RESOLUTION OIV-OENO 662G-2025

DETERMINATION OF TOTAL ACIDITY IN GRAPE JUICE, RECONSTITUTED GRAPE JUICE, CONCENTRATED GRAPE JUICE, AND GRAPE NECTAR BY TITRATION

Type IV method

THE GENERAL ASSEMBLY,

IN VIEW of Article 2, paragraph iv of the Agreement of 3 April 2001 establishing the International Organisation of Vine and Wine,

AT THE PROPOSAL of the "Methods of Analysis" Sub-Commission,

CONSIDERING that for concentrated grape juice use is proposed the application of the method for the analysis of grape sugar (rectified concentrated musts) OIV-MA-F1-05 (Total Acidity) of the Compendium of International Methods of Analysis of Wines and Musts.

CONSIDERING that for grape juice, reconstituted grape juice, and nectar is proposed the application of the Method OIV-MA-AS313-01 (Total Acidity) of the Compendium of International Methods of Analysis of Wines and Musts.

DECIDES to add the following method:

DETERMINATION OF TOTAL ACIDITY IN GRAPE JUICE, RECONSTITUTED GRAPE JUICE, CONCENTRATED GRAPE JUICE, AND GRAPE NECTAR BY TITRATION

1. Scope

The present method describes an analytical procedure for the determination of the total acidity of grape juice, reconstituted grape juice, concentrated grape juice, and grape nectar between 12.6 meq/L to 145.7 meq/L.

2. Definition

The total acidity of the grape juice, reconstituted grape juice, concentrated grape juice and grape nectar is the sum of its titratable acidities when it is titrated to pH 7.0 against a standard alkaline solution.

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Carbon dioxide is not included in the total acidity.

3. Principle

Potentiometric titration (preferably) or titration with bromothymol blue as an indicator and comparison with an end-point colour standard.

Note: Preference should be given to potentiometric titration as it is a more sustainable approach.

4. Reagents and materials

4.1. Reagents

4.1.1. Monopotassium phosphate (KH_2PO_4) CAS [7778-77-0].

4.1.2. Sodium hydroxide (NaOH) 1 M CAS [1310-73-2].

4.1.3. Potassium hydrogen phthalate ($C_8H_5KO_4$) CAS [877-24-7].

4.1.4. Bromothymol blue ($C_{27}H_{28}Br_2O_5S$) CAS [76-59-5].

4.1.5. Neutral ethanol 96 % v/v (C□H□OH) CAS [64-17-5].

4.1.6. Purified water for laboratory of equivalent quality according to EN ISO 3696.

4.2. Preparation of solutions

4.2.1. pH 7.0 buffer solution (1 L):

- Monopotassium phosphate (4.1.1): 107.3 g
- 1 M sodium hydroxide solution (4.1.2): 500 mL
- Water (4.1.6): to 1000 mL

4.2.2. pH 4.0 buffer solution (1L):

- Potassium hydrogen phthalate (4.1.3): 10.211 g
- Water (4.1.6): to 1000 mL

Note: Other calibration buffer solutions may be used to match the pH of the matrices to be analyzed. Commercial calibration buffer solutions traceable to the SI can also be

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used.

4.2.3. Solution of 0.1 M sodium hydroxide (1 L):

- 1 M sodium hydroxide solution (4.1.2): 100 mL
- Water (4.1.6) .to 1000 mL

4.2.4. Solution of 4 g/L bromothymol blue indicator (1 L):

- Bromothymol blue (4.1.4) : 4 g
- Neutral ethanol 96 % v/v (4.1.5): 200 mL

Dissolve and add:

- Water (4.1.6): 200 mL
- 1 M sodium hydroxide solution (4.1.2) sufficient to produce blue-green colour (pH 7.0) approximately: 7.5 mL
- Water (4.1.6) : to 1000 mL

Note: commercial solutions can be used for all of the solutions described.

5. Apparatus

5.1. Potentiometer with scale graduated in pH values, and electrodes
5.2. Vacuum system
5.3. 500 mL Vacuum flask
5.4. 250 mL and 600 mL Beakers or any appropriate recipient
5.5. 10 mL volumetric pipettes
5.6. 50 mL volumetric pipettes
5.7. Burettes
5.8. Agitator
5.9. Balance with 0.1 mg resolution

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6. Procedure

6.1. Preparation of sample

6.1.1. For concentrated grape juice:

Make up the concentrated grape juice to 40 % w/w. As example, introduce 200 g of accurately weighed concentrated grape juice in a 600 mL beaker. Make up to 500 g with water (4.1.6) and homogenize. (dilution factor F = 2.5).

