OIV-MA-AS315-09 Diethylene glycol (2-hydoxy-ethxyethanol)

Type IV method

1. Objective

The detection of diethylene glycol, $HOCH_2CH_2OCH_2CH_2OH$, in wine where its concentration is equal to or greater than 10 mg/L.

2. Principle

Separation of diethylene glycol from other constituents in wine by gas chromatography using a capillary column, after extraction with ether.

Note: The operating conditions described below are provided as an example.

3. Apparatus

3.1. Gas chromatograph equipped with:

- split-splitless injector,
- flame ionization detector,
- capillary column coated with a film of polyethyleneglycol (Carbowax 20 M), 50 m x 0.32 mm I.D.

Operating conditions:

- Injector temperature: 280°C.
- Detector temperature: 270°C.
- Carrier gas: hydrogen.
- Flow rate of carrier gas: 2 mL/min.
- Flow rate: 30 mL/min.
- Injection: splitless.

- Injection volume: 2 µL.
- Injection 35°C - flow closed after 40 seconds.
- Temperature program: 120°C to 170°C at 3°C/min.

2. Centrifuge

4. Reagents

4.1. 1,3-propanediol, 1 g/L, in alcohol, 20% (*v/v*), (internal standard).

4.2. Aqueous solution of diethyleneglycol 20 mg/L.

5. Procedure

Into a 50 mL flask, place:

- 10 mL of wine
- 1 mL of 1,3-propanediol solution
- 25 mL diethyl ether.

Shake and add sufficient quantity of neutral potassium carbonate to saturate the mixture. Shake. Separate the two phases by centrifugation.

Carry out a second extraction. Eliminate the diethyl ether by evaporation and recover the residue with 5 mL ethanol.

The yield of the extraction must be at least 90%.

Carry out the chromatography according to the conditions given in 3.1.

6. Results

The diethylene glycol is identified by comparing its retention time to the time of the reference solution, analyzed under the same conditions as the wine.

The amount is determined by comparison to the reference solution using the internal standard method.

It is recommended, if the concentration is equal to or less than 20 mg/l, to confirm the presence by mass spectrometry.

Bibliography

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