Method OIV-MA-VI-19 : R2000
Type IV method

Measurement of methanol, superior alcohols and ethyl acetate in vinegars

(OENO 70/2000)

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

1. 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).