

Method OIV-MA-VI-18 : R2000

Type IV method

## **Measurement of the acetoin content in vinegars**

(OENO 69/2000)

### **1. Introduction**

Acetoin ( $\text{CH}_3\text{COCHOHCH}_3$ ) 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  $\mu\text{L}$  of pentan-1-ol (according to their acetoin content, that is respectively  $<$  or  $>$  50 mg/L).

Neutralize the sample at  $\text{pH} \approx 7.00$  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  $\mu\text{L}$  of pentan-1-ol (according to the acetoin content).

Inject into the chromatograph 2  $\mu\text{L}$  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

1. Anonymous, 1993. *Métodos Oficiales de Análisis*, (Official Analytical Methods) Tomo II (Part II) Ministerio de Agricultura, Pesca y Alimentación, (Ministry for Agriculture, Fishing and Food) Madrid, Spain.
2. Gorostiza E., Gil de la Peña, M. et Cordobés M., *La Semana Vitivinícola: 1577-1578* (1976).
3. Llaguno C. and Polo M.C., 1991. *El Vinagre de Vino* (The Wine Vinegar) Consejo Superior de Investigaciones Científicas (High Council of Scientific Research) Madrid, Spain.