

SN

中华人民共和国出入境检验检疫行业标准

SN/T 4675.27—2016

出口葡萄酒碱性灰分的测定

Determination of alkaline ash in wine for export

2016-12-12 发布

2017-07-01 实施



中 华 人 民 共 和 国
国家质量监督检验检疫总局 发 布

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 2,4,6-三氯苯甲醚残留量的测定 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 27 部分。

本部分按照 GB/T 1.1—2009 给出的规则起草。

本部分等同国际葡萄与葡萄酒组织(OIV)的方法 OIV-MA-AS2-04《葡萄酒灰分》(Ash of Wines)和部分修改采用 OIV-MA-AS2-05《葡萄酒碱性灰分 滴定法》(Alkalinity of Ash of Wines Titrimetric Method)。本部分在技术内容上与两方法基本一致,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 MA-AS2-05 的个别内容作了技术性修改,修改的主要内容为:

- 增加了瓷坩埚;

——增加了盐酸标准滴定溶液(0.100 0 mol/L);

——增加了公式 $x_3 = \frac{c \times (10 - V_1) \times 0.069 \times 1\,000}{20}$ 。

请注意本文件的某些内容可能涉及专利,本文件的发布机构不承担识别这些专利的责任。

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:中华人民共和国中山出入境检验检疫局、中华人民共和国黄埔出入境检验检疫局、中华人民共和国广东出入境检验检疫局。

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出口葡萄酒碱性灰分的测定

1 范围

SN/T 4675 的本部分规定了葡萄酒中碱性灰分的测定方法。

本部分适用于葡萄酒中碱性灰分的测定。

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 601 化学试剂 标准滴定溶液的制备

GB/T 6682 分析实验室用水规格和试验方法

3 方法提要

灰分溶解于标准酸滴定溶液中,过量的酸以碱标准滴定溶液滴定,根据碱的消耗量乘以换算系数即为灰分的碱度。

4 试剂和材料

除另有规定外,所用试剂均为分析纯,试验用水为 GB/T 6682 规定的一级水。

4.1 甲基橙($\text{C}_{14}\text{H}_{14}\text{N}_3\text{SO}_3\text{Na}$)。

4.2 盐酸(HCl)。

4.3 硫酸(H_2SO_4)。

4.4 氢氧化钠(NaOH)。

4.5 盐酸标准滴定溶液($0.100\ 0\ \text{mol/L}$)或硫酸标准滴定溶液($0.050\ 0\ \text{mol/L}$):按 GB/T 601 进行配制及标定。

4.6 氢氧化钠标准滴定溶液($0.100\ 0\ \text{mol/L}$):按 GB/T 601 进行配制及标定。

4.7 甲基橙指示剂(0.1%):称取 $0.1\ \text{g}$ 甲基橙(4.1),用少量水溶解,全部转移至 $100\ \text{mL}$ 容量瓶中,用水定容至刻度。

5 仪器和设备

5.1 恒温水浴锅。

5.2 分析天平:感量为 $0.1\ \text{mg}$ 。

5.3 加热板。

5.4 干燥器(内有干燥剂)。

5.5 铂坩埚或瓷坩埚。

5.6 马弗炉。

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5.7 碱式滴定管:10 mL。

5.8 玻璃棒。

5.9 水平振荡器。

5.10 超声波水浴。

6 分析步骤

取铂坩埚或瓷坩埚(5.5)置马弗炉(5.6)中,在 $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ 下灼烧 30 min,冷却至 $200\text{ }^{\circ}\text{C}$ 左右,取出,放入干燥器(5.4)中冷却 30 min,准确称量。恒重灼烧至前后两次称量相差不超过 0.5 mg。

