ICS 67.160.10
CCS X 61

National Standards of the People's Republic of China

GB/T 10345—2022
Replace GB/T 10345-2007

Liquor analysis method

Method of analysis for baijiu

Published on 2022-07-11

2023-02-01 Implementation

State Administration for Market Regulation
National Standardization Administration

release

Preface

table of contents

Preface

This document was drafted in accordance with the provisions of GB/T1.1-2020 "Guidelines for standardization work Part 1: Structure and drafting rules for standardization documents".

This document replaces GB/T10345-2007 "Analysis Method for Liquor". Compared with GB/T10345-2007, the main technical changes are as follows:

a) The method for determining alcohol content has been deleted (see Chapter 6 of the 2007 edition);

b) The determination method of total acid has been deleted (see Chapter 7 of the 2007 edition);

c) Added the determination method of total acid esters (see Chapter 8);

d) Added the determination methods for acetic acid and caproic acid (see Chapter 11 and Chapter 12);

e) The determination methods for ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate, n-propanol and 3-phenylethanol have been changed (see Chapter 10, Chapter 10, Chapter 13, Chapter 11, Chapter 12, Chapter 15 and Chapter 16 of the 2007 edition);

f) The determination method of ethyl propionate and diethyl ester of dibasic acids (pimelic acid, suberic acid, azelaic acid) has been changed (see Chapter 13 and Chapter 14, Chapter 14 and Chapter 18 of the 2007 edition);

g) The determination method for 3-methylmercaptopropanol (see Chapter 17 of the 2007 edition) has been deleted.

Please note that some of the contents of this document may involve patents. The issuing organization of this document does not assume the responsibility for identifying patents.

This document was proposed by China Light Industry Federation.

This document is under the jurisdiction of the National Technical Committee for Standardization of Liquor (SAC/TC358).

This document was drafted by: China Food Fermentation Industry Research Institute Co., Ltd., Luzhou Laojiao Co., Ltd., Anhui Gujing Gongjiu Co., Ltd., Yibin Wuliangye Co., Ltd., Kweichow Moutai Co., Ltd., Guangdong Food Industry Research Institute Co., Ltd., Sichuan Jiannanchun (Group) Co., Ltd., Shanxi Xinghuacun Fenjiu Factory Co., Ltd., Hubei Baiyunbian Liquor Co., Ltd., Anhui Kouzi Liquor Co., Ltd., Jinan Baotuquan Brewing Co., Ltd., Sichuan Gulin Langjiu Factory Co., Ltd., Hebei Hengshui Laobaigan Liquor Co., Ltd., Jinpai Co., Ltd., China Light Food Inspection and Certification Co., Ltd., Site Liquor Co., Ltd., Kweichow Moutai Distillery (Group) Xijiu Co., Ltd., Shanghai Liquor Product Quality Inspection Center Co., Ltd., Hubei Daohuaxiang Winery Co., Ltd., Guangdong Jiujiang Winery Co., Ltd., Beijing Shunxin Agriculture Co., Ltd. Niulanshan Winery, Sichuan Mianyang Fenggu Winery Co., Ltd., Gubeichun Group Co., Ltd., Jiangsu Yanghe Brewery Co., Ltd., Puyan (Shanghai) Standard Technical Service Co., Ltd., Shandong Jingzhi Winery Co., Ltd., Guangdong Radio and Television Metrology Testing (Chengdu) Co., Ltd., Guangxi Zhuang Autonomous Region Analysis and Testing Research Center, Anhui Guotai Zhongxin Testing Technology Co., Ltd., Guizhou Dongjiu Co., Ltd., Food and Drug Environmental Inspection Institute (Shandong) Group Co., Ltd., Shandong Bandaojing Co., Ltd., Sichuan Synas Analysis and Testing Co., Ltd., Infinitus (China) Co., Ltd.

The main drafters of this document are: Gao Hongbo, Meng Zhen, Zhang Suyi, Wang Li, Xu Zhancheng, Feng Zhiqiang, Guo Wenjie, Chen Hongkun, Guo Xinguang, Wang Fengxian, Yang Tuanyuan, Xu Qinxiang, Lv Zhiyuan, Shen Yi, Zhang Yuxing, Lei Mingming, Wu Shengwen, Zhong Fangda, Wang Minfeng, Chen Ping, He Songgui, Wei Jinwang, Lu Zhongming, Zhong Qiding, Du Xinyong, Zhong Yu, Wu Haiping, Yang Renjun, Jiang Jian, Huang Yifan, Chen Hongzhou, Wang Yi, Sun Xuewen, Zhang Fengguo, Sun Hongmei, Luo Jin, Zhou Fangmei, Gao Xiaojuan, Yin Changwei, Chen Yanhe, Chang Di, Liu Weide, Ji Yu.

