OIV-MA-F1-03 Determination of the acquired alcoholic strength by volume (ASV) of concentrated musts (CM) and grape sugar (or rectified concentrated musts, RCM)

Type IV method

1. Introduction

Concentrated musts (CM) and grape sugar (RCM) are viscous products with low alcohol contents; to determine their acquired ASV, a method must be used, the characteristics of which (linearity, repeatability, reproducibility, specificity, and detection and quantification limits) must be such that it is possible to measure alcohol contents of less than 1% vol.

2. Field of application

The method applies to concentrated musts and grape sugar.

3. Principle

A known mass of concentrated must (CM) or grape sugar is made alkaline by a suspension of calcium hydroxide and then distilled. The alcoholic strength by volume of the distillate is determined by electronic densitometry or by densitometry using a hydrostatic balance.

4. Reagents

- Suspension of 2M calcium hydroxide of analytical quality obtained by carefully pouring one litre of hot water (60°C to 70°C) on to about 120 g of unslaked lime (CaO).
- Antifoam solution obtained by dilution of 2 ml of concentrated silicone antifoam in 100 ml of water.
- Purified water for laboratory use and of quality EN ISO 3696.

5. Equipment

- Standard laboratory equipment including volumetric flasks
- Analytic balance capable of weighing to within 0.1 g.
- Any type of distillation or steam distillation apparatus may be used provided that

it satisfies the following test:

- Distil an ethanol-water mixture with an alcoholic strength of 10% vol. five times in succession. The distillate should have an alcoholic strength of at least 9.9% vol. after the fifth distillation; i.e. the loss of alcohol during each distillation should not be more than 0.02% vol.
- Electronic density meter or hydrostatic balance.

6. Procedure

- Homogenise the test sample by inverting the flask several times.
- In a 500 ml volumetric flask, weigh about 200 g of concentrated must or rectified concentrated must (to within 0.1 g). Note the weight (TS) of this test sample. Fill up to the mark with deionised water. This solution is about 40% m/v in must.

Obtaining the distillate

- Transfer 250 ml of the 40% solution to the distillation flask, add to the flask about 10 ml of calcium hydroxide in suspension, about 5 ml of antifoam solution and, where applicable, a boiling regulator (e.g. pieces of porcelain).
- Gently bring to the boil.
- Recover the distillate in a 100 ml volumetric flask (about 90 ml).
- Leave the distillate to return to ambient temperature, then fill up to the mark with deionised water.

Measurement of ASV

This is performed by electronic densitometry or by hydrostatic balance.

7. Calculation

$$Acquired\ alcoholic\ strength\ by\ volume = \frac{ASV\ measured\ \times 200 \times MV}{TS}$$

- ASV measured = alcohol content given by the density meter, as % vol.
- TS = test sample of concentrated must or grape sugar, in weight.
- MV= density of concentrated must or rectified concentrated must, in g/ml

The results are expressed to 2 decimal places and rounded to within 0.05 %vol.

8. Characteristics of the method

8.1. Linearity of response

The linearity of the density meter for low ASV values is one of the critical parameters of this method. A standard range of 10 aqueous-alcoholic solutions of ASV ranging between 0 and 5%vol. was prepared. Each solution was analysed 3 times.

The response of the density meter is perfectly linear within this range as shown by the calibration line in Figure 1.

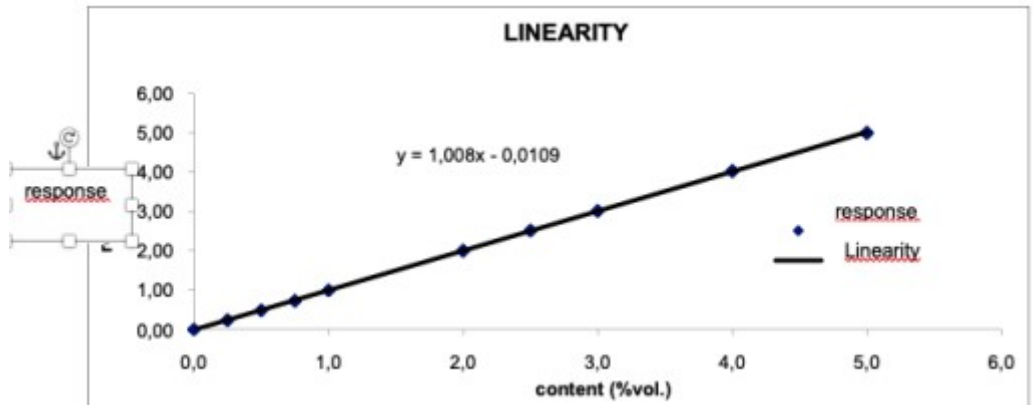


Figure 1: Linearity of determination of the ASV by electronic densitometry between 0 and 5% level

