OIV-MA-VI-18 Measurement of the acetoin content

Type IV method

1. Introduction

Acetoin ($CH_3COCHOHCH_3$) is always present in wines and in vinegars. According to the bibliography its content in wines is of the order of 10 mg/L. In vinegars, contents can vary with the manufacturing technology between 100 mg/L and over 400 mg/L.

The acetoin content in the wine vinegars is an important reference factor for quality and origin.

2. Principle

Neutralization of the sample at pH 7.00 with calcium hydroxide. Direct measurement of the acetoin via gas chromatography.

3. Reagents

3.1. purified acetoin. Eliminate any diacetyl via distillation.

3.2. acetoin reference solutions: dilute acetoin (3.1) with water to prepare 10 to 500 mg/L reference solutions.

3.3. Pentan-1-ol (in-house standard solution)

3.4. Ethanol

3.5. in-house standard solution sample: in a 100 ml volumetric flask, dissolve 2 ml of pentan-1-ol in an aqueous-alcoholic solution at 50%. Make up to the mark with this solution.

3.6. calcium hydroxide

4. Devices and utensils

Standard laboratory material, plus:

4.1. gas chromatograph with a flame ionization detector.

4.2. column for gas chromatography 2 m long and 1/8" in diameter: FFAP 2.5% on G Chromosorb (HP), with the addition of 0.5% of 1500 Carbowax (or any other system able to perform an acceptable separation of acetoin).

5. Technique

Add some in-house standard solution (3.5) to the acetoin reference solutions (3.2), in sufficient quantity for these solutions to have, per L, 15 or 35 µL of pentan-1-ol (according to their acetoin content, that is respectively < or > 50 mg/L).

Neutralize the sample at pH □ 7.00 by addition of calcium hydroxide (solid). Add enough of the in-house standard solution (3.5), for the solution to have, per L, 15 or 35 µL of pentan-1-ol (according to the acetoin content).

Inject into the chromatograph 2 µL of the neutralized sample, the reference solutions, and the in-house standard solution. Temperature of the oven is 70° C, output of the vector gas (nitrogen) is 12.5 ml/min. Temperature of the detector is 180° C.

6. Results

6.1. Calculation

Taking:

- A_1 the surface of the peak of the acetoin in the reference solution 1
- P_1 the surface of the peak of pentan-1-ol in the reference solution 1
- A_X the surface of the peak of the acetoin in the solution to be measured
- P_X the surface of the peak of pentan-1-ol in the solution to be measured

Calculate the ratios A_1 / P_1 for the various reference solutions.

Draw two curves to express graphically these ratios according to the acetoin content of the reference solutions (0 to 50 mg/L and 50 to 500 mg/L).

The acetoin content of the sample, expressed in mg/L, is shown by the ratio A_x/P_x.

6.2. Presentation

Round results as mg per L to integer values.

7. 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.
- Gorostiza E., Gil de la Peña, M. et Cordobés M., La Semana Vitivinícola: 1577-1578 (1976).
- 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.