RESOLUTION OENO 69-2000

XVIII. WINE VINEGAR - MEASUREMENT OF THE ACETOIN CONTENT

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

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