

**OIV-MA-AS315-02B Ethyl acetate**

## Type IV method

**1. Principle of the method**

Ethyl acetate is separated by distillation of wine brought to pH 6.5. After saponification and suitable concentration in an alkaline environment, the distillate is acidified and the vapor condensed to separate the acetic acid liberated by saponification; the acid portion is titrated with the alkaline solution.

**2. Method***2.1 Reagents*

2.1.1. Sodium hydroxide solution, 1 M

2.1.2. pH 6.5 Buffer solution

Potassium *di*-hydrogen phosphate,  $KH_2PO_4$  5 g

Sodium hydroxide solution 1 M 50 mL

Water to 1 L

2.1.3. Crystalline tartaric acid

2.1.4. Sodium hydroxide solution, 0.02 M

2.1.5. Neutral phenolphthalein solution, 1%, in alcohol, 96% (v/v).

*2.2. Usual method*

Into a 500 mL volumetric flask, place 100 mL of non-decarbonated wine neutralized with  $n$  mL of 1 M sodium hydroxide solution,  $n$  being the volume of sodium hydroxide solution, 0.1 M, used for measuring the total acidity of 10 mL of wine. Add 50 mL of pH 6.5 buffer solution and distill. The distillation must be conducted using a tapered tube into a 500 mL round-bottom flask containing 5 mL of 1 M sodium hydroxide solution, on which a mark has been made indicating a volume of approximately 35 mL. Collect 30 mL of distillate.

Stopper the flask and allow to stand for one hour. Concentrate the contents of the flask to approximately 10 mL by placing it in a boiling water bath and blowing a rapid stream of air into the bowl of the flask. Allow to cool. Add 3 g tartaric acid (2.1.3). Eliminate carbon dioxide by shaking under a vacuum. Transfer the liquid from the

# COMPENDIUM OF INTERNATIONAL METHODS OF WINE AND MUST ANALYSIS

## Ethyl Acetate (titrimetry) (Type-IV)

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concentrating flask to the bubbling chamber of a steam distillation apparatus and rinse the flask twice with 5 mL of water. Steam distill and recover at least 250 mL of distillate.

Titrate with a 0.02 M sodium hydroxide solution, in the presence of phenolphthalein.

### 2.3. Calculation

Let  $n$  be the number of milliliters of sodium hydroxide solution, 0.02 M (2.1.4) used. 1 mL corresponds to 1.76 mg ethyl acetate.

The concentration of ethyl acetate in milligrams per liter is given by:

$$17.6 \times n$$

### Bibliography

#### *Usual method:*

- PEYNAUD E., *Analyse et contrôle des vins*, Librairie Polytechnique Ch.-Béranger, 1958.