6.1.2. For gasified grape juice, reconstituted grape juice, and grape nectar:

Place approximately 50 mL of the sample in a vacuum flask; apply vacuum to the flask using a vacuum system for one to two minutes while shaking continuously. Other CO_2 elimination systems may be used if the CO_2 elimination is guaranteed.

6.2. Potentiometric titration

6.2.1. Calibration of the pH meter

The pH meter must be calibrated to be fit for the purpose of the method.

6.2.2. Method of measurement

Into a beaker (5.4), introduce a 10 mL of the sample, prepared as described in 6.1.2 or 50 ml of the sample, prepared as described in 6.1.1. Add about 10 mL of distilled water (4.1.6) without CO2 and titrate with sodium hydroxide solution, 0.1 M (4.2.3) with a burette until the pH is equal to 7.0 at 20 °C. The sodium hydroxide solution must be added slowly, and the solution stirred continuously. Let *n* mL be the volume of sodium hydroxide 0.1 M added.

Note: volume adjustments can be made to enable the use of automatic titrators

6.3. Titration with indicator (bromothymol blue)

6.3.1. Preliminary test: end-point colour determination.

Into a beaker (5.4) place 25 mL of water (4.1.6), 1 mL of bromothymol blue solution (4.2.4) and 10 mL of the sample prepared as in 6.1.2 or 50 mL of the sample, prepared as described in 6.1.1. Add the 0.1 M sodium hydroxide solution (4.2.3) until the colour changes to blue-green. Then add 5 mL of the pH 7 buffer solution (4.2.1).

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6.3.2. Measurement

Into a beaker (5.4) place 30 mL of water (4.1.6), 1 mL of bromothymol blue solution (4.2.4) and 10 mL of the sample, prepared as described in 6.1.2. or 50 mL of the sample, prepared as described in 6.1.1. Add 0.1 M sodium hydroxide solution (4.2.3) until the same colour is obtained as in the preliminary test above (6.3.1). Let *n* mL be the volume of 0.1 M sodium hydroxide solution added.

7. Calculation and expression of results

7.1. Method of calculation for concentrated grape juice

- The total acidity expressed in milliequivalents per kilogram (meq/Kg) of concentrated juice is given by:

A = (1000 x *n* x M / v) x F

A = 5 x *n*;

n = volume (mL) of 0.1 M sodium hydroxide solution used

M = NaOH molarity – 0.1 M

v = sample volume – 50 mL

F = dilution factor – 2.5

- The total acidity expressed in milliequivalents per kilogram (meq/Kg) of total sugars is given by:

A = (500 x *n*)/P;

P = % concentration (w/w) of total sugars.

n = volume (mL) of 0.1 M sodium hydroxide solution used

The results are quoted to one decimal place.

7.2. Method of calculation for grape juice, reconstituted grape juice, and grape nectar

- The total acidity expressed in milliequivalents per litre (meq/l) is given by:

A = 10 *n.*

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n = volume (mL) of 0.1 M sodium hydroxide solution used
It is recorded to one decimal place.

- The total acidity expressed in grams of tartaric acid per litre is given by:

A' = 0.075 x A
A = total acidity expressed in milliequivalents per litre (meq/l)
The result is quoted to two decimal places.

- The total acidity expressed in grams of sulfuric acid per litre is given by:

A' = 0.049 x A
A = total acidity expressed in milliequivalents per litre (meq/l)
The result is quoted to two decimal places.

8. Method characteristics

A validation study was carried out for the potentiometric titration approach regarding the precision of the method.

8.1. Precision of the method

The parameters taken into account were repeatability (r) and intermediate precision (PI). The repeatability was expressed as standard deviation of repeatability (Sr) of 21 measurements in a red grape juice. The intermediate precision (IP) was expressed as the Standard Deviation (SIP) of 15 measurements of the same red grape juice sample, in different days. Table 1 shows the values of these parameters.

T*able 1. Characteristics of the method*

Linearity range (meq/L)	Correlation coefficient (r^2)	Repeatability standard Deviation (Sr) (meq/L)	Intermediate precision Standard Deviation (SIP) (meq/L)
12.6 – 145.7	0.9994	0.8	2.1

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9. Bibliography

1. OIV, Compendium of International Methods of Analysis of Wines and Musts. Method OIV-MA-F1-05 (for concentrated grape juice).
2. OIV, Compendium of International Methods of Analysis of Wines and Musts. Method OIV-MA-AS313-01 (for grape juice, reconstituted grape juice, and nectar).
3. BS EN ISO 3696, Water for Analytical Laboratory Use — Specification and Test Methods, 1995.

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