OIV-MA-AS322-10 Cadmium

Type IV method

1. Principle

Cadmium is determined directly in the wine by graphite furnace atomic absorption spectrophotometry.

2. Apparatus

All the glassware must be washed in concentrated nitric acid prior to use, heated to 70 to 80 □C and rinsed in double distilled water.

2.1. Atomic absorption spectrophotometer equipped with a graphite furnace, background correction and a recorder.

2.2. Cadmium hollow cathode lamp

2.3. 5 µl micropipettes with special tips for atomic absorption measurement.

3. Reagents

The water used must be double distilled prepared using borosilicate glass apparatus, or water of a similar purity. All reagents must be of recognized analytical reagent grade and, in particular, free of cadmium.

3.1. Phosphoric acid ($□_{20}$ = 1.71 g/mL), 85%.

3.2. Phosphoric acid solution obtained by diluting 8 mL of phosphoric acid with water to 100 mL.

3.3. 0.02 M Ethylenediaminetetraacetic acid disodium (EDTA) solution.

3.4. pH 9 buffer solution: dissolve 5.4 g of ammonium chloride in a few milliliters of water in a 100 mL volumetric flask, add 35 mL of 25% (*v/v*) ammonium hydroxide solution. Ammonium hydroxide solution, $□_{20}$= 0.92 g/mL, diluted to 25% (*v/v)* and made up to 100 mL with water.

3.5. Eriochrome black T, 1% (*m/m*) solution in sodium chloride.

3.6. Cadmium sulfate, $3CdSO_4.8H_20$.

The concentration of the cadmium sulfate must be verified using the following method:

Weigh exactly 102.6 mg of the cadmium sulfate sample into a beaker with some water and shake until dissolved; add 5 mL of the pH 9 buffer solution and approximately 20 mg of Eriochrome black T. Titrate with the EDTA solution (3.3) until the indicator begins to turn blue.

The volume of EDTA added must be equal to 20 mL. If the volume is slightly different, correct the weighed test portion of cadmium sulfate used in the preparation of the reference solution accordingly.

3.7. Cadmium reference solution at 1 g per liter.

Use of a standard commercial solution is preferred. Alternatively this solution may be prepared by dissolving 2.2820 g of cadmium sulfate in water and making up to one liter. Keep the solution in a borosilicate glass bottle with a ground glass stopper.

4. Procedure

4.1. Preparation of the sample

The wine is diluted 1/2 (*v/v*) with the phosphoric acid solution (3.2).

4.2. Preparation of calibration standards

Using the cadmium reference solution, prepare successive dilutions 2.5, 5, 10 and 15 µg of cadmium per liter respectively.

4.3. Determination

4.3.1. Furnace Programming (for guidance only):

Dry at 100□C for 30 seconds

Mineralize at 900 □C for 20 seconds

Atomize at 2250 □C for 2 to 3 seconds

Nitrogen flow (flushing gas): 6 liters/minute

Note: At the end of the procedure, increase the temperature to 2700 □C to clean the furnace.

4.3.2. Atomic absorption measurements

Select an absorption wavelength of 228.8 nm. Set the zero on the absorbance scale with double distilled water. Using a micropipette, introduce into the furnace three 5 µl portions of each of the solutions in the calibration range and the sample solution to be analyzed. Record the absorbance measured. Calculate the mean absorbance value from the results for the three portions.

5. Expression of results

1. Method of calculation

Draw the absorbance variation curve as a function of the concentration of cadmium in the solutions in the calibration range. The curve is linear. Enter the mean absorbance value of the sample solution on the calibration curve and obtain the cadmium concentration C. The cadmium concentration expressed in micrograms per liter of

wine is equal to 2C.

Bibliography

- MEDINA B., *Application de la spectrométrie d absorption atomique sans flamme au dosage de quelques métaux dans les vins,* Thèse Doct. en œnologie, Bordeaux II, 1978.
- MEDINA B. and SUDRAUD P., FV O.I.V 1979, nº 695.