OIV-MA-AS321-04 Total phosporus

Type IV method

1. Principle

After nitric oxidation and ashing, and dissolution in hydrochloric acid, phosphoric acid is determined colorimetrically as the yellow phospho-vanadomolybdate complex.

2. Apparatus

2.1. Boiling water□bath 100□C

2.2. Hot plate

2.3. Temperature□controlled electric furnace.

2.4. Spectrophotometer measuring absorbance at wavelengths between 300 and 700 nm

3. Reagents

3.1. Nitric acid, (ρ_{20} = 1.39 g/mL).

3.2. Hydrochloric acid, approx. 3 M; hydrochloric acid (ρ_{20} = 1.15 - 1.18 g/mL) diluted ¼ with water.

3.3. Vanadomolybdate reagent:

Solution A: dissolve 40 g of ammonium molybdate, $(NH_4)_6Mo_7O_{24}.4H_2O$, in 400 mL water.

Solution B: dissolve 1 g of ammonium vanadate, NH_4VO_3, in 300 mL water and 200 mL nitric acid (ρ_{20} = 1.39g/L) (3.1). Leave to cool.

Vanadomolybdate reagent: place first solution B then solution A into a 1 liter flask, and make up to the mark with water. Reagent to be used within 8 days of preparation.

3.4. P_2O_5 solution, 0.1 g/L.

Prepare a P_2O_5 solution 1 g/L by dissolving 2.454 g of *di*-potassium hydrogen phosphate, K_2HPO_4 , in a liter of water. Dilute 10% (*v/v*).

4. Procedure

4.1. *Ashing*

Place 5 mL[*] wine or must in a platinum or silica dish and evaporate on a boiling water□bath (2.1). When the residue is nearly dry add 1 mL nitric acid (3.1), place the dish on a hot plate (2.2) for 1 hour then in a furnace (2.3) at 600□650 oC until the ash is

white.

4.2. Determination

Add 5 mL of hydrochloric acid, approximately 3 M (3.2) to the ash and transfer the solution to a 100 mL volumetric flask. Rinse the dish with 50 mL distilled water and pour the washings into the flask. Add exactly 25 mL of vanadomolybdate reagent, stir and leave for 15 to 20 min to allow the color to develop. Determine the absorbance at 400 nm.

Simultaneously, prepare standard solutions. Place in five 100 mL volumetric flasks, 5, 10, 15, 20 and 25 mL respectively of P_2O_5 solution, 0.1 g/L (3.4). Make up to 50 mL with distilled water and add 25 mL vanadomolybdate reagent. Leave for the exact same time as the samples, to allow the color to develop. Make up to the mark with water and measure the absorbance at 400 nm.

In order to remain in the best absorbance zone do not reset to zero with distilled water, but set the deviation of the spectrophotometer galvanometer on a given absorbance for a determined concentration.

5. Expression of results

5.1. *Calculation*

The total phosphorous content expressed in milligrams per liter of phosphoric anhydride, P_2O_5 , is obtained by entering the absorbance of the wine sample on the calibration graph and interpolating the total phosphorus concentration.

The total phosphorous content is expressed in milligrams per liter P_2O_5 to the nearest whole number.

Bibliography

- A.F.N.O.R., Norme U, 42-246, Tour Europe, Paris.
- Sudraud P., *Bull. O.I.V.*, 1969, 462-463, 933.

[*] A 5 mL sample volume is suitable for P_2O_5 content, of between 100 and 500 mg/L. Outside these concentration limits, increase or decrease the sample volume.