OIV-MA-AS322-09 Silver

Type IV method

1. Principle

The method is based on the use of atomic absorption spectrophotometry after ashing the sample.

2. Apparatus

2.1. Platinum dish.
2.2. Water bath, thermostatically controlled to 100 °C
2.3. Furnace set at 500 to 525 °C.
2.4. Atomic absorption spectrophotometer.
2.5. Silver hollow cathode lamp.
2.6. Gas supplies: air, acetylene.

3. Reagents

3.1. Silver nitrate, $AgNO_3$.
3.2. Nitric acid, (ρ_{20} = 1.38 g/mL), 65%.
3.3. Nitric acid, diluted 1/10 (*v/v*) with distilled water.
3.4. Solution containing 1 g of silver per L.
Use of a standard commercial silver solution is preferred. Alternatively this solution may be prepared by dissolving 1.575 g of silver nitrate in dilute nitric acid and making up to a volume of 1,000 mL with dilute nitric acid (3.3).Solution containing 10 mg of silver per L.
3.5. Take 10 mL of the 1 mg/L solution and make up to 1 L with dilute nitric acid.

4. Procedure

4.1. Preparation of sample
Place 20 mL of the sample in a platinum dish and evaporate to dryness over a boiling water bath. Ash in the furnace at a temperature of 500 to 525 °C. Moisten the white

ash with 1 mL of concentrated nitric acid (3.2). Evaporate over a boiling water bath, repeat the addition of 1 mL nitric acid (3.2) and evaporate a second time. Add 5 mL of dilute nitric acid (3.3) and heat gently until dissolved.

4.2. Calibration

Pipette 2, 4, 6, 8, 10 and 20 mL of solution (3.5) respectively into each of size 100 mL volumetric flasks and make up to the mark with dilute nitric acid (3.3): the solutions contain 0.20, 0.40, 0.60, 0.80, 1.0 and 2.0 mg of silver per liter respectively.

4.3 Set the absorbance wavelength to 328.1 nm. Adjust zero using double distilled water. Measure the absorbance directly of successive standard solutions (4.2) and carry out in duplicate.

5. Expression of results

Plot a graph showing the variation in absorbance as a function of the silver concentration in the standard solutions.

Using the measured absorbance of the sample read the concentration C in mg/L from the calibration curve.

The concentration of silver in the wine is given in milligrams per liter by

$$0.25 \times C$$

It is quoted to two decimal places.

Note: Select the concentration of the solutions for the preparation of the calibration curve. The volume of sample taken and the final volume of the liquid should be appropriate for the sensitivity of the apparatus to be used.