吸取 20 mL 葡萄酒(起泡酒需预先脱气,将 100 mL 试样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。)置于预先恒重的铂坩埚或瓷坩埚中,在恒温水浴锅(5.1)上蒸发至干;之后将盛有蒸发残留物的铂坩埚或瓷坩埚置于 $200\text{ }^{\circ}\text{C}$ 加热板上碳化,直到不再产生烟为止;再将铂坩埚或瓷坩埚置于 $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ 的马弗炉中,灼烧 15 min;冷却至 $200\text{ }^{\circ}\text{C}$ 左右,从马弗炉中取出铂坩埚或瓷坩埚,向铂坩埚或瓷坩埚中加入 5 mL 蒸馏水,置于恒温水浴锅上,重新加热、碳化,然后再移入 $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$ 的马弗炉中灼烧 10 min。冷却至 $200\text{ }^{\circ}\text{C}$ 左右,取出铂坩埚或瓷坩埚,待其在干燥器中冷却后称重。恒重灼烧至前后两次称量相差不超过 0.5 mg。

在盛有 20 mL 葡萄酒的灰分铂坩埚或瓷坩埚中,加入 10 mL 盐酸标准滴定溶液或硫酸标准滴定溶液(4.5),将铂坩埚或瓷坩埚置于沸水浴上约 15 min,用玻璃棒搅拌至灰分完全溶解。加入两滴甲基橙指示剂(4.7),用氢氧化钠标准滴定溶液(4.6)滴定过量的盐酸或硫酸标准滴定溶液,直至指示剂颜色变为黄色。

注:如果碳化不完全,可将碳化残渣重新加入 5 mL 蒸馏水,蒸发掉水分之后,再次灼烧。对于含糖分较高的葡萄酒,最好在第一次灰化前,在蒸发残留物上滴加若干滴纯植物油,以防止产生过多的泡沫。

7 结果计算

7.1 试样中碱性灰分用毫摩尔每升表示,按式(1)或式(2)进行计算:

$$x_1 = \frac{c \times (10 - V_1) \times 1\,000}{20} \quad \dots\dots\dots (1)$$

$$x_2 = \frac{2 \times c \times (10 - V_1) \times 1\,000}{20} \quad \dots\dots\dots (2)$$

式中:

x_1 ——试样中碱性灰分含量(测定时加盐酸标准滴定溶液),单位为毫摩尔每升(mmol/L);

x_2 ——试样中碱性灰分含量(测定时加盐酸标准滴定溶液),单位为毫摩尔每升(mmol/L);

c ——盐酸或硫酸标准滴定溶液的浓度,单位为摩尔每升(mol/L);

V_1 ——试样消耗氢氧化钠标准滴定溶液体积,单位为毫升(mL)。

结果保留两位有效数字。

7.2 试样中碱性灰分另一种表达方式以碳酸钾计,用克每升表示,按式(3)或式(4)进行计算:

$$x_3 = \frac{c \times (10 - V_1) \times 0.069 \times 1\,000}{20} \quad \dots\dots\dots (3)$$

$$x_4 = \frac{c \times (10 - V_1) \times 0.138 \times 1\,000}{20} \quad \dots\dots\dots (4)$$

式中:

x_3 ——试样中碱性灰分含量(以碳酸钾计)(测定时加盐酸标准滴定溶液),单位为克每升(g/L);

x_4 ——试样中碱性灰分含量(以碳酸钾计)(测定时加硫酸标准滴定溶液),单位为克每升(g/L);

c ——盐酸或硫酸标准滴定溶液的浓度,单位为摩尔每升(mol/L);

V_1 ——试样消耗氢氧化钠标准滴定溶液体积,单位为毫升(mL);

0.069——与 1.00 mL 盐酸标准滴定溶液 [$c(\text{HCl}) = 1.000 \text{ mol/L}$] 相当的碳酸钾的质量,单位为克(g);

0.138——与 1.00 mL 硫酸标准滴定溶液 [$c(\text{H}_2\text{SO}_4) = 1.000 \text{ mol/L}$] 相当的碳酸钾的质量,单位为克(g)。

结果保留两位有效数字。

8 重复性

以两次平行测定结果的算术平均值作为测定结果,两次平行测定结果的绝对差值不得超过其算术平均值的 10%。

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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-trichloroaniline 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 butyric 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 27 of the standard.

