OIV-MA-F1-09 Specific methods for the analysis of grape sugar

Type I method

ANNEX A: TOTAL CATIONS

1. Principle

The test sample is treated by a strongly acid cation exchanger. The cations are exchanged with H^+. Total cations are expressed by the difference between the total acidity of the effluent and that of the test sample.

2. Apparatus

2.1. Glass column of internal diameter 10 to 11 mm and length approximately 300 mm, fitted with a drain tap.

2.2. pH meter with a scale graduated at least in 0.1 pH units.

2.3. Electrodes:

- glass electrode, kept in distilled water,
- calomel/saturated potassium chloride reference electrode, kept in a saturated solution of potassium chloride,
- or a combined electrode, kept in distilled water.

3. Reagents

3.1. Strongly acid cation exchange resin in H^+ form pre-swollen by soaking in water overnight.

3.2. Sodium hydroxide solution, 0.1 M.

3.3. Paper pH indicator.

The water used must be purified water for laboratories, with specific conductivity below 2 µS cm-1 at 20°C, for example EN ISO 3696 type II water.

4. Procedure

The pH meter must be calibrated according to the method OIV MA AS313-15

4.1. Preparation of sample

Use the solution obtained by diluting the rectified concentrated must to 40% (m/v). Introduce 200 g of accurately weighed rectified concentrated must. Make up to the mark with 500 ml water. Homogenize.

4.2. Total acidity of the rectified concentrated must

Titrate the acidity of the concentrated must in 100 ml of sample prepared as in 4.1 with the 0.1M sodium hydroxide solution until the pH is equal to 7 at 20 °C. The alkaline solution should be added slowly and the solution continuously shaken. Let n1 ml be the volume of 0.1 M sodium hydroxide solution used.

4.3. Preparation of the ion exchange column

Introduce into the column approximately 10 ml pre-swollen ion exchanger in H^+ form. Rinse the column with distilled water until all acidity has been removed, using the paper indicator to monitor this.

4.4. Ion exchange

Pass 100 ml of the rectified concentrated must solution prepared as in paragraph 4.1 through the column at the rate of one drop every second. Collect the effluent in a beaker. Rinse the column with 50 ml of distilled water. Titrate the acidity in the effluent (including the rinse water) with the 0.1 M sodium hydroxide solution until the pH is 7 at 20°C. The alkaline solution should be added slowly and the solution continuously shaken. Let n2 ml be the volume of 0.1 M sodium hydroxide solution used.

5. Expression of the results

The total cations are expressed in milliequivalents per kilogram of total sugar to one decimal place.

5.1. Calculations

Total acidity of the rectified concentrated must in milliequivalents per kilogram:

- a=2.5 n1

Acidity of the effluent expressed in milliequivalents per kilogram of rectified concentrated must:

- E= 2.5 n2.

Total cations in milliequivalents per kilogram of total sugars:

$$(n2 - n1)/(P)) \times 250$$

- P=percentage concentration (m/m) of total sugars.

5.2. Repeatability (r)

- r = 0.3