The previous versions of this document and the documents it replaces are as follows:

- First published in 1989 as GB/T10345.1-1989~GB/T 10345.8-1989;
- First revised in 2007 as GB/T 10345-2007;
- This is the second revision.

Liquor analysis method

This document specifies the general principles, basic requirements and detailed test procedures for liquor analysis.

This document is applicable to the analysis of various liquors.

2 Normative references

The contents of the following documents constitute the essential clauses of this document through normative references in this text. Among them, for referenced documents with dates, only the versions corresponding to the dates are applicable to this document; for referenced documents without dates, the latest versions (including all amendments) are applicable to this document.

GB/T 601 Preparation of standard titration solutions for chemical reagents

GB/T 603 Chemical reagents - Preparation of preparations and products used in test methods

GB/T 6682-2008 Specifications and test methods for water used in analytical laboratories

3 Terms and definitions

There are no terms or definitions that require definition in this document.

4 General principles

4.1 The terms, terms and measurement units used in this document shall comply with the provisions of relevant national standards.

4.2 The instruments in this document are those necessary for analysis. General laboratory instruments are no longer included.

4.3 The water used in this document is Grade II water or above as specified in GB/T6682-2008 for chromatographic analysis. If no other requirements are specified, it shall comply with the specifications of Grade III or above (including Grade III) water in GB/T6682-2008. All reagents used are of analytical grade (A.R.) unless other specifications are specified.

4.4 Unless otherwise specified, solutions in this document refer to aqueous solutions.

4.5 The chromatographic reference conditions in the chromatographic analysis test can be selected through experiments based on the conditions of the instrument and chromatographic column to completely separate the chromatographic peak of the internal standard component (ethanol) from the chromatographic peak of the component to be tested. The concentration of the standard working solution series can be appropriately adjusted according to the content of the component to be tested in the sample.

4.6 Stock solutions of standard substances used in this document (the stock solutions of internal standard substances are referred to as “internal standard solutions” (ethanol) in this document)

After preparation, transfer to a reagent bottle and store in a sealed container at 0℃~4℃.

4.7 When there are two or more analytical methods for the same test item, the first method shall be the arbitration method.

5 basic requirements

5.1 For the determination of samples, parallel tests shall be conducted and the analysis results shall be reported as the arithmetic mean of the measured data without conversion according to alcohol content.

5.2 The significant figures in the analytical method indicate the precision required to be achieved when pipetting or weighing.

5.3 Constant weight means that the difference between the two weighing values of the sample after drying is less than 2 mg.

6 Sensory evaluation

6.1 Principles

Wine tasters use their eyes, nose, mouth and other sensory organs to analyze and evaluate the color, appearance, aroma, taste, texture and style characteristics of liquor samples.

6.2 Wine Tasting Environment

The wine tasting room should have sufficient, soft and appropriate lighting; the temperature should be between 16℃ and 26℃, and the relative humidity should be between 30% and 70%; the air indoors should be fresh, without any aroma or unpleasant odors.

6.3 Wine Evaluation Requirements

6.3.1 Wine tasters should have sensitive sensory organs, undergo special training and assessment, meet the requirements of sensory evaluation, be familiar with the sensory evaluation terms of their own wines, and master the characteristics of relevant liquors.

6.3.2 Comments should be fair, scientific and accurate.

6.3.3 The dimensions of the standard wine tasting glass are shown in Figure 1. There are two types: with a stem (see Figure 1a) and without a stem (see Figure 1b). Both are made of colorless transparent glass, with a full capacity of 50 mL to 55 mL and a capacity of 15 mL to 20 mL at the maximum liquid level. If conditions permit, capacity scales may be added to the wall of the glass.

