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SN/T 4675.26—2016

出口葡萄酒浊度的测定 散射光法

Determination of turbidity of wine for export—Diffused radiation method

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前 言

SN/T 4675《出口葡萄酒质量安全分析方法》共分为 30 个部分：

- SN/T 4675.1 出口葡萄酒中甘油的测定 酶法；
- SN/T 4675.2 出口葡萄酒中 2,3-丁二醇的测定 气相色谱法；
- SN/T 4675.3 出口葡萄酒中乙醇稳定碳同位素比值的测定；
- SN/T 4675.4 出口葡萄酒中乳酸的测定 酶法；
- SN/T 4675.5 出口葡萄酒中有机酸的测定 离子色谱法；
- SN/T 4675.6 出口葡萄酒中葡萄糖、果糖和蔗糖的测定；
- SN/T 4675.7 出口葡萄酒中乙醛的测定 气相色谱-质谱法；
- SN/T 4675.8 出口葡萄酒中 5-羟甲基糠醛的测定 液相色谱法；
- SN/T 4675.9 出口葡萄酒中二甘醇的测定 气相色谱-质谱法；
- SN/T 4675.10 出口葡萄酒中赭曲霉毒素 A 的测定 液相色谱-质谱/质谱法；
- SN/T 4675.11 出口葡萄酒中 7 种花色苷的测定 超高效液相色谱法；
- SN/T 4675.12 出口葡萄酒中溶菌酶的测定 液相色谱法；
- SN/T 4675.13 出口葡萄酒中 2,4,6-三氯甲苯醚残留量的测定 气相色谱-质谱法；
- SN/T 4675.14 出口葡萄酒中纳他霉素的测定 液相色谱-质谱/质谱法；
- SN/T 4675.15 出口葡萄酒中水杨酸、脱氢乙酸和对氯苯甲酸的测定 液相色谱法；
- SN/T 4675.16 出口葡萄酒中富马酸的测定 液相色谱-质谱/质谱法；
- SN/T 4675.17 出口葡萄酒中丁基锡含量的测定 气相色谱-质谱/质谱法；
- SN/T 4675.18 出口葡萄酒中二硫代氨基甲酸酯残留量的测定 顶空气相色谱法；
- SN/T 4675.19 出口葡萄酒中钠、镁、钾、钙、铬、锰、铁、铜、锌、砷、硒、银、镉、铅的测定；
- SN/T 4675.20 出口葡萄酒中稀土元素的测定 电感耦合等离子体质谱法；
- SN/T 4675.21 出口葡萄酒中可溶性无机盐的测定 离子色谱法；
- SN/T 4675.22 出口葡萄酒中总二氧化硫的测定 比色法；
- SN/T 4675.23 出口葡萄酒及葡萄汁中氨氮的测定 连续流动分析仪法；
- SN/T 4675.24 出口葡萄酒福林-肖卡指数的测定 分光光度计法；
- SN/T 4675.25 出口葡萄酒颜色的测定 CIE 1976(L*a*b*)色空间法；
- SN/T 4675.26 出口葡萄酒浊度的测定 散射光法；
- SN/T 4675.27 出口葡萄酒碱性灰分的测定；
- SN/T 4675.28 出口葡萄酒细菌、霉菌及酵母的计数；
- SN/T 4675.29 出口葡萄酒中酒香酵母检验 实时荧光 PCR 法；
- SN/T 4675.30 出口葡萄酒中拜氏接合酵母检验 实时荧光 PCR 法。

本部分为 SN/T 4675 的第 26 部分。

本部分按照 GB/T 1.1—2009 给出的规则起草。

本部分等同采用国际葡萄与葡萄酒组织(OIV)的方法 OIV-MA-AS2-08《Wine turbidity-Determination by Nephelometric Analysis》。本部分在技术内容上与方法一致,但考虑到我国标准本身的特点及汉语表达习惯,对 OIV 的方法 OIV-MA-AS2-08 的个别内容作了编辑性修改。

本部分由国家认证认可监督管理委员会提出并归口。

本部分起草单位：中华人民共和国北京出入境检验检疫局、中华人民共和国广东出入境检验检疫局。

本部分主要起草人：韩深、刘莹、王珮玥、冯鑫、郝欣、徐姗、刘青、李志勇。

出口葡萄酒浊度的测定 散射光法

1 范围

SN/T 4675 的本部分规定了葡萄酒浊度的散射光测定方法。

本部分适用于葡萄酒浊度的测定。

2 规范性引用文件

下列文件对于本文件的应用是必不可少的。凡是注日期的引用文件,仅注日期的版本适用于本文件。凡是不注日期的引用文件,其最新版本(包括所有的修改单)适用于本文件。

GB/T 6682 分析实验室用水规格和试验方法

3 术语和定义

下列术语和定义适用于本文件。

3.1

浊度 turbidity

液体中的悬浮物质对光线透过时产生的阻碍程度。

4 方法提要

本部分以福尔马肼(Formazine)作为浊度标准溶液,用散射光原理的浊度计测定葡萄酒样品的浊度,结果以 NTU(Nephelometric Turbidity Unit,散射式浊度单位)表示。

