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-methylpentan2-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).

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 □ 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 µL 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

- 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:

$$C_i = \frac{c_i S_e S_i c_e}{c_e S_i S_e} f$$

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).