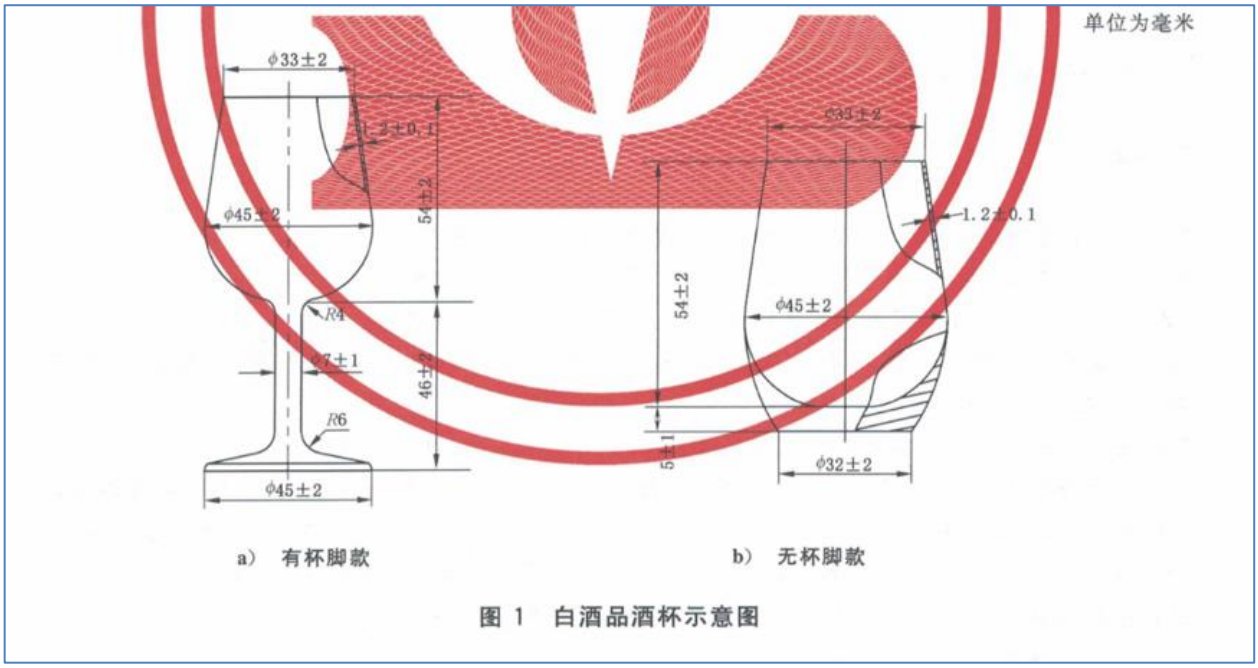

图 1 白酒品酒杯示意图

a) With a cup foot b) No stem

Figure 1 Schematic diagram of liquor tasting glass

6.4 Evaluation

6.4.1 Sample preparation

Place the liquor sample in an environment of 20℃~25℃ (or keep it warm in a water bath of 20℃~25℃) to balance the temperature, mark it with a code and conduct sensory evaluation. Before tasting, inject the liquor sample into a clean, dry tasting glass with an injection volume of 15mL~20mL.

Unit is millimeters

6.4.2 Color and appearance

Pick up the wine glass, and with a white wine evaluation table or white paper as the background, observe the wine sample from the front, from below, and from above to see if it has color and the depth of its color. Then shake it gently to observe the clarity of the wine, the presence of suspended matter and sediment, and record its color and appearance.

6.4.3 Aroma

General smelling: First, lift the wine glass, place it 10mm~20mm below your nose, tilt it 30°, lower your head slightly, and smell its static aroma in a steady and slow way. When smelling, you can only inhale the wine, not exhale. Then gently shake the wine glass to increase the evaporation and concentration of the aroma, then smell it and record its aroma.

Special case: Pour out the wine, let it sit for a while and then smell the empty cup to see the aroma.

6.4.4 Taste and mouthfeel

Pour the sample into a clean, dry wine glass, drink a small amount of sample (0.5 mL~2.0 mL) into your mouth, taste it carefully with your taste buds, and write down the taste and mouthfeel characteristics.

6.4.5 Style

Based on the aroma, taste, mouthfeel and other characteristics, and combined with the style characteristics of each liquor, a summary evaluation is made to determine whether it has a typical style or a unique style (personality).

7 total esters

7.1 Indicator method

7.1.1 Principle

Neutralize the free acid in the sample with alkali, then accurately add a certain amount of alkali, heat under reflux to saponify the esters, perform neutralization titration with standard sulfuric acid solution, and calculate the total ester content by the amount of acid consumed.

7.1.2 Instruments

7.1.2.1 All-glass distiller: 500 mL.

7.1.2.2 All-glass reflux device: reflux flask 1000mL, 250mL, condenser tube not shorter than 45cm.

7.1.2.3 Alkali burette: 25mL or 50mL.

7.1.2.4 Acid burette: 25mL or 50mL.

7.1.3 Reagents and solutions

7.1.3.1 Ethanol solution (95%, volume fraction): Measure 950 mL of ethanol, add 50 mL of water and mix well.

7.1.3.2 Sodium hydroxide standard titration solution [c(NaOH)=0.1 mol/L]: prepared and calibrated according to GB/T601.

7.1.3.3 Sodium hydroxide solution [c(NaOH)=3.5 mol/L]: Weigh 110 g of sodium hydroxide, dissolve in 100 mL of carbon dioxide-free water, shake well, inject into a polyethylene container, seal and place until the solution becomes clear, measure 18.9 mL of the supernatant, dilute to 100 mL with carbon dioxide-free water, and shake well.

