RESOLUTION OENO 65/2000

XIV. WINE VINEGAR - MEASUREMENT OF ZINC CONTENT (OIV-MA-VI-14)

1. INTRODUCTION

As for copper, the presence of zinc in vinegars mainly has its origin in contaminations from contact materials during their manufacture, and, likewise, excessive content could cause hazes or even serious alterations in the colour.

2. PRINCIPLE

Direct measurement by atomic absorption spectrophotometry.

3. REAGENTS

3.1. zinc metal

3.2. concentrated nitric acid 65% (p_{20} = 1.38 g/ml).

3.3. diluted nitric acid at 1:2 (v/v).

3.4. diluted nitric acid at 1% (v/v).

3.5. acetic acid solution at 5% (v/v).

3.6. standard solution of zinc 1.000 g per L.

Weigh 1.000 g of zinc metal, and transfer it entirely into a 1000 ml volumetric flask. Add diluted nitric acid (3.3) in strictly sufficient quantity to dissolve the metal and make up to the mark with diluted nitric acid (3.4).

This solution can be purchased ready-made.

3.7. standard solutions of zinc, from 0.05 to 2.00 mg/L.

Prepare standard solutions of 0.05 to 2.00 mg/L, by diluting the solution (3.6) with the acetic acid solution (3.5).

4. Devices and utensils

Standard laboratory material, plus:

4.1. atomic absorption spectrophotometer

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

4.2. zinc ion hollow cathode lamp

5. Preparation of the sample

Shake the sample to homogenize and filter if necessary.

6. TECHNIQUE

Select a 213.9 nm wavelength. Adjust the absorbence scale to zero with the acetic acid solution (3.5). Suck the vinegar sample directly into the burner of the spectrophotometer, then successively the standard solutions (3.7). Read the absorbencies. The measurements should be done at least twice.

7. RESULTS

7.1. Calculation

Draw the absorbence variation curve according to the zinc concentration of the standard solutions. Write down the absorbencies middle value of the vinegar sample and measure the zinc content, expressed in milligrams per L of the sample.

7.2. Presentation

Round the results expressed in milligrams of zinc per L, to the first decimal place.

8. Inter-laboratory validation (Hitos et al., 00)

Units: mg/L

Sample	R	S_r	RSD_r	R	S_R	RSD_R	RSD_R (Horwitz)	HORRAT Index
1 - 0.39 mg/L	0.0390	0.014	3.55	0.2531	0.090	23.02	18.44	1.25
2 - 0.06 mg/L	0.0454	0.016	25.41	0.0932	0.033	52.22	24.44	2.14

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

3 - 0.16 mg/L	0.0618	0.022	14.13	0.1800	0.064	41.15	21.08	1.95
4 - 0.25 mg/L	0.0503	0.018	7.28	0.2183	0.078	31.61	19.71	1.60
5 - 0.63 mg/L	0.0689	0.025	3.92	0.4157	0.148	23.64	17.15	1.38

9. BIBLIOGRAPHY

1. Anonymous, 1993. *Métodos Oficiales de Anàlisis*, (Official Analytical Methods) Tomo II (Part II) Ministério de Agricultura, Pesca y Alimentación, (Ministry for Agriculture, Fishing and Food) Madrid, Spain.
2. Hitos P., Pons A., Martin de la Hinojosa I., Gomez R., Hernandez A. and Muñoz J., 00. Validation des méthodes d'analyse pour l'acidité totale, fixe et volatile, les substances réductrices non volatiles, le cuivre et le zinc dans les vinaigres de vin (Validation of analytical methods for the total acidity, fixed and volatile, the non volatile reducing substances, the copper and zinc in wine vinegars), *Feuillet Vert de l'OIV* (OIV Green Leaflet) no 1115.
3. Llaguno C. and Polo M.C., 1991. *El Vinagre de Vino* (The Wine Vinegar) Consejo Superior de Investigaciones Cientificas (High Council of Scientific Research) Madrid, Spain.

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