Method OIV-MA-BS-28 : R2009

Type IV method

Measurement of turbidity by nephelometry in spirit drinks of viti-vinicultural origin

OENO 6/94

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1. Purpose

The purpose of the present document is to describe an optical method for determining the turbidity index (or diffusion index) of an alcohol or alcoholic beverage.

2. Scope

The method described is applicable to alcohol and alcoholic beverages with a high optical purity and containing low quantities of suspensoid material.

Its application is of less interest in liquids containing suspended solids (very turbid).

3. Principle

Turbidity is an optical effect.

The diffusion index is an intrinsic property of liquids used to characterize their optical appearance.

This optical effect is caused by the presence of very fine particles distributed in a liquid dispersion medium; the refractive index of the particles differs from that of the dispersion medium.

If optically clean water contained in a known volume is illuminated and if we measure the luminous flux diffused by the incident beam, the value read for this diffused flux characterizes the molecular diffusion of water.

If the value obtained with the water analyzed is greater than that corresponding to the molecular diffusion, which is constant for a given wavelength, for the same incident flux from the same angle of measurement, for the same tank geometry and a given

temperature, the difference is attributed to the light diffused by the solid, liquid or gaseous particles suspended in the water.
The measurement of diffused luminous flux, performed as described, is a nephelometric measurement.

4. Definition

4.1. Unit of expression of the turbidity index

The turbidity unit used is the N T U - NEPHELOMETRIC TURBIDITY UNIT which corresponds to the measurement of light diffused by a standard suspension of Formazine, with a value of 1 NTU, at an angle of 90° with respect to the direction of the incident beam.

4.2. Preparation of the standard Formazine suspension.

4.2.1. Water for the preparation of control solutions.

Soak a filter membrane with pore size of 0.1 microns (the type used in bacteriology) for 1 h in 100 ml of distilled water. Filter 250 ml of distilled water twice through the membrane and retain the water for the preparation of the standard solutions.

4.2.2. Formazine ($C_2H_4N_2$) solutions.

The combination referred to as Formazine, the formula for which is $C_2H_4N_2$, is not commercially available. It is obtained by means of the following solutions:

- Solution A: Dissolve 10.0 g of hexamethylenetetramine (formula $CH_2)_6N_4$ in distilled water prepared according to 4.2.1. Then fill to 100 ml with distilled water prepared according to 4.2.1
- Solution B: Dissolve 1.0 g of hydrazinium sulphate $N_2H_6SO_4$ in distilled water prepared according to 4.2.1. Then fill to 100 ml with distilled water prepared according to 4.2.1.

Warning*:* Hydrazinium sulphate is poisonous and may be carcinogenic*.*

4.3. Procedure

Mix 5 ml of solution A and 5 ml of solution B. After 24 h at 25°C ± 3°C, dilute the solution to 100 ml with water. (4.2.1).

The turbidity of this standard solution is: 400 NTU

In the dark, the standard suspension can be kept at room temperature for approximately 4 weeks.

By dilution to 1/400 with distilled water according to 4.2.1, we obtain a turbidity of 1 NTU.