5 试剂和材料

本部分所用试剂和水,除另有规定,应使用分析纯试剂和符合 GB/T 6682 中的二级水。

福尔马肼标准溶液(Formazine):浊度值分别为 <0.1 NTU, 20 NTU, 100 NTU, 200 NTU 和 400 NTU, 配制方法参见附录 A。

6 仪器和设备

6.1 散射光浊度计(参见附录 B):

- 浊度计应配有在 620 nm 波长下测量的干扰过滤装置,如果光源是红外线则不需要;
- 入射光的谱带宽度应小于或等于 60 nm;
- 入射光为平行光不应该有发散,收敛度不能超过 1.5° ;
- 入射光光轴与发散光的光轴之间的测量角度应该为 $90^\circ \pm 2.5^\circ$;
- 装置由杂散光造成的误差在 0~0.1 NTU 范围内不能大于 0.01 NTU。

6.2 仪器配套样品池(含盖):直径为 25 mm,高度为 95 mm。

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6.3 恒温水浴锅:20℃±5℃。

6.4 具塞锥形瓶:250 mL。

7 测定步骤

7.1 仪器调试与校准

7.1.1 按浊度计使用说明书进行仪器的清洁与调试。

7.1.2 用福尔马肼标准溶液(第5章)校正浊度计,保存校准曲线。

7.2 样品前处理

7.2.1 将样品置于20℃±5℃的恒温水浴锅(6.3)10 min~20 min 摇匀待测。摇匀时应避免剧烈震动,否则可能会引起乳化现象。

7.2.2 起泡葡萄酒需预先脱气。将100 mL 试样倒入带排气塞的瓶中,在室温下使用水平振荡器或超声波水浴脱气,直至无气泡逸出。

7.3 测定

用少量恒温、脱气后的样品润洗样品池2次~3次后,将样品缓慢注入样品池中,操作时应避免气泡产生。将样品池放入仪器中,静置2 min~3 min,待示数稳定后读取浊度值。

8 结果计算和表述

样品的浊度值由浊度计示数直接读出,单位为NTU。

葡萄酒浊度值记录和表述如下:如果浊度小于1 NTU,则保留到0.01 NTU;如果浊度在1 NTU 和10 NTU 之间,则保留到0.1 NTU;如果浊度在10 NTU 和100 NTU 之间,则保留到1 NTU。

附 录 A

(资料性附录)

福尔马肼溶液的配制

A.1 零浊度水:选用孔径为 $0.1\ \mu\text{m}$ (或 $0.2\ \mu\text{m}$)的微孔滤膜过滤 GB/T 6682 中规定的二级水,反复过滤两次以上,所获的滤液即为零浊度水。将该水贮存于清洁的、并用该水冲洗后的玻璃瓶中。

A.2 硫酸肼溶液(10 g/L):称取 1.00g 硫酸肼($\text{N}_2\text{H}_6\text{SO}_4$),用零浊度水(A.1)溶解并定容至 100 mL。

A.3 六次甲基四胺溶液(100 g/L)的配制:称取 10.00 g 六次甲基四胺($(\text{CH}_2)_6\text{N}_4$),用零浊度水(A.1)溶解并定容至 100 mL。

A.4 福尔马肼储备液的配制:分别量取 5 mL 硫酸肼溶液(A.2)和 5 mL 六次甲基四胺溶液(A.3),用 $25\ ^\circ\text{C} \pm 3\ ^\circ\text{C}$ 恒温 24 h 的零浊度水(A.1)稀释至 100 mL,得到福尔马肼标准储备液,该标准溶液浊度值为 400 NTU,该标准溶液可以在室温下避光储存 4 周。

A.5 福尔马肼工作液的配制:分别吸取福尔马肼储备液(A.4)0 mL,5 mL,25 mL 和 50 mL 于 4 个 100 mL 的容量瓶中,用零浊度水(A.1)稀释至刻度,摇匀,该标准浊度液分别为 $<0.1\ \text{NTU}$, 20 NTU, 100 NTU, 200 NTU。

注:硫酸肼有毒,可能致癌,实验时请在通风厨内操作。

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附 录 B
(资料性附录)