7.1.3.4 Sulfuric acid standard titration solution [c(H_2SO_4)=0.1 mol/L]: prepared and calibrated according to GB/T 601.

7.1.3.5 Ethanol (ester-free) solution (40%, volume fraction): Measure 600 mL of 95% ethanol solution (7.1.3.1) into a 1000 mL reflux flask, add 5 mL of sodium hydroxide solution (7.1.3.3), heat and reflux for saponification for 1 hour. Then transfer to an all-glass distiller (7.1.2.1) for redistillation, and then prepare ethanol (ester-free) solution (40%, volume fraction).

7.1.3.6 Phenolphthalein indicator solution (10 g/L): prepared according to GB/T 603.

7.1.4 Test steps

7.1.4.1 Pipette 50.0 mL of sample into a 250 mL reflux flask, add 2 drops of phenolphthalein indicator solution (7.1.3.6), and titrate with sodium hydroxide standard titration solution (7.1.3.2) until the color turns slightly red and does not fade for 30 seconds (do not use excessive amounts), and record the number of milliliters of sodium hydroxide standard titration solution consumed.

7.1.4.2 Accurately add 25.00 mL of sodium hydroxide standard titration solution (7.1.3.2) (if the total ester content of the sample is high, add 50.00 mL), shake well, put in a few zeolites or glass beads, install a condenser (the cooling water temperature should be lower than 15°C), reflux in a boiling water bath for 30 min, remove and cool.

7.1.4.3 Titrate with sulfuric acid standard titration solution (7.1.3.4) until the red color completely disappears, and record the volume V_1 of sulfuric acid standard titration solution consumed. At the same time, take 50 mL of ethanol (ester-free) solution (40%, volume fraction) (7.1.3.5), perform a blank test in the same way as above, and record the volume V of sulfuric acid standard titration solution consumed.

7.1.5 Result calculation

The total ester content in the sample was calculated according to formula (1):

X1 -

C1 –

V_0 -

V_1 -

The total ester content in the sample is expressed as mass concentration (in terms of ethyl acetate) in grams per liter (g/L);

The actual molar concentration of the sulfuric acid standard titration solution is in moles per liter (mol/L);

-The volume of sulfuric acid standard titration solution consumed by the blank test sample, in milliliters (mL); The volume of sulfuric acid standard titration solution consumed by the sample, in milliliters (mL);

88 - the molar mass of ethyl acetate is in grams per mole (g/mol) [M(CH3COOC2H5)=88];

50.0 - volume of sample aspirated in milliliters (mL).

The calculation result is expressed to two decimal places.

7.1.6 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 2% of their arithmetic mean.

7.2 Potentiometric titration

7.2.1 Principle

Use alkali to neutralize the free acid in the sample, then add a certain amount of alkali and reflux for saponification. Use sulfuric acid standard titration solution for neutralization titration, take pH=8.70 as the indicated end point, and calculate the total ester content based on the amount of sulfuric acid standard titration solution consumed.

7.2.2 Instruments

7.2.2.1 All-glass distiller: 500 mL.

7.2.2.2 All-glass reflux device: reflux flask 1000mL, 250mL, condenser tube not shorter than 45cm.

7.2.2.3 Alkali burette: 25mL or 50mL.

7.2.2.4 Acid burette: 25mL or 50mL.

7.2.2.5 Potentiometric titrator (or acidity meter): accuracy 0.01 pH, with magnetic stirring device.

7.2.2.6 pH glass acid-base electrode.

7.2.3 Reagents and solutions

Same as 7.1.3.

7.2.4 Test steps

7.2.4.1 Install and debug the instrument according to the instruction manual and calibrate the position according to the solution temperature.

7.2.4.2 Pipette 50.0 mL of sample into a 250 mL reflux flask, add 2 drops of phenolphthalein indicator solution (7.1.3.6), and titrate with sodium hydroxide standard titration solution (7.1.3.2) until the color turns slightly red and does not fade for 30 seconds (do not use excessive amounts), and record the number of milliliters of sodium hydroxide standard titration solution consumed.

7.2.4.3 Accurately add 25.00 mL of sodium hydroxide standard titration solution (7.1.3.2) (if the total ester content of the sample is high, add 50.00 mL), shake well, put in a few zeolites or glass beads, install a condenser (the cooling water temperature should be lower than 15°C), reflux in a boiling water bath for 30 min, remove and cool. Transfer the sample solution into a 100 mL beaker, rinse the reflux bottle with 10 mL of water several times, and add the washing solution into the beaker.

