OIV-MA-AS313-14B Sorbic acid

Type IV method

1. Principle of Methods

Determination by gas chromatography

Sorbic acid extracted in diethyl ether is determined by gas chromatography using an internal standard.

2. Determination by gas chromatography

2.1. Apparatus

2.1.1. Gas chromatograph fitted with a flame ionization detector and a stainless steel column (4 m x 1/8 inch) previously treated with dimethyldichlorosilane and packed with a stationary phase consisting of a mixture of diethyleneglycol succinate, 5%, and phosphoric acid, 1%, (DEGS - H_3PO_4), or of a mixture of diethyleneglycol adipate, 7%, and phosphoric acid, 1%, (DEGA - H_3PO_4) bonded on Gaschrom Q 80 - 100 mesh.

Treatment of column with dimethyldichlorosilane (DMDCS): pass a solution containing 2 to 3 g of (DMDCS) in toluene through the column.

Immediately wash with methanol, followed by nitrogen and then wash with hexane followed by more nitrogen. The column is now ready to be packed.

Operating conditions:

- Oven temperature: 175 °C
- Temperature of the injector and detector: 230 °C.
- Carrier gas: nitrogen (flow rate = 200 mL/min)

Note: Other types of columns can also give a good separation, particularly capillary columns (e.g. FFAP). The working method described below is given as an example.

2.1.2. Microsyringe, 10 µL capacity graduated in 0.1 µL.

2.2. *Reagents*

2.2.1. Diethyl ether distilled just before use

2.2.2. Internal standard: solution of undecanoic acid, $C_{11}H_{22}O_2$, 1 g/L in ethanol,

95% (*v/v*)

2.2.3. Aqueous solution of sulfuric acid, H_2SO_4, (ρ$_{20}$ = 1.84 g/mL) diluted 1/3 (*v/v*)

2.3. *Procedure*

2.3.1. Preparation of sample to be analyzed

Into a glass test tube of approximately 40 mL capacity and fitted with a ground glass stopper, place 20 mL of wine, 2 mL of the internal standard (2.2.2) and 1 mL of dilute sulfuric acid.

After mixing the solution by repeatedly turning the tube over, add 10 mL of diethyl ether (2.2.1). Extract the sorbic acid into the organic phase by shaking the tube for five minutes. Allow to settle.

2.3.2. Preparation of the spiked sample

Select a wine for which the chromatogram of the ether extract shows no peak corresponding to the elution of sorbic acid. Fortify this wine with sorbic acid at a concentration of 100 mg/L. Treat 20 mL of the sample prepared in this way according to the procedure described in 2.3.1.

2.3.3. Chromatography

Inject 2 µL of the ether-extract phase obtained in 2.3.2, into the chromatograph using a microsyringe, followed by 2 µL of the ether-extracted phase obtained in 2.3.1.

Record the respective chromatograms: check the identity of the respective retention times of the sorbic acid and the internal standard. Measure the height (or area) of each of the recorded peaks.

2.4. *Expression of results*

2.4.1. Calculation

The concentration of sorbic acid in the analyzed wine, expressed in mg/L, is given by:

$$100 \times \frac{h}{H} \times \frac{I}{i}$$

where

H = height of the sorbic acid peak in the spiked solution

h = height of the sorbic acid peak in the sample for analysis

I = height of the internal standard peak in the spiked solution

i = height of the internal standard peak in the sample for analysis

Note: The sorbic acid concentration may be determined in the same way from measurements of the respective peak areas.

Bibliography

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