散射光浊度计测定浊度的光学原理

仪器测定原理见图 B.1。

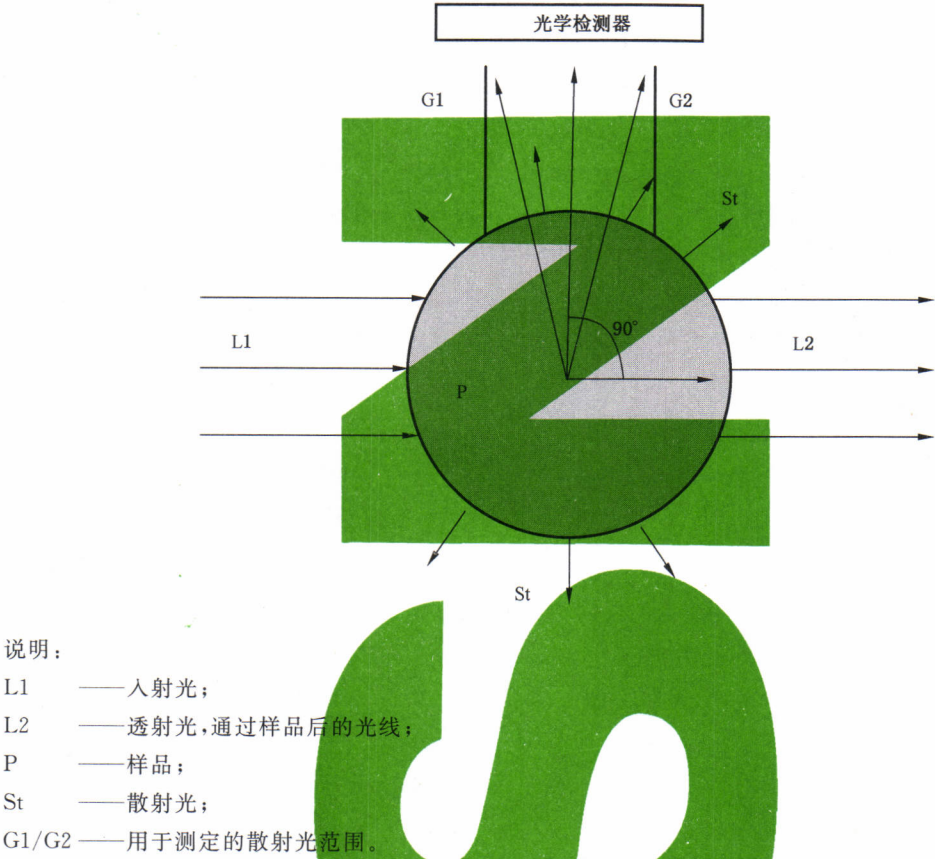


图 B.1 光学原理图

图 B.1 为入射光透过样品后光的透射与散射示意图。散射光式浊度计测定与入射光传播方向 90° 夹角方向的散射光强。在光程一定的情况下,该散射光强与浊度值成线性关系。通过测定散射光强可得到样品的浊度值。

Foreword

Standard (SN/T 4675) "Methods of export wine analysis" includes 30 parts:

- SN/T 4675.1: Determination of glycerol in wine for export—Enzymatic method;
- SN/T 4675.2: Determination of 2,3-butanediol in wine for export—GC method;
- SN/T 4675.3: Determination of stable carbon isotope ratio of ethanol in wine for export;
- SN/T 4675.4: Determination of lactic acid in wine for export—Enzymatic method;
- SN/T 4675.5: Determination of organic acid in wine for export—Ion chromatography method;
- SN/T 4675.6: Determination of glucose, fructose and sucrose in wine for export;
- SN/T 4675.7: Determination of acetaldehyde in wine for export—GC/MS method;
- SN/T 4675.8: Determination of 5-hydroxymethylfurfural in wine for export—HPLC method;
- SN/T 4675.9: Determination of diethylene in wine for export—GC/MS method;
- SN/T 4675.10: Determination of ochratoxin A in wine for export—HPLC/MS/MS method;
- SN/T 4675.11: Determination of 7 anthocyanins in wine for export—UHPLC method;
- SN/T 4675.12: Determination of lysozyme in wine for export—HPLC method;
- SN/T 4675.13: Determination of 2,4,6-trichloroanole in wine for export—GC/MS method;
- SN/T 4675.14: Determination of natamycin in wine for export—HPLC/MS/MS method;
- SN/T 4675.15: Determination of salicylic acid, dehydroacetic acid and 4-chlorobenzoic acid in wine for export—HPLC method;
- SN/T 4675.16: Determination of fumaric acid in wine for export—HPLC/MS/MS method;
- SN/T 4675.17: Determination of butyltin compounds in wine for export—GC/MS/MS method;
- SN/T 4675.18: Determination of dithiocarbamates(salt) residues in wine for export—Headspace

