RESOLUTION OIV-OENO 683-2022

UPDATE TO METHOD OIV-MA-AS323-02B – Quantification of total nitrogen according to the Dumas method (musts and wines)

IMPORTANT: This draft resolution modifies the following resolution:
- OENO 13/2002

THE GENERAL ASSEMBLY,

IN VIEW OF Article 2, paragraph 2 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 the proposed modifications are intended to improve the method performance,

DECIDES to modify the resolution OENO 13/2002 and consequently the method OIV-MA-AS323-02B in Annex A of the Compendium of International Methods of Analysis of Wines and Must*s* by making the following amendments:

Method OIV-MA-AS323-02

Type IV Method

UPDATE TO METHOD OIV-MA-AS323-02B

Quantification of total nitrogen according to the Dumas method (musts and wines)

(Resolution OENO 13/2002)

1. Field of application

This method applies to the analysis of total nitrogen in musts and wine up to 1000 mg/L.

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2. Principle

The analysis of total nitrogen in an organic matrix can be carried out using the Dumas method. This involves the total combustion of the matrix in the presence of oxygen at a temperature higher than 900°C, followed by an additional oxidation. The nitrogen oxides are then reduced in dinitrogen, which is quantified using a thermal conductivity detector (katharometer), after elimination of the other oxides, water traces and carbon dioxide.

The nature and positioning of the traps varies according to the type of material.

3. Reagents and materials

3.1. Nitrogen (technical quality)

3.2. High-purity helium for GC (e.g. $H_2O \leq 3$ ppm; $O_2 \leq 2$ ppm; CnHm ≤ 1 ppm; $N_2 \leq 5$ ppm)

3.3. High-purity oxygen for GC (e.g. $H_2O \leq 3$ ppm; Ar ≤ 3 ppm; CnHm $\leq 0,2$ ppm; $N_2 \leq 5$ ppm)

3.4. Demineralised water (e.g. ISO 3696 type I or HPLC grade)

3.5. Oxidant (e.g. copper oxide [1317-38-0], chromium sesquioxide [1308-38-9] or silvered cobaltous/cobaltic oxide [1308-06-1])

3.6. Reducer (e.g. copper [7440-50-8])

3.7. Dehydrating agents (e.g. sodium hydroxide on silica or mixed with quartz crystals [1310-73-2], anhydrous magnesium perchlorate [10034-81-8] or calcium sulfate [7778-18-9])

3.8. Product for calibration range (e.g. tris(hydroxymethyl)aminomethane [77-86-1] or atropine [51-55-8])

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3.9. Internal standard (e.g. glutamic acid hydrochloride [138-15-8], or a sample from an inter-laboratory proficiency-testing programme)

4. Apparatus

4.1. Centrifuge

4.2. ultrasonic bath

4.3. Total nitrogen analyser optionally equipped with a sample changer

4.4. Precision balance with precision of ± 0.01 mg between 0.5 mg and 30 g

5. Sampling

If the sample contains a significant amount of carbon dioxide, degas for example by bubbling nitrogen (3.1) for 5 to 10 min or by using an ultrasound bath. For musts, centrifuge (4.1).

6. Procedure

6.1. Preparation of the calibration range (given by way of example)

From a 1 g N/L solution, prepare five samples of TRIS (3.8) at 800, 500, 250, 100 and 50 mg N/L. The calibration curve therefore passes through seven points, from a blank to the 1 g N/L solution and follows a linear model. It is recorded in the instrumental method. Analyse the 500 mg N/L standard before starting each analytical sequence, and as soon as necessary to adjust the analyser.