7.2.4.4 Insert the electrode, put in a magnetic stirrer, place on the electromagnetic stirrer, and start stirring. In the initial stage, the sulfuric acid standard titration solution (7.1.3.4) can be added quickly. When the pH of the sample solution reaches 9.00, slow down the titration speed and add half a drop of solution each time until the pH reaches 8.70, which is the end point. Record the volume V_1 of the sulfuric acid standard titration solution consumed.

7.2.4.5 At the same time, draw 50.00 mL of ethanol (ester-free) solution (40%, volume fraction) (7.1.3.5), perform a blank test in the same manner as above, and record the volume V of the consumed sulfuric acid standard titration solution.

7.2.5 Result calculation

Same as 7.1.5.

7.2.6 Precision

Same as 7.1.6.

8 Total amount of acid esters

8.1 Indicator method

8.1.1 Principle

The free acid in the sample is neutralized with alkali, and then a certain amount of alkali is added, heated under reflux to saponify the esters, and the remaining alkali is neutralized with acid. The total amount of acid esters is obtained by calculating the total consumption of alkali.

8.1.2 Instruments

Same as 7.1.2.

8.1.3 Reagents and solutions

Same as 7.1.3.

8.1.4 Test steps

8.1.4.1 Neutralize the free acid in the sample with alkali. The test steps are the same as 7.1.4.1. Record the volume V2 of the sodium hydroxide standard titration solution consumed.

8.1.4.2 Heat to reflux and neutralize the remaining alkali. The test steps are the same as 7.1.4.2 to 7.1.4.3. Record the volume of sulfuric acid standard titration solution V consumed by the blank test sample and the volume of sulfuric acid standard titration solution V_1 consumed by the sample.

8.1.5 Result calculation

The total amount of acid esters in the sample was calculated according to formula (2).

In the formula:

[C3XV2+C1X (V-V_1)]×1000 X2 =

50.0

C3 - actual molar concentration of sodium hydroxide standard titration solution, in moles per liter (mol/L);

V2 - the volume of sodium hydroxide standard titration solution consumed by the total acid in the sample, in milliliters (mL);

V_2 - the total amount of acid esters in the sample, in millimole per liter (mmol/L);

C1 - actual molar concentration of sulfuric acid standard titration solution, in moles per liter (mol/L);

Vo - the volume of sulfuric acid standard titration solution consumed by the blank test sample, in milliliters (mL);

V_1--The volume of sulfuric acid standard titration solution consumed by the sample, in milliliters (mL);

50.0 - Volume of sample aspirated in milliliters (mL).

The calculation result is expressed to one decimal place.

8.1.6 Precision

Same as 7.1.6.

8.2 Potentiometric titration

8.2.1 Principle

Same as 8.1.1.

8.2.2 Instruments

Same as 7.2.2.

8.2.3 Reagents and solutions

Same as 7.2.3.

8.2.4 Test steps

8.2.4.1 Install and debug the instrument according to the instruction manual and calibrate the pH electrode according to the solution temperature.

8.2.4.2 Pipette 50.0 mL of sample (if a composite electrode is used, the sampling volume may be increased as appropriate) into a 100 mL beaker, insert a pH glass acid-base electrode, put in a magnetic rotor, place it on a magnetic stirring device, and start stirring. In the initial stage, sodium hydroxide standard titration solution (7.1.3.2) can be added quickly. When pH = 8.00, slow down the titration speed and add half a drop of solution each time until pH = 9.00 is the end point. Record the volume V2 of sodium hydroxide standard titration solution consumed, then transfer the sample solution to a distillation flask, rinse the beaker with a small amount of water several times, and transfer it to the distillation flask.

8.2.4.3 Heat to reflux and neutralize the remaining alkali. The test steps are the same as 7.2.4.3 to 7.2.4.5. Record the volume of sulfuric acid standard titration solution V consumed by the blank test sample and the volume of acid standard titration solution V_1 consumed by the sample.

8.2.5 Result calculation

Same as 8.1.5.

8.2.6 Precision

Same as 7.1.6.

9 Solids

9.1 Principle

After evaporation and drying of liquor, non-volatile substances remain in the dish and are determined by weighing method after constant weight.

