilute with water to obtain a total volume of 50 ml. Add 6 ml of water to 4 ml of filtrate and conduct the following test: add 1 ml of 6M nitric acid (R) and 1 ml 0f 0.1M silver nitrate (R). Shake the sample after 5 minutes. There should be no more clouding than that seen in the control test conducted with the comparison solution.

(Limiting value : 100 mg/kg.)

Certified in conformity Paris, 23th June 2000
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET

© OIV 2000

7.5.1. Preparation of the Comparison Solution

Dilute 4 ml of 0.1M sodium chloride solution (23.4 ml/100 ml) with water to yield a total volume of 100 ml. 1 ml corresponds to 142 µg Cl-. Prepare the solution just before use.

7.5.2. Control Test with the Comparison Solution

Take 1 ml of the sodium chloride solution (Par. 7.5.1) and proceed in the same manner as for the test on the substance.

7.6. Lead

Using the technique explained in the Compendium, determine the lead content in the test solution (Par. 7.1). (Lead content should be less than 5 mg/kg).

7.7. Mercury

Using the technique explained in the annex, determine the mercury content in the test solution (Par. 7.1). Mercury content should be less than 1 mg/kg.

7.8. Arsenic

Using the technique explained in the annex, determine the arsenic content in the test solution (Par. 7.1). (Arsenic content should be less than 3 mg/kg).

7.9. Quantitative Analysis

Weigh exactly 0.50 g of the substance and dissolve in 20 ml of water. Add 5 ml 6M acetic acid and 2 g potassium iodide. Titrate with a 0.1M sodium thiosulfate solution in the presence of starch (R).

1 ml of a 0.1M sodium thiosulfate solution which corresponds to 6.354 mg of Cu(II), or, if the result is expressed in terms of substance, to 24.97 mg of $CuSO_4{,}5H_2O$.

8. STORAGE

Copper sulfate should be stored in a dry place in hermetically sealed containers.

Certified in conformity Paris, 23th June 2000
The Director General of the OIV
Secretary of the General Assembly
Georges DUTRUC-ROSSET

© OIV 2000