GC method;

—SN/T 4675.19: Determination of sodium, magnesium, potassium, calcium, chromium, manganese, iron, copper, zinc, arsenic, selenium, silver, cadmium and lead in wine for export;

—SN/T 4675.20: Determination of rare-earth elements in wine for export—ICP-MS method;

—SN/T 4675.21: Determination of soluble inorganic salts in wine for export—Ion chromatography method;

—SN/T 4675.22: Determination of total sulfur dioxide in wine for export—Colorimetric method;

—SN/T 4675.23: Determination of ammonium nitrogen in wine and grape juice for export—Continuous flow analysis(CFA) method;

—SN/T 4675.24: Determination of Folin & Ciocalteu index of wine for export—Spectrophotometry method;

—SN/T 4675.25: Determination of chromatic characteristics of wine for export—CIE Lab color space system;

—SN/T 4675.26: Determination of turbidity of wine for export—Diffused radiation method;

—SN/T 4675.27: Determination of alkaline ash of wine for export;

—SN/T 4675.28: Method for enumeration of colony-forming units of yeasts, moulds and bacteria in cork stoppers and wine for export;

—SN/T 4675.29: Determination of *brettanomyces* in wine for export—Real-time PCR method;

—SN/T 4675.30: Determination of *zygosaccharomyces bailii* in wine for export—Real-time PCR method.

This part is part 26 of the standard.

This part is drafted according to GB/T 1.1—2009.

This part is identical to the method of Organization of international Vine and wine(OIV) MA-AS2-28, “Wine turbidity-Determination by Nephelometric Analysis”. The technical content was the same except for some editorial changes considering Chinese expression custom.

This part was proposed by and was under the jurisdiction of Certification and Accreditation Administration of the People’s Republic of China.

This part was drafted by Beijing Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China and Guangdong Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China.

The main drafters of this part were Han Shen, Liu Ying, Wang Peiyue, Feng Xin, Hao Xin, Xu Shan, Liu Qing and Li Zhiyong.

Determination of turbidity of wine for export—Diffused radiation method

1 Scope

This standard specifies the methods of determination of wine turbidity.

This standard is applicable to the determination of turbidity in wines.

2 Cited normative references

The following referenced documents are indispensable for the application of this document. For dated references, only edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 6682 Water for analytical laboratory use—Specification and test methods

3 Terms and definitions

For the purpose of this document, the following terms and definitions apply.

3.1 Turbidity: reduction of the transparency of a liquid due to the presence of undissolved substances.

4 Principle

Formazine is used as the standard solution of turbidity. Wine turbidity is determined by the apparatus with the principle of diffused radiation. The unit of the result is expressed by NTU (Nephelometric Turbidity Unit). The unit of the result obtained by the diffused radiation turbidimeter.

5 Reagents and materials

Unless otherwise specified, all reagents are analytical grade and water is the second grade water prescribed by GB/T 6682.

Formazine standard solutions; the values respectively are <0.1 NTU, 20 NTU, 100 NTU, 200 NTU and 400 NTU. They can be prepared according to the method in Annex A.

6 Apparatus and equipment

6.1 Diffused radiation turbidimeter(see Annex B):

- a) The turbidity meter 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;
- b) The width of the spectral band of the incident radiation should be less than or equal to 60 nm;
- c) There should be no divergence in the parallelism of the incident radiation, and convergence must not exceed 1.5° ;
- d) The angle of measurement between the optical axis of the incident radiation and that of the diffused radiation should be $90^\circ \pm 2.5^\circ$;
- e) The apparatus must not cause error due to stray light greater than: 0.01 NTU of random light error within a range of: 0 to 0.1 NTU.

6.2 Measuring tank with cover: diameter: 25 mm, height: 95 mm.

6.3 Thermostat water bath: $20^\circ\text{C} \pm 5^\circ\text{C}$.

6.4 Conical flask with cover: 250 mL.

7 Measurement procedures

7.1 Turbidimeter checking and calibration

7.1.1 Clean and check of the apparatus in accordance with the recommendations of the manufacturer.

7.1.2 Calibrate the turbidity meter with formazine standard solutions (5) to gain a calibration curve.

7.2 Sample preparation

7.2.1 Put the test sample in the thermostat water bath with $20^\circ\text{C} \pm 5^\circ\text{C}$ in 10 min~20 min, careful-

ly homogenize the sample without making any abrupt movement that could create an emulsion and be ready for analysis.