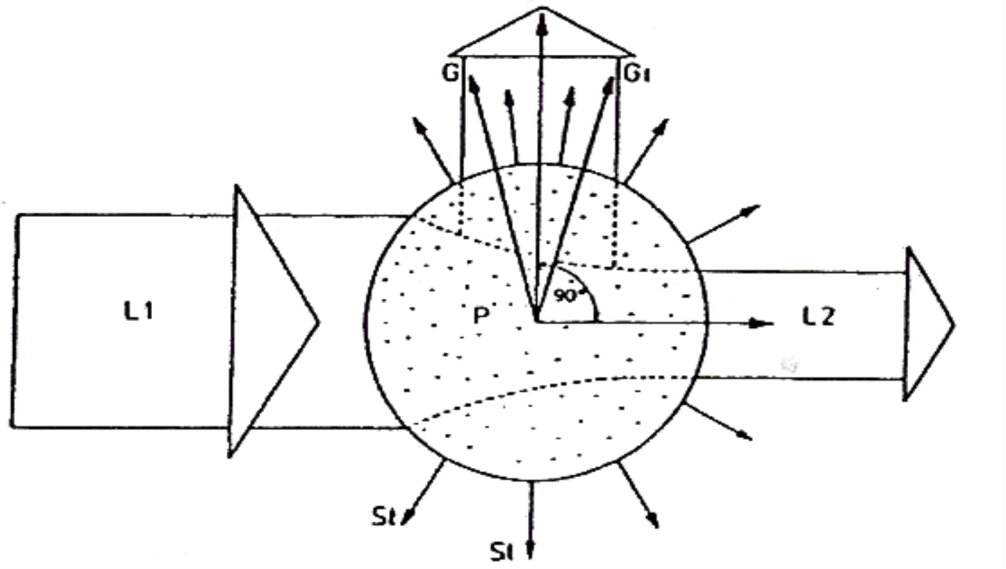
This solution is only stable for one week.

NB: Formazine standards have been compared to standards based on polymers.

The discrepancies observed may be considered to be negligible. Polymer-based standards have the following disadvantages, however: their cost is very high and their service life is limited. They must be handled carefully to avoid breaking the polymer particles, which changes the turbidity index. This possibility is offered as an alternative to Formazine.

4.4. Principle of optical measurement

Measuring principle

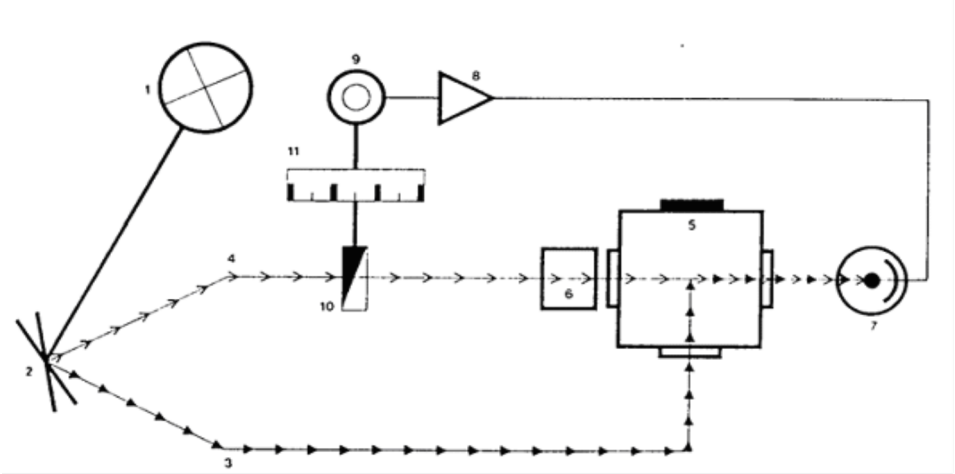


- L1 =Incident light beam
- L2 = beam having passed through the sample
- P = Sample
- St = diffused light
- G/G1 = limit rays of the diffused light beam, used for the measurement
- G/GL: Limit rays of the diffused beam of light for measurement.
- Observation of the diffused light should be at 90 degrees in relation to the direction of the incident beam.

5. Apparatus

This method refers to the use of apparatus making the measurement by optical compensation with two light beams.

5.1. Optical principle



A light source (1), powered by the mains, sends a beam of light onto the oscillating mirror (2) which reflects alternatively a measuring beam (3) and a comparison beam (4) about 600 times per second.

The measuring beam (3) is propagated in the fluid to be measured (5), while the

comparison beam (4) is propagated in an optically stable turbidity comparison standard (6).

The diffused light produced in the fluid (5) by the particles which generate turbidity and the light diffused by the comparison standard (6) are alternately received by a photoelectric cell (7).

This cell thus receives, at the same frequency, on the one hand a measuring beam (3), and on the other hand, a comparison beam (4) whose luminous intensities are different.

