OIV-MA-VI-07 Determination of ash content

Type II method

1. Introduction

The main purpose of determining the ash content is to detect certain frauds, for instance the addition of water or an acetic acid aqueous solution (very low ash content) or the addition of non-volatile substances (very high ash content) for the correct interpretation of the results, in which case it is necessary to have a database for the type and origin of the vinegar being analyzed.

2. Definition

Vinegar ashes refer to all the incineration products of the evaporation residue of a known volume of vinegar, carried out in such a way as to obtain all the cations (except for ammonium) in the form of carbonates and other anhydrous mineral salts.

3. Principle

Incineration of the vinegar extract between 500°C and 550°C through to complete combustion of the carbon.

4. Equipment and utensils

Standard laboratory equipment including:

4.1. Water bath at 100 °C

4.2. Scales sensitive to within 1/10th of a milligram

4.3. Hot plate or infrared evaporator

4.4. Temperature controlled electric oven

4.5. Platinum (or quartz) capsules, 70 mm in diameter and 25 mm high with a flat bottom.

5. Preparation of sample

Homogenize the sample by stirring, then filter if necessary.

6. Technique

Add 20 ml of the sample to a previously calibrated platinum capsule and evaporator in a water bath at 100°C until a syrupy consistency is obtained. Heat the residue on a hot plate to 200°C or using an infrared evaporator through to carbonization. When the residue no longer gives off vapor, put the capsule in the electric oven brought to 525°C $\pm$25°C. After 5 min. of carbonization, remove the capsule from the oven, allow

to cool and add 5 ml of distilled water, which can then be evaporated in the water bath at 100 °C or under the infrared evaporator. Heat again to 525°C for 10 min.

If the carbonated particles do not bur up entirely, repeat the adding water, evaporation and incineration steps.

After cooling in a drying chamber, weigh the capsule (with the ashes).

7. Results

7.1. Calculation

Considering:

- p0 the mass of the empty capsule in grams
- p1 the mass of the capsule containing the ash in grams

The content of ash expressed in g/l is given by

$$50\,(p1 - p0)$$

7.2. Presentation

Round off the results expressed in g/l to the second decimal.

8. INTER-LABORATORY VALIDATION

r = 0.30 g/l

R = 1.0 g/l

9. Bibliography

- Anonymous, 1993, *Métodos Oficiales de Análisis,* Tomo II, Ministério de Agricultura, Pesca y Alimentación, Madrid, Spain.
- CPIV, 1995. *Methods of Analysis*, Annex 4 to the minutes of CPIV Technical Committee's Meeting, Stuttgart, Germany.
- Curvelo-Garcia A.S., 1996. Wine vinegars. Methods of Analysis (Part Two). *Green Sheet of OIV No. 1033.*
- Llaguno C. et Polo M.G., 1991. *El Vinagre de Vino*, Consejo Superior de Investigaciones Cientificas, Madrid, Spain.

- OIV, 1990. *Collection of international analysis methods of wine and must,* OIV, Paris, France.