7.2.2 Sparkling wine need to be degassed Pour the wine sample of about 100 mL into a beaker flask with air evacuation valve. Placed the wine in an ultrasonic water tank(or horizontal oscillator) at room temperature after a certain period of time until no gas escapes.

7.3 Taking measurement

Carefully wash the measuring tank twice or three times with a small amount of the prepared sample to be analyzed. Carefully pour the sample 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. Put the tank in the turbidimeter, wait 2 min~3 min and read the value after it is stable.

8 Calculation and expression of result

The index values are read directly from the turbidimeter.

The turbidity index of the wine 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.

Annex A
(informative)

The preparation of formazine standard solutions

A.1 Zero turbidity water: the second grade water prescribed by GB/T 6682 is filtered more than twice through 0.1 μm or 0.2 μm microporous filter membrane. Storage the water in a clean bottle washed by the zero turbidity water and retain this water for preparation of standard solutions.

A.2 Hydrazinium sulfate (10 g/L): dissolve 1.0 g hydrazinium sulfate ($\text{N}_2\text{H}_6\text{SO}_4$) in water (A.1), then fill to a volume of 100 mL using the water (A.1).

A.3 Hexamethylene – tetramine (100 g/L): dissolve 10.0 g Hexamethylene – tetramine ($(\text{CH}_2)_6\text{N}_4$) in the water (A.1), then fill to a volume of 100 mL using the water (A.1).

A.4 Formazine standard stock solution: mix 5 mL of Hydrazinium sulfate solution (A.2) and 5 mL of Hexamethylene – tetramine solution (A.3). Dilute the mixed solution to a volume of 100 mL with water (A.1) after 24 hours at $25\text{ }^\circ\text{C} \pm 3\text{ }^\circ\text{C}$. The turbidity of this standard stock solution is 400 NTU. This standard suspension will keep for approximately 4 weeks at room temperature in the dark.

A.5 Formazine standard working solution: separately draw 0 mL, 5 mL, 25 mL and 50 mL formazine standard stock solution (A.4) and dilute the solution to a volume of 100 mL with water (A.1). The turbidity of these standard working solutions is <0.1 NTU, 20 NTU, 100 NTU and 200 NTU.

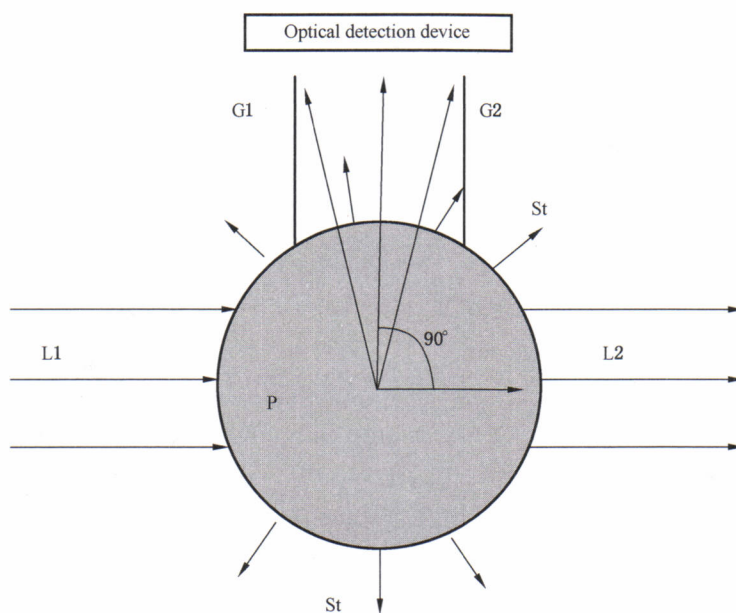
NOTE: Hydrazinium sulfate is poisonous and may be carcinogenic. The operation should be under the protection.

Annex B

(informative)

Optical principle of diffused radiation turbidimeter

Apparatus measurement principle is showed in Figure B.1.



- L1 — Incident light beam;
 L2 — Beam after passing through sample;
 P — Sample;
 St — diffused light;
 G1/G2 — Limiting rays from the diffused light beam used for measurement.

Figure B.1—Optical principle chart

Figure B.1 is the diagram of transmission light and diffused light after incident light beam passing through samples. Diffused radiation turbidimeter takes measurement of the diffused light intensity at an angle of 90° to the direction of propagation of the incident beam. In the circumstance of certain optical path, the diffused light intensity has linear relationship with turbidity value. The turbidity value of the sample can be obtained by measuring the diffused light intensity.



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