

## RESOLUTION OIV-OENO 413-2011

### MONOGRAPH ON COPPER CITRATE

THE GENERAL ASSEMBLY

IN VIEW of article 2, paragraph 2 iv of the Agreement of 3 April 2001, by which the International Organisation of Vine and Wine was founded,

CONSIDERING resolution OENO 1/2008 adopted by the OIV in 2008 concerning the treatment of wine with copper citrate to remove bad tastes and odours due to hydrogen sulphide and possible derivatives

Following a proposal made by the "Specifications of oenological products" experts group

HAS HEREBY DECIDED to add to the international Oenological Codex the following monograph:

#### COPPER CITRATE x 2.5-HYDRATE

Copper citrate hemipentahydrate

$Cu_2C_6H_4O_7 \times 2.5 H_2O$

CAS-number: 10402-15-0

molecular weight: 360 g/mol

#### 1. Subject, origin and field of application

Copper citrate is applied for the treatment of wine to remove fermentation and storage related off-flavours (sulphide off-flavours, flavours caused by reduction reactions, flavours caused by the presence of hydrosulfuric acid and mercaptans).

The copper sulphide formed during the treatment precipitates in wine as it is a very poorly soluble compound and can be separated by filtration.

Addition to the wine can be made directly or preferably by means of bentonite as a carrier material. The purity requirements of Codex resolution OENO 11/2003 apply for the bentonite used.

The addition to the wine is limited (resolution OENO 1/2008), legally permitted limits of copper contents in wine must be respected.

## 2. Labelling

The product concentration, the batch number, the date of expiry, safety warnings and storage conditions have to be indicated on the label.

## 3. Appearance

Light green to light blue granulated crystals.

## 4. Content (Assay)

Minimum of 98 % copper citrate x 2.5-hydrate.

With products on carrier material, a minimum of 2 % copper citrate x 2.5-hydrate.

## 5. Identification

Copper citrate dissolved in 10 % nitric acid reacts with ammonium hydroxide (R) to form a dark blue complex of tetramine copper.

Heating to dryness at more than 180 °C leads to the carbonization of the citrate.

## 6. Solubility

Less than 0.05 g/L in water at 20 °C, in methanol and in ethanol.

Approx. 250 g/L in 10 % HCl (R).

Approx. 140-150 g/L in 10 %  $HNO_3$  (R).

## 7. Tests

### 7.1. Preparation of the test solution

Dissolution of 10 g copper citrate in 100 mL 10 %  $HNO_3$  (R).

### 7.2. Appearance of the test solution

The solution is light blue.

### 7.3. Iron

Determination by atomic absorption spectrophotometry (AAS); content below 200

mg/kg.

Remark:

The method described in the OIV resolution (OENO 25/2000) leads to a dissolving-out of the iron in the test solution by shaking with 4-methylpentan-2-one and transfer into the organic phase. The iron concentration in the organic phase is not determined. The analysis is merely to test whether the second aqueous phase still contains iron.

By means of AAS, the iron content can be measured directly and precisely.

With a maximum of 200 mg iron per kilogramme of copper citrate and a treatment with at the most 1 g copper citrate per 100 litres of wine, the increase of the iron concentration in wine amounts to a maximum of 0.002 mg per litre and is thus negligible.

## **7.4. Nickel**

Determination by atomic absorption spectrophotometry (AAS); content below 5 mg/kg.

## **7.5. Chloride**

Add 1 mL silver nitrate solution 0.1 M to 4 mL of the test solution. After 5 minutes mix the sample.

The turbidity must not exceed that of the test conducted with the reference solution.

Reference solution:

Dilute 4 mL sodium chloride solution 0.1 M with water to a volume of 100 mL.

Blank test:

1 mL of the freshly prepared reference solution is used instead of the test solution. Proceed in the same way as in the test mentioned above.

Remark:

A pretreatment of the test solution is not necessary, since it is nitric acid.

## **7.6. Lead**

Determination by atomic absorption spectrophotometry (AAS); content below 5 mg/kg.

## **7.7. Mercury**

Determination by atomic absorption spectrophotometry (AAS); content below 1 mg/kg.

## 7.8. Arsenic

Determination by atomic absorption spectrophotometry (AAS); content below 3 mg/kg.

## 7.9. Assay

Pour 1 mL test solution, 20 mL water, 2 mL acetic acid 6 M, 2 g potassium iodide and 2 mL starch solution (R) into a titration vessel. Titration is conducted with 0.1 M sodium thiosulphate solution up to the colour change.

A consumption of 1 mL sodium thiosulphate solution corresponds to 6.354 mg Cu(II) or to 18 mg expressed as copper citrate.

## 8. Storage

Copper citrate must be stored dry, protected from odours in hermetically closed packaging.