9.2 Instruments

9.2.1 Electric drying oven: temperature control accuracy ±2°C.

9.2.2 Analytical balance: sensitivity 0.1 mg.

9.2.3 Porcelain evaporating dish or glass evaporating dish: 100 mL.

9.2.4 Dryer: use color-changing silica gel as desiccant.

9.3 Test steps

GB/T 10345-2022

Take 50.0 mL of the sample and pour it into a 100 mL porcelain evaporating dish or glass evaporating dish that has been dried to constant weight. Place it in a boiling water bath and evaporate it to dryness. Then place the evaporating dish in a (103±2)℃ electric drying oven, dry it for 2 hours, take it out, place it in a desiccator for 30 minutes, and weigh it. Then place it in a (103±2)℃ electric drying oven, dry it for 1 hour, take it out, place it in a desiccator for 30 minutes, and weigh it. Repeat the above operation until constant weight is reached.

9.4 Result calculation

The solid content in the sample was calculated according to formula (3).

X3= m-m1 50.0 × 1 000

In the formula:

X3 ————Solid content in the sample, expressed as mass concentration, in grams per liter (g/L);

m————the mass of the solid matter and the evaporating dish, in grams (g);

————The mass of the evaporating dish, in grams (g); m1-

50.0 ————The volume of the sample taken, in milliliters (mL).

The calculation result is expressed to two decimal places.

9.5 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 5% of their arithmetic mean.

10 Ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate, n-propanol, β-phenylethanol

10.1 Principle

After the sample is gasified, it is separated by a chromatographic column. Since the components to be measured have different distribution coefficients in the gas and liquid phases, the components to be measured after separation have different distribution coefficients in the gas and liquid phases.

The measured components flow out of the chromatographic column in sequence and enter the hydrogen flame ionization detector for detection. The qualitative analysis is performed based on the retention value of each component peak on the chromatogram compared with the standard product, and the quantitative analysis is performed using the peak area (or peak height) with the internal standard method.

10.2 Reagents and Materials

10.2.1 Ethanol: chromatographic grade.

10.2.2 Standard substances such as ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate, n-propanol, 3-phenylethanol, etc.: purity ≥ 99%, or standard substances certified by the state and awarded with standard substance certificates.

10.2.3 Tert-amyl alcohol, n-amyl acetate, n-butyl acetate standard substances: (ethanol) purity ≥ 99%, or standard substances certified by the state and awarded with standard substance certificates, used as internal standards.

10.2.4 Ethanol solution (50%, volume fraction): Measure 250 mL of ethanol (10.2.1), add 250 mL of water, and mix thoroughly.

10.2.5 n-Propanol standard substance stock solution (10000 mg/L): Accurately weigh 1.0 g (accurate to 1 mg) of n-propanol standard substance (10.2.2), add an appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

10.2.6 β-Phenylethanol standard substance stock solution (500 mg/L): Accurately weigh 0.05 g (accurate to 1 mg) of β-phenylethanol standard substance (10.2.2), add an appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

10.2.7 Mixed stock solution of ester standard substances (ethyl acetate, ethyl caproate and ethyl lactate are all 25000 mg/L, and ethyl butyrate is 2500 mg/L): accurately weigh 2.50 g (accurate to 1 mg) of ethyl acetate standard substance (10.2.2), ethyl caproate standard substance (10.2.2), ethyl lactate standard substance (10.2.2), and 0.25 g (accurate to 1 mg) of ethyl butyrate standard substance (10.2.2), respectively, add appropriate amount of ethanol (10.2.1) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

10.2.8 tert-Amyl alcohol and n-amyl acetate mixed internal standard solution (ethanol) (20000 mg/L): used as internal standard when using capillary chromatographic columns. Accurately weigh 2.0 g (accurate to 1 mg) of tert-amyl alcohol standard substance (10.2.3) and n-amyl acetate standard substance (10.2.3), add appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

10.2.9 n-Butyl acetate internal standard solution (ethanol) (20000 mg/L): used as internal standard when using packed chromatographic columns. Accurately weigh 2.0 g (accurate to 1 mg) of n-butyl acetate standard substance (10.2.3), add an appropriate amount of ethanol solution (50%, volume fraction) (10.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

10.2.10 Ester and alcohol series mixed standard working solution 1 (applicable to 10.4.1.1): pipette 0.1 mL, 0.2 mL, 0.4 mL, 0.6 mL, and 1.0 mL of n-propanol standard substance stock solution (10.2.5), β-phenylethanol standard substance stock solution (10.2.6), and ester standard substance mixed stock solution (10.2.7) into five 10 mL volumetric flasks, then add 0.1 mL of tert-amyl alcohol and n-amyl acetate internal standard (ethanol) solution (10.2.8), respectively, make up to volume with ethanol solution (50%, volume fraction) (10.2.4), and mix thoroughly. A series of mixed standard working solutions were prepared with 250 mg/L, 500 mg/L, 1000 mg/L, 1500 mg/L and 2500 mg/L of ethyl acetate, 50 mg/L, 100 mg/L, 150 mg/L and 250 mg/L of ethyl butyrate, 100 mg/L, 200 mg/L, 400 mg/L, 600 mg/L and 1000 mg/L of n-propanol and 5 mg/L, 10 mg/L, 20 mg/L, 30 mg/L and 50 mg/L of ß-phenylethanol for immediate use.

