



## OIV-OENO 644-2020 Monograph on calcium sulfate

THE GENERAL ASSEMBLY,

IN VIEW OF THE ARTICLE 2, paragraph 2 b) ii of the Agreement of 3<sup>rd</sup> April 2001 establishing the International Organisation of Vine and Wine,

CONSIDERING the work of the "Specifications of Oenological Products" Expert Group during its March 2014 session,

CONSIDERING the oenological practice II.2.1.3.1.1.1 CALCIUM SULPHATE in chapter 2 'musts' (OIV-OENO 583-2017) already adopted by the OIV,

DECIDES, at the proposal of Commission II "Oenology", to add the following monograph in chapter 1 of the *International Oenological Codex*:

### Monograph on calcium sulfate

$CaSO_4 \times 2H_2O$  (Dihydrate)

CAS Number 10101-41-4

#### 1. Objective and scope of application

This product is used for must acidification in the production of liqueur wines. Calcium sulfate added reacts with tartrate ions of the must producing insoluble calcium tartrate and releasing ion sulfate in the must. These facts originate modifications in ions equilibria that liberates proton ions and reduces the pH without increasing the titratable acidity.

#### 2. Labeling

The label should indicate the nature of calcium sulfate, batch number and the storage and safety requirements.

#### 3. Stoichiometric composition

$CaSO_4$ :79.1%

$H_2O$ :20.9%

#### 4. Properties

Calcium sulfate dihydrate exists as a white amorphous powder. Not to be confused

with the anhydrous form which is very hygroscopic and sets in contact with must.

## 5. Solubility

Slightly soluble in water and soluble in hydrochloric, sulphuric and nitric acid solutions.

## 6. Tests

### 6.1. Desiccation losses

Free water: Weigh 50 g of calcium sulphate in a dish. Place it in an oven at 40 °C until constant weight. Weight loss should not exceed 2 %.

Free and bonded water: Place another sample in an oven at 200 ° C during 4 h. Total weight loss should not exceed 23 %.

### 6.2. Preparing the Solution for Tests

Weigh 10 g of calcium sulphate. In a 500 ml erlenmeyer flask which can be hermetically sealed, add 200 ml of tartaric acid solution at 5 g/L per litre and bring to pH 3 with HCl 0.1 N. Put this in a magnetic mixer, sprinkle gently the calcium sulphate and mix for 1 hour at a temperature of  $20 \pm 2^{\circ}\text{C}$ . Allow to settle and filter by eliminating the first 50 ml of filtrate. Collect at least 100 ml of clear liquid.

### 6.3. Lead

Using the technique described in the Compendium, analyse quantitatively the lead in the solution prepared for testing (Par. 6.2). Lead content in calcium sulfate should be less than 2 mg/kg.

### 6.4. Mercury

Using the technique described in the Compendium, analyse quantitatively the mercury in the solution prepared for testing (Par. 6.2). Mercury content in calcium sulfate should be less than 1 mg/kg.

### 6.5. Arsenic

Using the technique described in the Compendium, analyse quantitatively the arsenic in the solution prepared for testing (Par. 6.2). Arsenic content in calcium sulfate should be less than 3 mg/kg.

### 6.6. Iron

Using the technique described in the Compendium, analyse quantitatively the iron in the solution prepared for testing (Par. 6.2). Iron content should be less than 200 mg/kg.

### 6.7. Quantitative Analysis

Any method of analysis included in the Compendium could be used. In the case of using the gravimetric method OIV-MA-AS321-05A, use the following procedure. Weigh 250 milligrams of the sample dried at 40 °C with a precision of 1 mg and dissolve it in 10 mL HCl 1M. Take 5 mL of this solution and add 0,5 mL of HCl 2 M and 1,5 mL of a solution of BaCl<sub>2</sub> 400 g/L. Stir with a glass stirrer; rinse the stirrer with a little distilled water and leave to stand for 5 min. Centrifuge for 5 min at 3.000 rpm, then carefully decant the supernatant liquid. Wash the barium sulfate precipitate as follows: add 10 mL hydrochloric acid 2 M, place the precipitate in suspension and centrifuge for 5 min at 3.000 rpm, then carefully decant the supernatant liquid. Repeat the washing procedure twice as before using 15 mL distilled water each time. Quantitatively transfer the precipitate, with distilled water, into a tared platinum capsule and place over a water bath at 100°C until fully evaporated. The dried precipitate is calcined several times briefly over a flame until a white residue is obtained. Leave to cool in a desiccator and weigh:

### Calculations

Content of calcium sulfate dihydrate in the product (%) =  $p \times 0.59021$

Where  $p$  is the measured weight of BaSO<sub>4</sub> in mg.

If other method of analysis of sulphates included in the Compendium is used to analyse the initial solution of calcium sulfate prepared for quantitative analysis:

Content of calcium sulfate dihydrate in the product (%) =  $c \times 3.9522 \cdot 10^{-3}$

Where  $c$  is the concentration of sulfates in mg/L of K<sub>2</sub>SO<sub>4</sub>

The wine-making product must contain a minimum of 90 pp 100 calcium sulfate.

## 7. Storage

Calcium sulfate should be stored in a dry place in hermetically sealed containers away from volatile elements it could adsorb.