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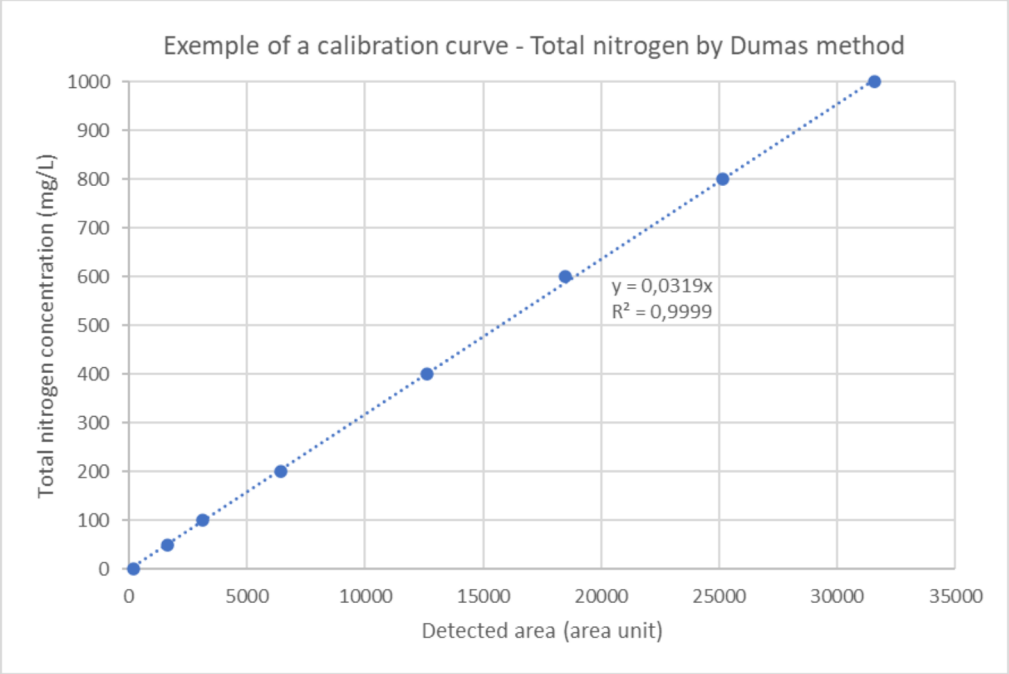

6.2. Preparation of control samples (given by way of example)

Control samples are used regularly at the beginning and in the middle of analysis series. They can be made up using the hydrochloride form of glutamic acid (3.9) at 150, 300 and 600 mg N/L in demineralised water (3.4).

$$C_{glutamic\ acid} = \frac{M_{glutamic\ acid} \times C_N}{M_N}$$

With :

- $C_{glutamic\ acid}$ the glutamic acid concentration in the solution, expressed in g/L
- C_N the nitrogen concentration in the solution, expressed in g/L
- $M_{glutamic\ acid}$ the molar mass of glutamic acid. $M_{glutamic\ acid}$ = 183,59 g/mol
- M_N the molar mass of nitrogen. M_N = 14,007 g/mol

Weigh (4.4) 7.864 g of glutamic acid (3.9) and dilute in demineralised water (3.4) and

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make up to 1 L to obtain a 600 mg N/L solution. Dilute by 50% by transferring 250 mL of this solution and diluting to 500 mL to obtain a 300 mg N/L solution. Repeat with 250 mL of the 300 mg N/L solution to 500 mL to obtain a 150 mg N/L solution.

6.3. Preparation of samples

Weigh (to the nearest 0.01 mg) the volume defined for the analysis of the must or wine with the precision balance (4.4). Place the samples on the analyser awaiting analysis.

6.4. Analysis of samples

Carry out the analyses of samples and standards as per instrument manufacturer's instructions.

7. Expression of results

The results are expressed in mg/L of nitrogen, rounded to the nearest whole number.

8. Precision

Number of laboratories	Average contents	Repeatability r	Reproducibility R
11	591 mg/L	43 mg/L	153 mg/L

9. Bibliography

1. DUMAS, A.: *Annales de chimie*, vol. 33, 1826, p.342.
2. Buckee, G. K.: Determination of total nitrogen in Barley, Malt and Beer by Kjeldahl procedures and the Dumas combustion method, Collaborative trial. J. Inst. Brew., vol. 100, 1994, pp. 57-64.
3. ISO 3696: Water for analytical laboratory use — Specification and test methods.
4. Instrument manufacturer's instruction manual, or equivalent.

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