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

This part completely adopted the method of Organization of International Vine and wine (OIV) MA-AS2-04, “Ash of Wines” and modified MA-AS2-05 “Alkalinity of Ash of Wines Titrimetric Method”. The technical content was the same except for some technical changes as follows:

—Porcelain crucible was increased;

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—Hydrochloric acid standard titration solution(0.100 0 mol/L) was increased;

—Formula of $x_3 = \frac{c \times (10 - V_1) \times 0.069 \times 1\,000}{20}$ was increased.

Please note that some of the content of the standard may involve patents.Publication of the present standard does not bear the responsibility of identifying these patents.

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 Zhongshan Entry-Exit Inspection and Quarantine Bureau of the People's Republic of China, Huangpu 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.

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Determination of alkaline ash in wine for export

1 Scope

This part specifies the method for the determination of alkalinity ash in wine.

This part is applicable to the determination of alkalinity ash in wine.

2 Quoted normative documents

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 601 Chemical reagent—Preparations of standard volumetric solutions.

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

3 Principle

The ash is dissolved in acid standard titration solution, the excess acid solution is determined by Alkali standard titration solution, the consumption of alkali content multiplied by the conversion coefficient factor is the alkalinity of ash.

4 Reagents and materials

Unless others specified, the reagents should be analytical pure. And the water should accord with GB/T 6682.

4.1 Methyl orange ($\text{C}_{14}\text{H}_{14}\text{N}_3\text{SO}_3\text{Na}$).

4.2 Hydrochloric acid (HCl).

4.3 Sulfuric acid (H_2SO_4).

4.4 Sodium hydroxide (NaOH).

4.5 Hydrochloric acid standard titration solution (0.100 0 mol/L) or sulfuric acid standard solution (0.050 0 mol/L): Preparation and calibration of GB/T 601.

4.6 Sodium hydroxide standard titration solution (0.100 0 mol/L): Preparation and calibration of GB/T 601.

4.7 Methyl orange indicator (0.1%): Take 0.1 g methyl orange (4.1), dissolved with a small amount of water, transferred to a volumetric flask (100 mL), dilute to 100 mL.

5 Apparatus and equipment

5.1 Thermostatic bath.

5.2 Analysis balance: sensitive to 0.1 mg.

5.3 Heating plate.

5.4 Dryer (desiccant).

5.5 Platinum crucible or porcelain crucible.

5.6 Muffle furnace.

5.7 Alkali Burette: 10 mL.

5.8 Glass rod.

5.9 Horizontal oscillator tube.

5.10 Ultrasonic bath.

6 Analytical procedure

Put platinum crucible or porcelain crucible (5.5) in a muffle furnace (5.6), burning for 30 min at $525\text{ }^{\circ}\text{C} \pm 25\text{ }^{\circ}\text{C}$, cooling to $200\text{ }^{\circ}\text{C}$, remove and place in a dryer (5.4) cooling for 30 min, accurately weighing. Burning to constant weight, the weight difference of before and after burning times is under 0.5 mg.

Pipette 20 mL wine (sparkling wine need to pre-degas, 100 mL sample will be degassed by horizontal oscillator or ultrasonic wave at room temperature until no bubbles) into the previously platinum crucible or porcelain crucible (constant weight), evaporate sample wine to dry on the Thermostatic bath

(5.1), then carbonizing the evaporating residues on the heating plate at 200 °C, until no smog appeared. Put the crucible into the furnace and burn for 15 min at 525 °C ± 25 °C, cooling to 200 °C, and take out the crucible and add 5 mL water, and put into Thermostatic bath(5.1), then re-heating, re-carbonizing, then put into muffle furnace, burning for 10 mins, cooling to 200 °C, take out platinum crucible or porcelain crucible, then weigh it after being cooled in dryer. Burning to constant weight, the weight difference of before and after burning times is under 0.5 mg.

