

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

#### Type IV method

#### **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).

# COMPENDIUM OF INTERNATIONAL METHODS OF ANALYSIS FOR VINEGARS

## Measurement of the acetoin content (Type IV)

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Neutralize the sample at 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  $\mu$ L of pentan-1-ol (according to the acetoin content).

Inject into the chromatograph 2  $\mu$ 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

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