10.2.11 Ester and alcohol series mixed standard working solution 2 (applicable to 10.4.1.2): pipette 0.1 mL, 0.2 mL, 0.4 mL, 0.6 mL, and 1.0 mL of n-propanol standard substance stock solution (10.2.5) and ester standard substance stock solution (10.2.7) into five 10 mL volumetric flasks, then add 0.1 mL of n-butyl acetate internal (ethanol) standard solution (10.2.9) to each flask, make up to volume with ethanol solution (50%, volume fraction) (10.2.4), and mix thoroughly. Prepare a series of mixed standard working solutions of 250 mg/L, 500 mg/L, 1000 mg/L, 1500 mg/L, 2500 mg/L for ethyl acetate, 500 mg/L, 100 mg/L, 150 mg/L, 2500 mg/L for ethyl lactate; 25 mg/L, 50 mg/L, 100 mg/L, 150 mg/L, 250 mg/L for ethyl butyrate; 100 mg/L, 200 mg/L, 400 mg/L, 600 mg/L, 1000 mg/L for n-propanol, and use them immediately after preparation.

10.3 Instruments and Equipment

10.3.1 Gas chromatograph, equipped with flame ionization detector (FID).

10.3.2 Analytical balance: sensitivity is 0.1 mg.

10.3.3 Pipette: 0.1 mL~1.0 mL.

10.4 Test procedures

10.4.1 Chromatographic reference conditions

10.4.1.1 Capillary columns

10.4.1.1.1 Polyethylene glycol capillary column (60m×0.25 mm×0.25 μm) or (50m×0.25mm×0.20 μm) or other chromatographic columns with equivalent analytical performance.

10.4.1.1.2 Heating procedure: Initial temperature 35°C, hold for 1 min, increase to 70°C at 3.0°C/min, increase to 180°C at 3.5°C/min, then increase to 210°C at 15°C/min, hold for 6 min.

10.4.1.1.3 Detector temperature: 250°C.

10.4.1.1.4 Inlet temperature: 250 °C.

10.4..1.5 Constant flow mode: 1.0 mL/min.

10.4.1.1.6 Injection volume: 1.0 μL.

10.4.1.1.7 Diversion ratio: 40:1.

10.4.1.2 Filling the chromatographic column

10.4.1.2.1 Filling column: The column length shall not be less than 2m.

10.4.1.2.2 Carrier: Chromosorb W (AW) or white carrier 102 (acid washed, silanized), 80 mesh to 100 mesh (0.18 mm to 0.125 mm).

10.4.1.2.3 Fixative: 20% dinonyl phthalate (DNP) plus 7% Tween 80, or 10% polyethylene glycol (PEG) 1500 or PEG 20 mol/L.

10.4.1.2.4 Carrier gas (high purity nitrogen): flow rate is 150 mL/min.

10.4.1.2.5 Hydrogen: flow rate is 40 mL/min.

10.4.1.2.6 Air: flow rate is 400 mL/min.

10.4.1.2.7 Detector temperature: 150°C.

10.4.1.2.8 Inlet temperature: 150 °C.

10.4.1.2.9 Column temperature: 90°C, isothermal.

10.4.2 Drawing a standard curve

10.4.2.1 Capillary columns

Take an appropriate amount of ester and alcohol series mixed standard working solution 1 (10.2.10), inject and measure according to the chromatographic reference conditions (10.4.1.1), and perform qualitative analysis based on the retention time of the single standard chromatographic peak of each ester and alcohol component. The ratio of the concentration of each ester and alcohol to the corresponding internal standard concentration (ethanol) is the horizontal axis, and the ratio of the peak area of each ester and alcohol component to the corresponding internal standard peak area is the vertical axis. Among them, tert-amyl alcohol is used as the internal standard (ethanol) for n-propanol and 3-phenylethanol; n-amyl acetate (ethanol) is used as the internal standard for ethyl acetate, ethyl butyrate, ethyl hexanoate, and ethyl lactate. Draw a standard working curve.

