Method OIV-MA-VI-05 : R2000
Type II method

Determination of the residual alcohol content in vinegars

(OENO 56/2000)

1. Definition

The residual alcohol content is the percentage by volume of ethanol still contained in the vinegar after acetic fermentation.

2. Principle[1]

Distillation of vinegar, oxidization of ethanol by potassium dichromate and determination of its content by titling the excess potassium dichromate by a solution of iron sulfate (11) and ammonium.

3. Reagents

3.1. Sulfuric acid □20 = 1.84 g/ml.

3.2. Sulfuric acid solution at 50% (v/v).

3.3. Solution of potassium dichromate

In a 1 l calibrated flask, dissolve 33.6 g (to 0.001 g) of potassium dichromate in water. Bring up to the line with water.

1 ml of this oxide solution 7.8924 mg of ethanol.

The solution must be preserved in a flask with a ground glass stopper.

3.4. Solution of iron sulfate (II) and ammonium (Mohr's salt).

In a 1 l calibrated flask, dissolve 135 g of iron sulfate (II) and ammonium in 20 ml of sulfuric acid (3.1) and bring up to the mark with water.

1 ml of this solution, recently prepared, corresponds to approximately 2.5 ml of potassium dichromate solution.

Given that this solution oxidizes easily, it must be titled frequently with potassium dichromate as described in 6.2 and 6.3, but replacing 10 ml of distillate by 10 ml water.

3.5. potassium permanganate solution

In a 1 l calibrated flask, dissolve 1.088 g of potassium permanganate in water and bring up to the line.

3.6. orthophenantroline solution

In a 100 ml calibrated flask, dissolve in water 0.695 g of $FeS0_4$ $7H_20$ and 1. 485 of mono-hydrated orthophenantroline.

Heat to facilitate distillation, cool and bring up to the line with water.

3.7. Calcium hydroxide solution at 120 of CaO per L.

4. Equipment and utensils

Standard laboratory equipment including:

4.1. Distillation apparatus comprising a 1 l calibrated flask connected to a ground glass union with a rectification column at least 20 cm long or another equivalent device, and a condensation column.

4.2. 250 ml conical flasks with ground glass stoppers.

5. Preparation of sample

Homogenize the sample by stirring and filter if necessary.

6. Technique

6.1. Distillation

Add 200 ml of vinegar to a distillation flask. Add calcium hydroxide solution (3.7) until alkalization is observed by litmus paper contact.

Add a few pieces of pumice stone, porcelain or glass balls and one drop of silicone aqueous solution to prevent the formation of foam. Heat to boiling point and gather approximately 90 ml in a 100 ml flask. Allow to cool and bring up to the line with water.

6.2. Oxidization

In a conical flask with a ground glass stopper, add 200 ml of the potassium dichromate (3.3) solution and 20 ml of sulfuric acid. Stir.

Add 10 ml of distillate. Wet the stopper with a drop of sulfuric acid, stop the flask and shake.

Wait at least 30 min. and shake from time to time.

6.3. Titling

Title the excess potassium dichromate with the iron sulfate solution (II) and ammonium (3.4). When the green color turns to greenish-blue, add 4 drops of orthophenantroline (3.6) solution. Titling is terminated when the solution turns to green-brown.

If the change point is slightly exceeded, come back to the exact point of change of color using the potassium permanganate (3.5) solution. One-tenth of the volume of the solution used must be deducted from the volume of the iron sulfate (II) and ammonium solution used.

6.4. Specimen test

Carry out a specimen test with 10 ml of water instead of 10 ml of distillate.

7. Results

7.1. Calculation

Considering:

- V the volume of ml of the iron sulfate solution (II) and of aluminum used in titling (6.3).
- V' the volume in ml of the iron sulfate solution (II) and of ammonium used in the specimen test (6.4).

The residual alcohol content expressed as a percentage (v/v) at 20°C, as given by:

$$(V, - V)/V'$$

7.2. Presentation

Round off the results to the nearest decimal.

8. Inter-laboratory validation (Curvelo-Garcia, 1996)

- F < 0.02% (v/v)
- R < 0.04% (v/v)

9. Bibliography

1. Anonymous, 1993, *Métodos Oficiales de Análisis,* Tomo II, Ministério de Agricultura, Pesca y Alimentación, Madrid, Spain.
2. Curvelo-Garcia A.S. 1996. *Wine vinegars, Analysis Methods (Part Two),* Green Book of OIV No. 1033.
3. AOF / WHO - Commission of Codex Alimentarius, *Methods of analyzing the European regional standard for vinegar*, Alinorm 83/19 and 85/19.

4. Llaguno C. et Polo M.G., 1991. *El Vinagre de Vino*, Consejo Superior de Investigaciones Cientificas, Madrid, Spain.

[1] CPIV has described another method (distillation of the sample and determination of the density of the distillate).