OIV-MA-VI-12 Measurement of sulphate content

Type IV method

1. Introduction

The main objective of the measurement of the sulphate content in vinegar is, as for the measurement of the chloride content, the detection of frauds (aimed at increasing the total dry extract).

2. Principle

Precipitation of sulphates with barium chloride, drying, calcination and weighing.

3. Reagents

3.1. barium chloride solution ($BaCI_2$, $2H_2O$) at 1% (m/v)

3.2. hydrochloric acid solution M

3.3. silver nitrate solution 1M.

4. Devices and utensils

Standard laboratory material, including:

4.1. flat-bottom platinum crucible (85 mm in diameter)

4.2. filter paper for fine filtering of the precipitate, with an ash content of no more than 0.01%

4.3. water bath at 100 °C

4.4. muffle kiln

4.5. dessiccator

5. Preparation of the sample

Shake the sample to homogenize and filter if necessary.

6. Technique

In a 250 ml conical flask, introduce 100 ml of the sample. Add 2 ml of the hydrochloric acid solution (3.2) and heat to boiling point. Add, drop by drop, 10 ml of the barium chloride solution (3.1), maintain at boiling point for 5 minutes and add hot water to maintain a constant volume. Leave to rest for 10 to 12 hours.

Filter and wash the precipitate with hot water, until the washing waters are free from chlorides, as can be verified by the absence of a precipitate with a silver nitrate solution (3.3).

Carefully transfer the filter with its content into the platinum crucible, previously tared, and kiln in a muffle kiln at 700 °C - 800 °C.

Cool in the dessiccator and weigh.

7. Results

7.1. Calculation

Taking:

- m_1 = the mass, in grams, of the empty platinum crucible
- m_2 = the mass, in grams, of the platinum crucible containing the kilned residue (less the weight of the filter paper ash)

The sulphate content, expressed in grams of potassium sulphate per L of the sample, is given by the expression:

$$7.4651 \times (m_2 - m_1)$$

7.2. Presentation

Round results expressed in grams of potassium sulphate per L, to the first decimal place.

8. Bibliography

- Anonymous, 1993. *Métodos Oficiales de Anàlisis*, (Official Analytical Methods) Tomo II (Part II) Ministério de Agricultura, Pesca y Alimentación, (Ministry for Agriculture, Fishing and Food) Madrid, Spain.
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