Add 10 mL hydrochloric acid standard titration solution or sulfuric acid standard solution(4.5) to the ash from 20 mL of wine in the platinum crucible or porcelain crucible. Place the crucible on the boiling water-bath for about 15 min, then stirring the solution by glass rod until the ash was totally dissolved. Add two drops of methyl orange solution (4.7) and titrate the excess hydrochloric acid or sulfuric acid against sodium hydroxide standard titration solution(4.6) until the color of the indicator changes to yellow.

Note: if the carbonization was not completely, can re-add 5 mL distilled water, then evaporated waters, burning again. For some sample wine which include high sugar content, when firstly carbonizing, it will be better to drop some plant oil in sample wine for preventing foam.

7 Expression of results

7.1 In the sample, the alkaline ash content, expressed in milliequivalents per liter was calculated by formula (1) or formula (2):

$$x_1 = \frac{c \times (10 - V_1) \times 1\,000}{20} \dots\dots\dots (1)$$

$$x_2 = \frac{2 \times c \times (10 - V_1) \times 1\,000}{20} \dots\dots\dots (2)$$

Where:

x_1 —alkaline ash content in the sample, (determination of the hydrochloric acid standard titration solution) mmol/L;

x_2 —the alkaline ash content in the sample, (determination of the sulfuric standard titration solution) mmol/L;

c —the concentration of the standard titration solution of hydrochloric acid or sulfuric acid, mol/L;

V_1 —the volume of the sample consumption of sodium hydroxide standard titration solution, mL;

The result is kept to two decimal places.

7.2 In the test sample, the alkaline ash content, another expressed in grams per liter of potassium carbonate was calculated by formula (3) or formula (4):

$$x_3 = \frac{c \times (10 - V_1) \times 0.069 \times 1\,000}{20} \dots\dots\dots (3)$$

$$x_4 = \frac{c \times (10 - V_1) \times 0.138 \times 1\,000}{20} \dots\dots\dots (4)$$

Where:

x_3 —alkaline ash content in the sample (in order to potassium carbonate) (determination of the hydrochloric acid standard titration solution), g/L;

x_4 —the alkaline ash content in the sample (in order to potassium carbonate) (determination of the sulfuric standard titration solution), g/L;

c —the concentration of the standard titration solution of hydrochloric acid or sulfuric acid, mol/L;

V_1 —the volume of the sample consumption of sodium hydroxide standard titration solution, mL;

0.069—the weight of potassium carbonate which was equal to the weight of potassium carbonate which was titrated by 1 mL hydrochloric acid standard titration solution [$c(\text{HCl}) = 1.000 \text{ mol/L}$], g;

0.138—the weight of potassium carbonate which was equal to the weight of potassium carbonate which was titrated by 1 mL sulfuric acid standard titration solution [$c(\text{H}_2\text{SO}_4) = 1.000 \text{ mol/L}$], g.

The result is kept to two decimal places.

8 Repeatability

In the two parallel test results, the absolute value of the two parallel test results must not exceed 10% of the average value of the results.

中华人民共和国出入境检验检疫
行 业 标 准
出口葡萄酒碱性灰分的测定
SN/T 4675.27—2016

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中国标准出版社出版
北京市朝阳区和平里西街甲2号(100029)
北京市西城区三里河北街16号(100045)
总编室:(010)68533533
网址 www.spc.net.cn

中国标准出版社秦皇岛印刷厂印刷

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开本 880×1230 1/16 印张 1 字数 24 千字
2017年11月第一版 2017年11月第一次印刷
印数 1—500

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书号: 155066·2-32296 定价 18.00 元



SN/T 4675.27-2016