# RESOLUTION OIV-OENO 617-2019

# UPDATE OF MONOGRAPH ON COLLOIDAL SILICON DIOXIDE AND RELEVANT SHEETS OF THE INTERNATIONAL CODE OF OENOLOGICAL PRACTICES

*WARNING: this resolution repeals the following resolution:*
- *OIV-OENO 44-2000*

THE GENERAL ASSEMBLY,

IN VIEW of article 2, paragraph 2 ii of the Agreement of 3rd April 2001 establishing the International Organisation of Vine and Wine,

IN VIEW of the work of the "Specifications of Oenological Products" expert group,

DECIDES, concomitantly with the modifications proposed for the monograph COEI-1-DIOSIL of the International Oenological Codex, to modify the relevant sheet of the International Code of Oenological Practices, and specifically the relevant sheets: 2.1.10, 2.3.7, 3.2.1, 3.2.4,

DECIDES to replace the monograph COEI-1-DIOSIL of the International Oenological Codex with the following modifications:

# SILICON DIOXIDE IN COLLOIDAL SOLUTION, IN AQUEOUS DISPERSION OR IN DRY POWDER FORM

## 1. Objective, origin and scope of application

Colloidal silicon dioxide solutions are aqueous dispersions of silicon dioxide particles which are hydroxylated on the surface and are, therefore, negatively charged.

Silica gel is the dried powder form of silicon dioxide

These preparations are used to clarify wines and are associated with protein-based clarifying agents.

## 2. Labeling

The label should indicate the silicon dioxide concentration (for solutions) and its



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safety and storage conditions.

# 3. Properties

Depending on the manner in which they are prepared, acidic solutions are obtained, or alkaline solutions containing sodium ions expressed as Na2O. Alkaline solutions are most often used.

Colloidal silicon dioxide solutions are free from organic compounds.

Their concentration as determined by drying at 110 °C is always equal to or greater than 15 % (m/m) and is most often between 15 and 30 %.

The density of colloidal silicon dioxide solutions at 20 °C (□20°C) is given as a function of the concentration C (m/m) by the equation:

□20°C = □20°C (water) x 1/(1-0.0056C)
□20°C (water) = density of water at 20 °C = 0.998203.

These preparations are sold in the form of opalescent or milky liquids with slightly bluish tints, or in gel form.

Dried silica gel is sold in the form of a white, free-flowing powder.

# 4. Tests

## 4.1. The solution or powder should have no disagreeable odour or taste.

## 4.2. pH

Depending on the preparation method and on whether acidic or alkaline solutions are employed, the pH should be between 3 and 4 or between 8 and 10.5.

The pH of silicon dioxide powder should be between 5.0 and 7.5 in 10 % aqueous solution.

## 4.3. Silicon Dioxide Concentration (Dry Extract at 110 °C)

The weight, P, of the dry residue expressed in g per 100 g of colloidal solution should correspond to within ± 0.5 g of the product's concentration.

For the powder the desiccation loss is determined by drying at 110°C for 4 hours. Weight loss should be not higher than 12 % of the initial weight. Silicon dioxide



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concentration in the dry powder after desiccation should be above 98%.

## 4.4. Alkalinity

For alkaline colloidal solutions, determine the alkalinity of a 5 g sample using 0.1M hydrochloric acid (R) in the presence of 2 drops of methyl orange solution (R). Alkalinity expressed in terms of Na2O for 100 g of product should be less than P/100.

## 4.5. Preparing the Solution for Tests

Place a volume of colloidal silicon dioxide solution corresponding to 10 g of dry extract or 10 g of dried silicon dioxide powder in a platinum dish 7 cm in diameter and 2.5 cm high. Evaporate until dry. Take up the residue after cooling with 5 ml hydrofluoric acid. Evaporate to dryness. Repeat this procedure until the silicon dioxide residue is eliminated. Evaporate to dryness. Take up the residue using 2 ml concentrated hydrochloric acid (R) and evaporate to dryness. Add 2 ml of concentrated hydrochloric acid (R). Decant in a 50 ml volumetric flask and fill to the mark with distilled water. Safety guidelines for use of concentrated acids have to be respected.

## 4.6. Heavy Metals

To 5 ml of the test solution prepared under paragraph 4.5, add 5 ml of water, 2 ml of pH 3.5 buffer solution (R) and 1.2 ml of thioacetamide reagent (R).

No precipitate should form. If a color appears it should be less intense than that of a control prepared as indicated in the Annex and filled to a volume of 25 ml.

Heavy metal content, expressed in terms of lead in dry extract form, should be less than 10 mg/kg.

## 4.7. Lead

Using the technique described in the OIV Compendium of international methods for analysis of wine and must, determine the lead content in the test solution (4.5).

The lead content should be less than 5 mg/kg.

## 4.8. Mercury

Using the technique described in the annex, determine the mercury content in the test solution (4.5).

The mercury content should be less than 1 mg/kg.



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### 4.9. Arsenic

Using the technique described in the annex, determine the arsenic content in the test solution (4.5).

The arsenic content should be less than 3 mg/kg.

### 4.10. Methanol

Place 50 ml of colloidal silicon dioxide solution in a 200 ml in a round-bottom flask. Distill and collect 50 ml of distillate.

Place 1 ml of distillate in a test tube with 4 drops of 50 % (m/m) orthophosphoric acid (R) and 4 drops of 5 % (m/v) potassium permanganate solution (R). Stir and let sit 10 minutes. Decolorize the permanganate with several drops (typically 8) of 2 % (m/v) of anhydrous potassium sulfite (R), while avoiding any excess. Add 5 ml of sulphuric-acid solution of chromotropic acid (R). Place in a 70 °C water bath for 20 minutes. No violet coloration should appear.

### 4.11. Formaldehyde

Place 10 ml of the distillate obtained under paragraph 4.10 in a test tube. Add 1 ml of rosaniline hydrochloride solution decoloured with sulfuric acid (R). No pink coloration should appear.

### 4.12. Medium (d50) and minimum particle size

Medium particle size of silicon dioxide powder should be between 10 and 100 µm measured by laser scattering particle size analyzer after dispersion in demineralized water. Minimum particle size should be above 1 µm.

### 4.13. Specific surface area (according to BET method)

The BET surface area of silicon dioxide powder is measured by determination of the specific surface area of solid matter by gas adsorption according to ISO 9277:2010.

The specific BET surface area of silicon dioxide powder should be between 300 – 500 $m^2/g$.

## 5. Storage

Colloidal solutions of silicon dioxide should be stored in hermetically sealed containers away from contaminants and at temperatures of above 0 °C (the product freezes at 0 °C with irreversible precipitation of the silicon dioxide).



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Silicon dioxide powder should be stored in sealed bags or boxes protected from off odours or humidity.



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