

## **OIV-MA-VI-19 Measurement of methanol, superior alcohols and ethyl acetate**

Type IV method

### **1. Introduction**

The method described here allows to measure, at the same time, several major volatile components of vinegars. The interest of this measurement is two-fold, organoleptic and possibly toxicologic.

### **2. Principle**

Neutralization of the sample at pH 7.00 with a sodium hydroxide solution.

Measurement, via gas chromatography, of some volatile components:

- Methanol
- propan-1-ol
- butan-2-ol
- 2-methylpropan-1-ol
- butan-1-ol and 2-methylbutan-1-ol + 3-methylbutan-1-ol.

### **3. Reagents**

3.1. aqueous-alcoholic solution at 5% v/v.

3.2. in-house standard solution (4-methylpentan-2-ol).

In a 1 L volumetric flask, dissolve 1110.0 mg (to an accuracy of 0.1 mg) of 4-methylpentan-2-ol into the aqueous-alcoholic solution (3.1). Make up to the mark with this solution.

3.3. reference solution

In a 1 L volumetric flask, dissolve, in the aqueous-alcoholic solution (3.1), 152.0 mg of ethyl acetate, 50.0 mg of methanol, 7.7 mg of propan-1-ol, 16.3 mg of butan-2-ol, 17.0 mg of 2-methylpropan-1-ol, 2.5 mg of butan-1-ol, 7.9 mg of 2-methylbutan-1-ol and 8.7 mg of 3-methylbutan-1-ol (to the accuracy of 0.1 mg). Make up to the mark with the solution (3.1).

3.4. reference solution added from the in-house standard solution. Add 1 ml of the solution (3.2) to 10 ml of the solution (3.3).

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3.5. sodium hydroxide solution at 40% (m/v).

### 4. Devices and utensils

Standard laboratory material, plus:

- 4.1. gas chromatograph with a 'split' type injector and a flame ionization detector
- 4.2. Supelcowax 10 glass column, 30 m long and 0.75 mm internal diameter (as an example).

### 5. Technique

Neutralize the sample at pH  $\approx$  7.00 with the sodium hydroxide solution (3.5), and record the initial and final volumes.

Add 1 ml of the neutralized sample solution at 10 ml (3.2).

Inject 1  $\mu$ L of each of these two solutions into the chromatograph. Temperatures of the injector and the detector are 250° C. Oven temperatures are: 6 mn at 50° C, 50° C to 70° C to 8° C/mn, 14 mn at 70° C, 70° C to 210° C to 8° C/mn and 16 mn at 210° C. The output of the vector gas (hydrogen) is 10 ml/mn.

### 6. Results

#### 6.1. Calculation

Taking:

- $c_i$  the component 1 content, in mg/L, in the reference solution (3.4)
- $c_e$  the in-house standard solution content, in mg/L, in the reference solution (3.4)
- $s_i$  the surface of the peak of component 1 of the reference solution (3.4)
- $s_e$  the surface of the peak of the in-house standard solution in the reference solution (3.4)
- $S_i$  the surface of the peak of component 1 in the neutralized sample solution plus the in-house standard solution
- $S_e$  the surface of the peak of the in-house standard solution in the neutralized sample solution plus the in-house standard solution
- $C_e$  the in-house standard solution content, in mg/L, in the neutralized sample solution plus the in-house standard solution

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- $f$  the dilution factor resulting from the neutralization of the vinegar sample.

The component  $i$  content  $C_i$ , expressed in milligrams per L of vinegar, is given by:

$$C_i = \frac{C_j S_e S_j C_e}{C_e S_i S_e} f$$

### 6.2. Presentation

Round results to the integer value.

## 7. Bibliography

- Climaco, M.C., *Estudo de um método de doseamento de compostos voláteis em vinagres por cromatografia em fase gasosa*, Relatório dactil., Estação Vitivinícola Nacional, Dois Portos (1993). (Study of a measurement method for volatile components in vinegars with gaseous chromatography).