8.2. Specificity of the method

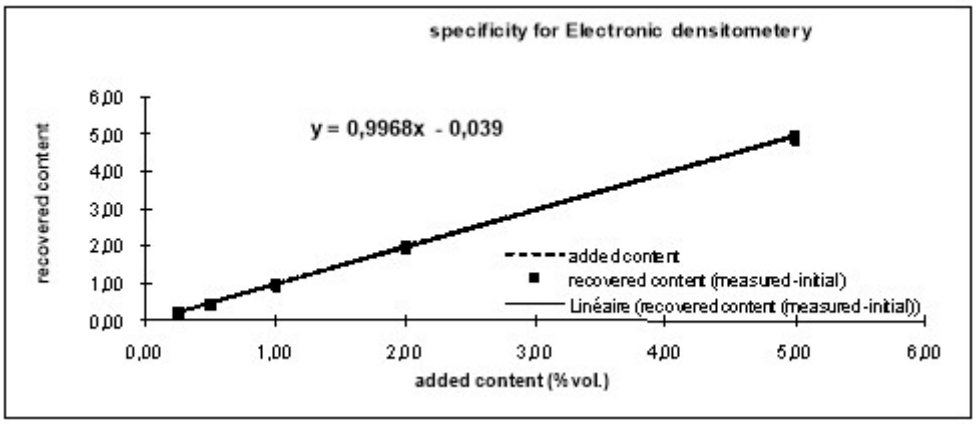
The second critical point of this method is the distillation of viscous musts containing small quantities of alcohol. In order to verify the specificity, known quantities of ethanol (from 0.25% vol to 5% vol) were added to CMs and grape sugar. The supplemented test specimens were distilled in the conditions defined earlier, then the distillates were analysed by electronic densitometry or by hydrostatic balance.

The results are shown in Table 1. The recovery rate is satisfactory, ranging between 88% and 99%. As shown by the line in Figure 2, the method is specific (slope in the vicinity of 1, intercept point in the vicinity of 0).

Table 1: Recovery rate for determination of the acquired ASV of CMs and Grape Sugar

Test specimen	Initial content (%vol.)	Added content (%vol.)	Recovered content (%vol.)	Recovery rate (%)

CM 1	0.00	0.25	0.22	88
CM 1	0.00	1.00	0.98	98
Grape Sugar (RCM) 1	0.00	1.00	0.94	94
Grape Sugar (RCM) 1	0.00	2.00	1.97	99
CM 2	0.00	0.50	0.44	88
Grape Sugar (RCM)2	0.00	5.00	4.94	99



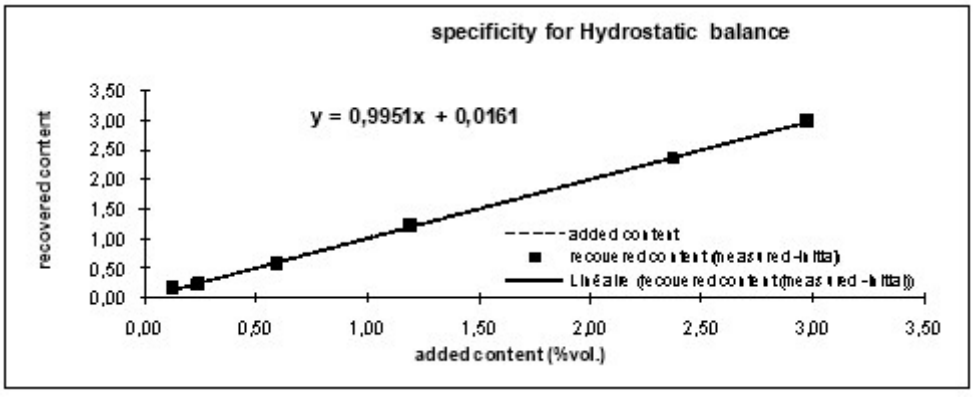


Figure 2 : Specificity of determination of the acquired ASV of CMs and Grape Sugar

8.3. Repeatability

The repeatability of the method was determined using 20 test specimens of CM or grape sugar supplemented with alcohol or not. Each CM or RCM test specimen was analysed 3 times, in order to ensure identical conditions. The repeatability limits obtained are as follows:

Table 2: Repeatability of determination of the acquired ASV of CMs and Grape Sugar

Repeatability for electronic densitometry	Calculated value
Standard deviation	0.009
CV or RSD as %	0.9%
r limit	0.024 %vol.
r limit as %	3%
Repeatability for Hydrostatic balance	**Calculated value**
Standard deviation	0.013
CV or RSD as %	1.7%
r limit	0.038 %vol.
r limit as %	5,3%

8.4. Reproducibility

The reproducibility of the results is determined by analysing the same must twice, at different dates during a given period of time. The results are given in Table 3.

Table 3 - Reproducibility of determination of the acquired ASV of CMs and grape sugar

Reproducibility for electronic densitometry	Calculated value
Standard deviation	0.043
CV or RSD as %	3%
R limit	0.12%vol.
R limit as %	9%

Reproducibility for Hydrostatic balance	Calculated value
Standard deviation	0.026
CV or RSD as %	3.4%
R limit	0.076%vol.
R limit as %	10.6%

8.5. Detection and quantification limits

The limits of detection (LD) and quantification (LQ) estimated based on the linearity study are as follows:

- LD = 0.01%vol.
- LQ = 0.05%vol.

The quantification limit was verified by analysis of musts having an ASV at a concentration level of 0.05%vol.

8.6. Uncertainty

Uncertainty, evaluated based on the reproducibility standard deviation, is 0.10%vol.