

**INDUSTRY STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

SN/T 4675.15-2016

**Detection of Salicylic acid, Dehydroacetic acid and
4-Chlorobenzoic acid in wine for export--HPLC method**

出口葡萄酒中水杨酸、脱氢乙酸、对氯苯甲酸的测定 液相色谱法

(English Translation)

Issued on: December 12, 2016

Implemented on: July 1, 2017

Issued by: General Administration of Customs

Table of Contents

Foreword

1 Scope

2 Normative references

3 Method summary

4 Reagents and materials

Foreword

SN/T 4675 "Method for Analysis of the Quality and Safety of Exported Wines" is divided into 30 sections.

---SN/T 4675.1 Determination of glycerol in wine exports Enzymatic;

---SN/T 4675.2 Determination of 2,3-butanediol in exported wines by gas chromatography;

---SN/T 4675.3 Determination of Stable Carbon Isotope Ratio of Ethanol in Export Wines;

---SN/T 4675.4 Determination of Lactic Acid in Export Wines Enzymatic;

---SN/T 4675.5 Determination of organic acids in exported wines by ion chromatography;

---SN/T 4675.6 Determination of glucose, fructose and sucrose in exported wines;

---SN/T 4675.7 Determination of acetaldehyde in exported wines by gas chromatography-mass spectrometry;

---SN/T 4675.8 Determination of 5-hydroxymethylfurfural in wines by liquid chromatography;

---SN/T 4675.9 Determination of diethylene glycol in exported wines by gas chromatography-mass spectrometry;

---SN/T 4675.10 Determination of ochratoxin A in wines for liquid chromatography-mass spectrometry/mass spectrometry;

---SN/T 4675.11 Determination of seven anthocyanins in wines exported by ultra-high performance liquid chromatography;

---SN/T 4675.12 Determination of Lysozyme in Export Wines by Liquid Chromatography;

---SN/T 4675.13 Determination of residues of 2,4,6-trichloroanisole in exported wines by gas chromatography-mass spectrometry;

---SN/T 4675.14 Determination of natamycin in wines for export - Liquid chromatography-mass spectrometry/mass spectrometry;

- SN/T 4675.15 Determination of salicylic acid, dehydroacetic acid and p-chlorobenzoic acid in wines by liquid chromatography;
- SN/T 4675.16 Determination of fumaric acid in wine exports by liquid chromatography-mass spectrometry/mass spectrometry;
- SN/T 4675.17 Determination of butyltin content in exported wines by gas chromatography-mass spectrometry/mass spectrometry;
- SN/T 4675.18 Determination of dithiocarbamate residues in exported wines. Headspace gas chromatography;
- SN/T 4675.19 Determination of sodium, magnesium, potassium, calcium, chromium, manganese, iron, copper, zinc, arsenic, selenium, silver, cadmium, and lead in wine exports;
- SN/T 4675.20 Determination of rare earth elements in wines exported by inductively coupled plasma mass spectrometry;
- SN/T 4675.21 Determination of soluble inorganic salts in exported wines by ion chromatography;
- SN/T 4675.22 Determination of total sulfur dioxide in wines by colorimetric method;
- SN/T 4675.23 Determination of ammonia nitrogen in wine and grape juice exported by continuous flow analyzer method;
- SN/T 4675.24 Determination of the export of the Folin-Shaughha index of the wine by spectrophotometry;
- SN/T 4675.25 Determination of color of exported wines CIE1976 ($L^*a^*b^*$) color space method;
- SN/T 4675.26 Determination of export turbidity scattered light method;
- SN/T 4675.27 Determination of alkaline ash content of exported wines;
- SN/T 4675.28 Export of wine bacteria, mold and yeast count;
- SN/T 4675.29 Real-time fluorescent PCR assay for wine yeast extraction in wine exports;
- SN/T 4675.30 Bayer splice yeast assay for real-time fluorescence PCR in exported wines.

This section is Part 15 of SN/T 4675.

This section was drafted in accordance with the rules given in GB/T 1.1-2009.

In the development of this section, reference was made to the OIV Method OIV-MA-AS4-O2B, Preservatives and Hair.

Detection of fermentation inhibitors (sorbic acid, benzoic acid, p-chlorobenzoic acid, salicylic acid, p-hydroxybenzoic acid and its esters), OIV-MA-AS313-20 "Liquid Chromatography for the Detection of Sorbic Acid, Benzoic Acid, Salicylic Acid in Wine", Inspection of OIV-MA-AS4-O2E "Preservatives and Fermentation Inhibitors" Measurement (dehydroacetic acid).

1 Scope

This part of SN/T 4675 specifies liquid chromatographic methods for the determination of salicylic acid, dehydroacetic acid, and p-chlorobenzoic acid in wine.

This section applies to the determination of salicylic acid, dehydroacetic acid, and p-chlorobenzoic acid in wine exports.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only dated versions apply to this article. For undated references, the latest version (including all amendments) applies to this document.

GB/T 6682 Analysis Laboratory Water Specifications and Test Methods

3 Method summary

The wine sample was directly diluted with water, measured with a liquid chromatograph equipped with a UV detector, and quantified by an external standard method.

4 Reagents and materials

Unless otherwise stated, the reagents used in this method are of analytical grade, and the water is a grade one water according to GB/T 6682.

4.1

Methanol (CH_3OH). Chromatographically pure.

4.2

Sodium hydroxide (NaOH).

4.3

Sodium hydroxide solution. 20g/L. Weigh 4g (accurate to 0.001g) of sodium hydroxide, dissolve with water and bring to 200 mL.

4.4

Acetic acid ($\text{C}_2\text{H}_4\text{O}_2$). Chromatographically pure.

4.5

Ammonium acetate solution. 0.02 mol/L. Weigh 1.54g (accurate to 0.001g) of ammonium acetate, dissolve with water and bring to 1L.

4.6

Reference material. salicylic acid (C₇H₆O₃), CAS number 69-72-7, purity ≥98%; dehydroacetic acid (C₈H₈O₄), CAS number 520-45-6, purity ≥98%; p-chlorobenzoic acid (C₇H₅ClO), CAS number 104-88-1, purity ≥98%.

4.7

Standard stock solution of salicylic acid. Accurately weigh the appropriate amount of salicylic acid standard product, dissolve it with methanol and prepare a standard stock solution of 1.0 mg/mL.

Stored at 4°C for 3 months.

4.8

Dehydroacetic acid standard stock solution. Accurately weigh an appropriate amount of dehydroacetic acid standard and dissolve it with 10.0 mL sodium hydroxide solution (4.3).

Dilute and make a standard stock solution of 1.0 mg/mL, store at 4 °C, and store for 3 months.

4.9

Standard stock solution of p-chlorobenzoic acid. Accurately weigh the appropriate amount of p-chlorobenzoic acid standard, dissolve it with methanol and prepare a standard of 1.0 mg/mL.

Stock solution, stored at 4°C, can be stored for 3 months.

4.10

mixed standard stock solution. take 10.0mL each of salicylic acid, dehydroacetic acid, and p-chlorobenzoic acid standard stock solution in a 100mL volumetric flask.

Diluted to the mark with water, this solution was a 100 mg/L mixed standard stock solution and stored at 4°C.

4.11

mixed standard working solution. The mixed standard stock solution was diluted with water to 20mg/L, 10mg/L, 5.0mg/L, 2.0mg/L, A 1.0 mg/L, 0.50 mg/L mixed standard working solution was stored at 4°C.