# RESOLUTION OENO 60/2000

# IX. WINE VINEGARS - DETERMINATION OF THE TOTAL SULFUR DIOXIDE CONTENT (OIV-MA-VI-09)

## 1. INTRODUCTION

In the wine vinegars industry, the addition of sulfur dioxide or salts (E 220 - E 227) is authorized according to defined standards and doses. Accordingly, the applied doses must be checked, above all, their $SO_2$ contents.

## 2. DEFINITION

Free sulfur dioxide is that found in the forms $H_2SO_3$, $HSO_3$ and $SO_3^{2-}$.

Combined sulfur dioxide is found in all other forms.

Total sulfur dioxide is the sum of the free sulfur dioxide and the combined sulfur dioxide.

## 3. PRINCIPLE[1]

Free sulfur dioxide - Iodometric direct titling with subtraction of the other oxidizable substances by iodine.

Combined sulfur dioxide - iodometric titling after double alkaline hydrolysis of vinegar whose free sulfur dioxide has been oxidized during the previous determination.

Total sulfur dioxide - sum of the free sulfur dioxide content and the combined sulfur dioxide content.

## 4. REAGENTS

4.1. Sulfuric acid (p20 = 1.84 g/ml)

4.2. Sulfuric acid solution to 1/10 (1 + 9 of water by volume)

4.3. Solution of sodium hydroxide 4 M

4.4. Solution of iodine at 0.025 M

4.5. Disodic ethylene diamine tetracetate ($EDTA.Na_2$)



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4.6. Starch solution at 5 g/l.

Dissolve 0.5 g of the soluble starch in a small amount of cold water in order to obtain a fluid base. Add to 100 ml of boiling water and maintain on the boil for 10 min. Allow to cool.

4.7. Ethanal solution at 7 g/l

4.8. Propanal solution at 10 g/l.

# 5. EQUIPMENT AND USTENSILS

Standard laboratory equipment.

# 6. TECHNIQUE

## 6.1. Free sulfur dioxide

In a 500 ml conical flask, add 50 ml of vinegar, 3 ml of sulfuric acid solution (4.2), 5 ml of starch solution (4.6) and 30 mg of EDTA $Na_2$ (4.5).

Title immediately with the iodine solution (4.4) until the blue coloring, first fleeting, becomes persistent for 10 to 15 s.

## 6.2. Combined sulfur dioxide

To the aforementioned conical flask (6.1), add the solution of sodium hydroxide (4.3) up to pH 11-12 (approximately 18 ml), stir and leave in contact for 5 min. Add, in one go and while shaking thoroughly, 17 ml of sulfuric acid solution (4.2).

Title immediately with the iodine solution (4.4).

Then add 20 ml of sodium hydroxide solution (4.3), stir and leave in contact for 5 min. Dilute with 200 ml of water, as cold as possible, stir thoroughly and add in one go, 30 ml of sulfuric acid solution (4.2). Title the liberated sulfur dioxide immediately using the iodine solution (4.4).

## 6.3. Interference of other substances

Some other substances may be oxidized by iodine in the acid environment; therefore, it is necessary to determine the quantity of iodine used up by such oxidization.

To do this, it is necessary to combine the free sulfur dioxide by excess ethanal or propanal before iodometric titling. To do this, add 50 ml of vinegar to an Erlenmeyer flask of 300 ml and add 5 ml of ethanal solution or 5 ml of propanal solution. Stop and allow to stand for at least 30 min. Add 3 ml of sulfuric acid solution (4.2) , 5 ml of



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starch solution (4.6) and the iodine solution (4.4) until a blue color is obtained.

# 7. RESULTS

## 7.1. Calculation

Considering:

**V** the volume in ml of the iodine solution used in 6.1

$\mathbf{V_1}$ the volume in ml of the iodine solution used for the first titling of 6.2

$\mathbf{V_2}$ the volume in ml of the iodine solution used for the second titling of 6.2

$\mathbf{V_3}$ the volume in ml of the iodine solution used in 6.3

The total sulfur dioxide content expressed in milligrams of $SO_2$ per l of vinegar is:

- $32 (V + V_1 + V_2 - V_3)$

## 7.2. Presentation

Round off the results expressed in milligrams of $SO_2$ per liter, to the nearest unit.

# 8. ALTERNATIVE METHOD

It is also possible to authorize the use of the reference method described in the *Collection of International Methods of Wine and Must Analysis*, after the publication of studies regarding its application to vinegars.

# 9. BIBLIOGRAPHY

1. Curvelo-Garcia A.S. and Godinho M.C., 1986. Determinação anatitica do dióxido de enxofre em vinagres. Optimização das condições operatórias, Cência e Técnica Vitivinicola, 5(1): 25 - 29.
2. AOF / WHO - Commission of Codex Alimentarius, 1982. Doc. CX/EURO 82/3, Part II, Annex I, Rome.

[1] The CPIV has described another method (determination of the absorbance at 550





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nm of the solution colored with products from the reaction between sulfur dioxide, formaldehyde and p-rosaniline). The *Instituto da Vinha e do Vinho* (Portugal) has developed a continuous flow method based on the same principle.



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