OIV-MA-AS2-08 Wine turbidity (Determination by Nephelometric Analysis)

Type IV method

1. Warning

Measurements of turbidity are largely dependent on the design of the equipment used. Therefore, comparative measurements from one instrument to another are not possible unless the same measuring principle is used.

The primary known sources of errors, which are linked to the type of turbidimeter employed, are:

- effect of stray light,
- effect of product color, especially in cases with low cloudiness values,
- electronic shifting due to aging electronic components,
- type of light source, photo detector and the dimensions and type of measurement the cell.

The present method uses a nephelometer incorporating a double beam with optical compensation design.

This category of instrument makes it possible to compensate for: electronic shift, fluctuations of mains voltage, and, in part, wine color. Furthermore, calibration is highly stable.

It should be noted that this method does not lend itself to a collation of data from various sources, given the impossibility of conducting an analysis in collaboration with others.

2. Purpose

The purpose of this document is to describe an optical method capable of measuring the turbidity (or diffusion) index of wine.

3. Scope of application

This method is used in the absence of instruments allowing a completely faithful duplication of measurements from one device to another, as well as full compensation for wine color. Therefore, findings are given for informational purposes only, and

must be considered with caution.

Above all, this technique is intended for use in production, where it is the most objective criterion of the measurement of clarity.

This method, which cannot be validated accordingly to internationally recognized criteria, will be classified as class 4[1].

4. General principle

Turbidity is an optical effect.

The diffusion index is an intrinsic property of liquids that makes it possible to describe their optical appearance. This optical effect is produced by the presence of extremely fine particles scattered in a liquid dispersion medium. The refraction index of these particles differs from that of the dispersion medium.

If a light is shown through a quantity of optically clean water placed in a container of known volume and the luminous flux diffused with respect to an incident beam is measured, the recorded value of this diffused flux will allow description of the molecular diffusion in the water.

If the value obtained for the water thus analyzed is greater than that of the molecular diffusion, which remains constant for a given wavelength, the same incident flux at the same angle measurement, in a tank of the same shape and at a given temperature, the difference can be attributed to the light diffused by solid, liquid or gaseous particles suspended in the water.

The measurement (taken as described) of the diffused luminous flux constitutes a nephelometric measurement.

5. Definitions

5.1. Turbidity

Reduction of the transparency of a liquid due to the presence of undissolved substances.

5.2. Units of Measurement of the Turbidity Index

The unit of turbidity used is: NTU - NEPHELOMETRIC TURBIDITY UNIT, which is the value corresponding to the measurement of the light diffused by a standard formazine suspension prepared as described under point 6.2.2, at a 90° angle to the direction of the incident beam.

6. Preparing the reference Formazine suspension ([1])

6.1. Reagents

All reagents must be of recognized analytical quality.

They must be stored in glass flasks.

6.1.1. Water for Preparing Control Solutions.

Soak a filter membrane with a pore size of 0.1 µm (like those used in bacteriology) for one hour in 100 ml of distilled water. Filter 250 ml distilled water twice through this membrane, and retain this water for preparation of standard solutions.

6.1.2. Formazine ($C_2H_4N_2$) Solutions

The compound known as formazine, whose formula is $C_2H_4N_2$, is not commercially available. It can be produced using the following solutions:

- Solution A: Dissolve 10.0 g hexamethylene-tetramine $(CH_2)_6N_4$ in distilled water prepared according to the instructions in 6.1.1. Then fill to a volume of 100 ml using distilled water.
- Solution B: Dissolve 1.0 g of hydrazinium sulfate, $N_2H_6SO_4$, in distilled water prepared according to the instruction in 6.1.1. Then fill to a volume of 100 ml using distilled water prepared according to 6.1.1.

Warning: Hydrazinium sulfate is poisonous and may be carcinogenic.

6.2. Working Method

Mix 5 ml of Solution A and 5 ml of Solution B. Dilute the solution to a volume of 100 ml with water after 24 hours at 25 °C $\pm$ 3 °C (6.1.1).

The turbidity of this standard solution is 400 NTU.

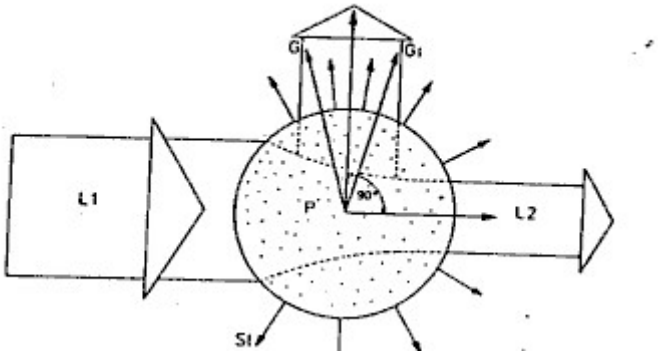
This standard suspension will keep for approximately 4 weeks at room temperature in the dark.

By diluting to 1/400 with recently prepared distilled water, a turbidity of 1 NTU will be obtained.

This solution remains stable for one week only.

N.B.: Standard formazine solutions have been compared to standard polymer-based solutions. The differences observed may be considered negligible. Nonetheless, polymer-based standard solutions have the following drawbacks: they are very expensive and they have a limited useful life. They must be handled with care to avoid breaking the polymer particles, as breakage would alter the turbidity value. Polymer use is suggested as an alternative to formazine.