The photoelectric cell (7) transforms these unequal luminous intensities into photoelectric currents which are then amplified (8) and fed to a synchronous motor (9) acting as a servomotor.

By means of a mechanical measuring diaphragm (10), the servomotor varies the intensity of the comparison beam until the two rays reach the photo-electric cell with the same luminous intensity.

This state of equilibrium can be used to measure the solid content of the measured fluid.

The absolute value of the measurement depends on the dimensions of the comparison standard and the position of the diaphragm.

5.2. Range of measurement

The apparatus must enable measurement in the range of 0 to 50 NTU

5.3. Measuring ranges

The measurements ranging from 0 to 5 NTU must be performed by placing the test sample in a glass measuring cell of optical quality with the following dimensions: 60 x 60 mm, i.e. a minimum sample volume of 140 ml.

The measurements ranging beyond 5 NTU must be performed by placing the sample in a glass measuring cell of optical quality with the following dimensions: 35 x 35 mm, i.e. a minimum sample volume of 60 ml.

Measuring cells of different shape but with identical characteristics in terms of optical path length and volumes may be used.

5.4. Limit of detection of stray light

Part of the light beam is diffused at the entry, exit and other areas of the measuring

cell, including when measuring clear, clean water.

Due to the apparatus, this diffused light also reaches the photoelectric cell. This light, referred to as: stray light, creates a slight error in each measured value.

The apparatus must not induce an error due to stray light greater than: 0.01 NTU on the measuring range of: 0 to 0.1 N**Effect of colouring of the test sample**

The apparatus must compensate for the colour of the product to be measured without affecting the turbidity measurement up to the following absorbance values:

NTU Measuring ranges	Total admissible absorbance
0- 0.1	0.5
0 - 0.2	0.5
0 - 0.5	0.7
0 - 1.0	0.8
0 - 2.0	0.9
0- 5.0	1.1
0-10.0	1.2
0-20.0	1.3
0-50.0	1.5

5.6. Temperature conditions

The measurement should be made at a temperature of between 15 and 25°C

6. Procedure for measurement

6.1. Verification of the apparatus

Before any measurement or series of measurements, check the electrical and mechanical operation of the apparatus in accordance with the recommendations of the manufacturer.

6.2. Verification of calibration of the measurement scale

Before any measurement or series of measurements, using a previously calibrated apparatus, check the calibration of its measurement scale in accordance with its principle of construction.

6.3. Cleaning the measuring cell

Clean the measuring cell with great care before any determination. Take all the precautions required to avoid the introduction of dust into the apparatus and even more so in the measuring cell before and during the determination of the turbidity index.

6.4. Measurement

- Operate at a temperature as close as possible to 20°C. Prior to the measurement, thoroughly mix and without any sudden movements the flask containing the product to be measured,
- Thoroughly rinse the measuring cell twice using a small volume of the product to be measured,
- Introduce the product to be measured with care into the measuring cell, avoiding any turbulent flow which might lead to the formation of air bubbles and carry out the test measurement,
- Wait one minute if the index value is stable
- Note the turbidity index obtained.

7. Expression of results

The turbidity index of the product examined is recorded and expressed in NTU

8. Test report

The test report must state the results obtained, the references and identification of the sample, and all the test conditions, the type of apparatus used and all the operating details, whether optional or not provided in the method, and any incidents which may have influenced the results.

Appliances using the optical compensation method of measurement include solid turbidity standards in which the measuring light is diffused by a standard suspension of Formazine.

The solid standards are to have been checked beforehand by an optical laboratory.

It is recommended to periodically check the calibration using three standards distributed in the measurement range habitually used.

These three standard solutions are obtained by dilution of the standard suspension of Formazine described in section 4.10 of the method.

The standard liquids must be packaged in bottles made of glass or another inert material of low capacity (e.g. 200 ml) to prevent multiple successive manipulations from altering their purity. Keep the bottles in a cool, dark place. Any opened bottle should be carefully and quickly recapped after sampling the volume required for measurement. The sample must never be used for a second determination.

9. Bibliography

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