OIV-MA-AS321-05A Sulfates

Type II method

1. Principle

Gravimetric determination following precipitation of barium sulfate. The barium phosphate precipitated at the same time is eliminated by washing the precipitate in hydrochloric acid.

In the case of musts or wine rich in sulfur dioxide, prior de□sulfiting by boiling in an airtight vessel is recommended.

2. Method

2.1. *Reagents*

2.1.1. Hydrochloric acid, 2 M.

2.1.2. Barium chloride solution, $BaCl_2$.2H2O, 200 g/L.

2.2. *Procedure*

2.2.1. General procedure:

Introduce 40 mL of the sample to be analyzed into a 50 mL centrifuge tube; add 2 mL hydrochloric acid, 2 M (2.1.1), and 2 mL of barium chloride solution, 200 g/L (2.1.2). Stir with a glass stirrer; rinse the stirrer with a little distilled water and leave to stand for five min. Centrifuge for five min, then carefully decant the supernatant liquid.

Wash the barium sulfate precipitate as follows: add 10 mL hydrochloric acid, 2 M (2.1.1), place the precipitate in suspension and centrifuge for five min, then carefully decant the supernatant liquid. Repeat the washing procedure twice as before using 15 mL distilled water each time.

Quantitatively transfer the precipitate, with distilled water, into a tared platinum capsule and place over a water bath at 100°C until fully evaporated. The dried precipitate is calcined several times briefly over a flame until a white residue is obtained. Leave to cool in a desiccator and weigh.

Let m = mass in milligrams of barium sulfate obtained.

2.2.2. Special procedure: sulfited must and wine with a high sulfur dioxide content.

Elimination of sulfur dioxide.

Measure 25 mL of water and 1 mL of concentrated hydrochloric acid ($□_{20}$= 1.15 to 1.18 g/mL) into a 500 mL conical flask equipped with a dropping funnel and an outlet tube. Boil the solution to remove the air and introduce 100 mL of wine through the dropping funnel. Continue boiling until the volume of liquid in the flask has been

reduced to about 75 mL and quantitatively transfer, after cooling, to a 100 mL volumetric flask. Make up to mark with water. Determine the sulfate in the 40 mL sample as indicated in 2.2.1.

2.3. Expression of results

2.3.1. Calculations:

The sulfate content, expressed in milligrams per liter of potassium sulfate, K2SO4 is given by: 18.67 x *m*

The sulfate content in musts or wine is expressed in milligrams per liter of potassium sulfate, to the nearest whole number.

2.3.2. Repeatability *(r)*:

- up to 1000 mg/L: *r* = 27 mg/L
- approx. 1500 mg/L: *r* = 41 mg/L

2.3.3. Reproducibility (R):

- up to 1000 mg/L: R = 51 mg/
- approx. 1500 mg/L: R = 81 mg/L

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