7. Optical Measurement Principle



S1

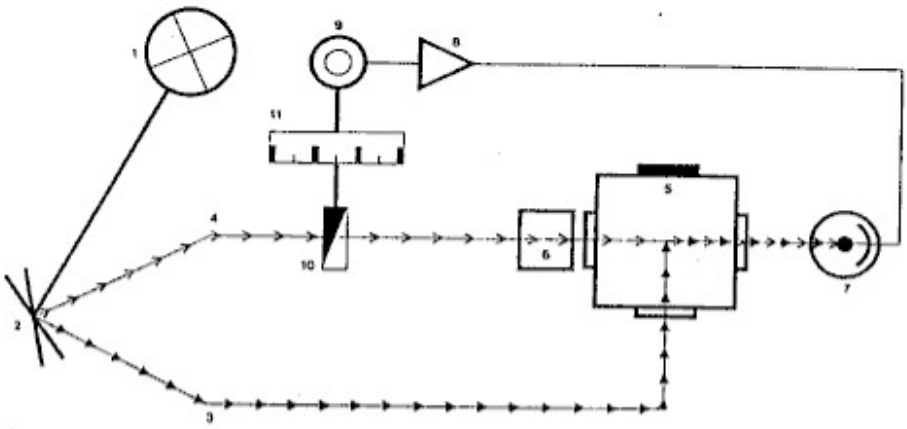
Measurement principle:

- L1= Incident light beam
- L2= Beam after passing through sample
- P= Sample
- St= diffused light
- G/G1 = Limiting rays from the diffused light beam used for measurement

The diffused light should be observed at an angle of 90° to the direction of propagation of the incident beam.

8. Instrumentation

1. Optical principle of the dual-beam and optical compensation nephelometer



A light source (1) powered by the electricity network projects a beam of light onto an oscillating mirror (2) which alternately reflects a measuring beam (3) and a comparison beam (4) at a rate of approximately 600 times per second.

The measuring beam (3) propagates through the fluid to be measured (5) while the comparison beam (4) propagates through an optically stable turbidity-comparison standard fluid (6).

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

Accordingly, this cell receives a measuring beam (3) and a comparison (4) having the same frequency, but different whose luminous intensities.

The photoelectric cell (7) transforms these unequal luminous intensities into electric current which are in turn amplified (8) and fed to a synchronous motor (9) functioning as a servo-motor.

This motor uses a mechanical measuring diaphragm (10) to vary the intensity of the control beam, until the two beams strike the photoelectric cell with equal luminous intensity.

This equilibrium state allows the solid particle content of the fluid to be determined.

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

8.2. Characteristics

Note: In order to take these measurements, regardless of the color of the wine, the nephelometer must be equipped with an additional interferential filter allowing measurement at a wavelength of 620 nm. However, the interferential filter is not needed if the light source is an infrared one.

8.2.1. The width of the spectral band of the incident radiation should be less than or equal to 60 nm.

8.2.2. There should be no divergence in the parallelism of the incident radiation, and convergence must not exceed 1.5°.

8.2.3. The angle of measurement between the optical axis of the incident radiation and that of the diffused radiation should be 90° $\pm$ 2.5°.

8.2.4. The apparatus must not cause error due to stray light greater than:

- NTU of random light error

within a range of:

- 0 to 0.1 NTU.

9. Operating method for measurement

9.1. Checking the Apparatus

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

9.2. Check Measurement Scale Adjustment

Before taking any measurement or series of measurements, use a previously calibrated instrument to check its measurement scale adjustment consistent with the principle underlying its design.

9.3. Cleaning the Measuring Unit

With the greatest care, clean the measuring tank before all analyses. Take all necessary precautions to avoid getting dust in the apparatus and especially in the measuring unit, before and during determination of the turbidity index.

9.4. Taking Measurements

The operating temperature should be between 15° and 25 °C (Take the temperature of the wine to be measured into consideration to ensure proper comparison). Prior to taking the measurement, carefully homogenize the product and, without making any abrupt movement that could create an emulsion, the flask holding the product to be analyze.

Carefully wash the measuring tank twice with a small amount of the product to be analyzed.

Carefully pour the product to be analyzed into the measuring tank, taking care to avoid any turbulence in the flow of the liquid, since this would lead to the formation of air bubbles. Carry out the test measurements.

Wait one minute if the index value is stable.

Record the resulting turbidity index.

10. Expressing the results

The turbidity index of the wine undergoing analysis is recorded and expressed in:

- NTU
- * if turbidity is less than 1 NTU, round off to 0.01 NTU
- * if turbidity is between 1 NTU and 10 NTU, round off to 0.1 NTU
- *if turbidity is between 10 NTU and 100 NTU, round off to 1 NTU

11. Test report

The test should contain the following information:

- reference to this method
- the results, expressed as indicated in 10
- any detail or occurrence that may have affected the findings.

Bibliography

- *AFNOR,* Standard NF EN 27027 (ISO 7027) - April 1994,"Water Quality = Turbidity Analysis"
- *OIV,* Compendium of International Methods for Spirits, Alcohols and the Aromatic Fractions in Beverages – 1994, "Turbidity – Nephelometric Analysis Method"
- SIGRIST PHOTOMETER SA, CH 6373 Ennetburgen, "Excerpts from technical instructions for nephelometers"

([1]) Care must be given to the precautions for handling, since Formazine is somewhat toxic.