Method OIV-MA-VI-11 : R2000
Type IV method

Measurement of chloride content in vinegars

(OENO 62/2000)

1. Introduction

The main objective of the measurement of chloride content is the detection of the fraudulent increase in the dry extract by the addition of sodium chloride.

2. Principle[1]

Potentiometric titration of Cl^- ions with a solution of silver nitrate, in an acidic environment, after prior measurement of the potential equivalent point of a standard chloride solution.

3. Reagents

3.1. chloride standard solution

Weigh, to an accuracy of ± 0.0001 g, 2.1027 g of potassium chloride (Br content <0.005% Br) dried first for several days in a dessiccator with an appropriate desiccant.
Place the KCl into a 1 litre volumetric flask, dissolve in water and make up to the mark.
1 ml of this solution contains 1 mg of Cl- ion.

3.2. silver nitrate standard solution

Weigh, to an accuracy of ± 0.0001 g, 4.7912 g of silver nitrate.
Place the $AgNO_3$ into a 1 litre volumetric flask, dissolve in a 10% (v/v) aqueous/alcoholic solution and make up to the mark.
1 ml of this solution corresponds to 1 mg of Cl- ion.

3.3. nitric acid (r_{20} = 1.40 g/ml).

4. Devices and utensils

Standard laboratory material, including:

4.1. graduated potentiometer - 2 by 2 mV, at least.

4.2. Ag/AgCl electrode and mercury sulphate (I) electrode or a combined (Ag/AgCl + reference) electrode.

4.3. micro burette graduated in 0.01 ml.

5. Preparation of sample

Shake the sample to homogenize and filter if necessary.

6. Technique

6.1. Measurement of the potential of the equivalent point

Put 5 ml of standard chloride solution (3.1) into a 200 ml flask, placed on a magnetic shaker.

Add 95 ml of water and 1 ml of nitric acid (3.3), introduce the electrodes into the flask and, with moderate shaking, add the silver nitrate solution (3.2), using a micro burette. Add the first 4 ml by 1ml parts and read the corresponding values of the potential difference in mV. Add 2 ml by 0.2 ml parts. Then, add fractions of 1 ml until a total volume of 10 ml is reached.

After every addition wait about 30 sec before making the corresponding reading of the potential difference in mV.

Graph on the ordinate axis the potential difference values found (in mV) and on the

abscissa axis the corresponding volumes of the silver nitrate solution (in ml). Measure the potential of the equivalent point situated at the point of inflection of the curve.

6.2. Verification of the potential of the equivalent point

In a 200 ml flask, introduce 5 ml of chloride standard solution (3.1), 95 ml of water and 1 ml of nitric acid (3.3). Introduce the electrodes and titrate, while shaking, until the potential of the equivalent point is reached. Repeat this operation until the results are in concordance. This verification must take place before every set of chloride measurements is made on the samples.

6.3. Measurement of the potential of the equivalent point of the sample

In a 200 ml flask, introduce 50 ml of vinegar, 50 ml of water and 1 ml of nitric acid (3.3). Titrate as indicated in 6.2.

7. Results

7.1. Calculation

Taking V as the volume in ml of the silver nitrate solution as in 6.3, the chloride content (expressed as mg of Cl - ion per L of vinegar) is given by:

$$20\,V^2$$

7.2. Presentation

Round results as mg of Cl - ion per L to integer values.

8. Bibliography

1. 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.

2. FAO/OMS, Commission du Codex Alimentarius (Codex Alimentarius Commission) Doc. CX/EURO 82/3 Part II, Rome (1982).
3. Llaguno C. and Polo M.C., 1991. *El Vinagre de Vino* (The Wine Vinegar) Consejo Superior de Investigaciones Cientificas (High Council of Scientific Research) Madrid, Spain.

[1]The Instituto da Vinha e do Vinho (Portugal) described a method by continuous flux, according to another principle (Green Leaflet of OIV 949).