RESOLUTION OENO 3/94

THE COMPENDIUM OF INTERNATIONAL METHODS OF ANALYSIS OF WINES AND MUSTS: NEW METHOD OF ANALYSIS FOR LEAD

THE GENERAL ASSEMBLY,

IN VIEW OF Article 5, paragraph 4 of the International Convention for the Unification of Wine Analysis and Appréciation Methods of the 13th of October 1954,

DECIDES:

TO SUBSTITUTE the lead method on pages 233-234 of the Compendium of International Methods of Analysis of Wines and Musts with the following method (Green Paper 928) which is the resuit of a complété quantitative analytical study.

The matrix modifier is slightly différent. The measurements shall be done at 283.3 nm. instead of 217 nm. The method was validated by collaborative analysis.

1. Principle of the method:

Lead is determined directly in wine by flameless atomic absorption spectrophotometry.

2. Equipment:

AU the glassware must be washed in advance with warm concentrated nitric acid (70-80 °C) and rinsed

with double distilled water.

2.1. Atomic absorption spectrophotometer equipped with a graphite fumace, a non spécifie absorption corrector and a mulüpotentiometric recorder.

2.2. Lead hollow cathode lamp.

2.3. 5 jug micropipettes fitted with spécial tips for atomic absorption measurements.

3. Reagents:

AU the reagents must be of recognised analytical purity and, in particular must be free of lead. The water used must either be double-distilled water in a borosilicate glass apparatus or be of an équivalent purity.

3.1. Phosphoric acid of 85 p. 100 (P_2Q — 1.71 g/mL).

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3.2. Phosphoric acid solution obtained by diluting 6 mL of phosphoric acid to 100 mL with water

3.3. Nitric acid (P_{20} - 1.38 g/mL)

3.4. Lead solution at 1 g/L.

Use a commercial standard solution. This solution may be obtained by dissolving 1.600 g of lead nitrate II, Pb $(NOj)_2$ in nitric acid diluted to 1% by volume and ajusting the volume to 1 liter. Store the solution in a borosilicate glass flask with a ground stopper.

3.5. Solution of nitric acid diluted to 1% by volume.

3.6. Solution obtained by diluting the 6 % phosphoric acid solution in half with the 1% nitric acid

solution.

4. Proceedure

4.1. Préparation of the sample.

Add to the wine test to be analysed, an equal volume of the solution (3.6.) of nitric and phosphoric acids. Détermine its absorbance.

If it is higher than 0.6, further dilute the wine (a 1 to 5 dilution suffices in most cases).

Préparé the solution to be determined by adding to the diluted wine to be analyzed, an equal volume of the solution of phosphoric and nitric acids.

4.2. Préparation of solutions for the calibration scale.

Starting with the lead reference solution, préparé dilutions containing the 50% solution of nitric and phosphoric acids (3.6). The concentration levels of the standard solutions dépend on the sensitivity of the apparatus. For example, préparé solutions containing 10-20-30- micrograms of lead per liter.

4.3. Détermination

4.3.1. Program of the furnace.

Stage	Temperature (°C)	Time (s) Nitrogen (1)	L/min	Reading
1	75	2.0	3.0	
2	95	20	3.0	
3	140	15.0	3.0	

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4	300	8.0	3.0	
5	450	7.0	3.0	
6	480	10.0	3.0	
7	900	20.0	3.0	
8	900	1.0	0.0	
9	2 250	0.7	0.0	*
10	2 250	1.0	0.0	*
11	2 250	2.0	3.0	

(1) Argon could be substituted for nitrogen, subject to similar results.

4.3.2. Measurements

Select a wavelength of 283.3 nm. Set zéro absorbance with double-distilled water. With a micropipette or an automatic sample injecter, make triplicate 5 microliter injections of each of the standard solutions and of the sample to be analysed.

Record the absorbances measurements. Calculate the average value of the absorbance based on the results of the 3 injections.

Absorbances are measured at peak values.

S. Expression of the results 5.1. Calculation

Plot the curve of the absorbance variations as a function of lead concentrations of the standard solutions. The variation is linear. Write out the average value of the absorbance of the sample solution from the standard curve, deducing from it the concentration C of lead. The lead concentration expressed in micrograms per liter of wine is equal to: C X F.

F = the dilution factor.

5.2. Repeatability r = 9.4 JLlg/L

5.3. Reproductibility R = 15 juG/L

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