10.4.2.2 Filling the chromatographic column

Pipette an appropriate amount of ester and alcohol series mixed standard working solution 2 (10.2.11), inject and measure according to the chromatographic reference conditions (10.4.1.2), and perform qualitative analysis based on the retention time of the single standard chromatographic peak of each ester and alcohol component. Draw a standard working curve with the ratio of the concentration of each ester and alcohol component to the concentration of the n-butyl acetate internal standard as the horizontal axis and the ratio of the peak area of each ester and alcohol component to the peak area of the n-butyl acetate internal standard as the vertical axis.

10.4.3 Sample determination

Take an appropriate amount of sample and place it in a 10mL volumetric flask, add 0.1mL of internal standard solution (ethanol) (10.2.8 or 10.2.9), use the same sample to make up to volume, mix thoroughly, and measure the sample according to the chromatographic reference conditions (10.4.1). Obtain the ratio of the mass concentration of each component to be measured in the sample to the mass concentration of the corresponding internal standard(ethanol) from the standard working curve;, and then calculate the content of each component to be measured in the sample according to the concentration of the internal standard (ethanol) corresponding to the component to be measured;.

10.5 Result calculation

10.5.1 The contents of ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate and n-propanol in the sample shall be calculated according to formula (4).

$$. X_i = (I_i * \rho_i)/ 000 \quad (4)$$

In the formula:

X_i - the content of ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate and n-propanol in the sample, expressed as mass concentration in grams per liter (g/L);

I_i - the ratio of the concentration of ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate, and n-propanol in the test solution to the corresponding internal standard concentration(ethanol) is obtained from the standard curve;

ρ_i - mass concentration of ethyl acetate, ethyl butyrate, ethyl caproate, ethyl lactate and n-propanol corresponding to the internal standard (ethanol), in milligrams per liter (mg/L);

1000 - unit conversion factor.

The calculation result is expressed to two decimal places.

10.5.2 The content of 3-phenylethanol in the sample is calculated according to formula (5).

$$X_{ß\text{-phenylethanol}} = I\text{-}_{ß\text{-phenylethanol}} \times \rho\text{-}_{ß\text{-phenylethanol}} \quad (5)$$

$X_{ß\text{-phenylethanol}}$ - the content of ß-phenylethanol in the sample, expressed as mass concentration in milligrams per liter (mg/L);

$I\text{-}_{ß\text{-phenylethanol}}$ l - ratio of the mass concentration of ß-phenylethanol in the test solution to the mass concentration of tert-amyl alcohol internal standard (ethanol) is obtained from the standard curve;

$\rho\text{-}_{ß\text{-phenylethanol}}$ - mass concentration of tert-amyl alcohol internal standard (ethanol), in milligrams per liter (mg/L).

The calculation result is expressed as an integer.

10.6 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 5% of their arithmetic mean.

11 acetic acid

11.1 Principle

Same as 10.1.

11.2 Reagents and Materials

11.2.1 Ethanol: chromatographic grade.

11.2.2 Acetic acid standard substance: purity ≥ 99%, or standard substance certified by the state and awarded with a standard substance certificate.

11.2.3 2-Ethylbutyric Acid Standard Material: purity ≥ 99%, or standard material certified by the state and awarded with a standard material certificate, used as internal standard.

11.2.4 Ethanol solution (50%, volume fraction): Same as 10.2.4.

11.2.5 Acetic acid standard substance stock solution (20000 mg/L): Accurately weigh 2.0 g (accurate to 1 mg) of acetic acid standard substance (11.2.2), add appropriate amount of ethanol solution (50%, volume fraction) (11.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

11.2.6 2-ethylbutyric acid internal standard solution (ethanol) (20000 mg/L): Weigh 2.0 g (accurate to 1 mg) of 2-ethylbutyric acid standard substance (11.2.3), add appropriate amount of ethanol solution (50%, volume fraction) (11.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

11.2.7 Acetic acid series standard working solutions: Accurately pipette 0.2 mL, 0.4 mL, 0.6 mL, 0.8 mL, and 1.0 mL of acetic acid standard stock solution (11.2.5) into 10 mL volumetric flasks, then add 0.1 mL of 2-ethylbutyric acid internal standard solution (11.2.6) respectively. Make up to volume with ethanol solution (50%, volume fraction) (11.2.4), shake well, and prepare 400 mg/L, 800 mg/L, 1200 mg/L, 1600 mg/L, and 2000 mg/L acetic acid series standard working solutions for immediate use.

11.3 Instruments and Equipment

Same as 10.3.

11.4 Test procedures

11.4.1 Chromatographic reference conditions

Same as 10.4.1.1.

11.4.2 Drawing a standard curve

Take an appropriate amount of acetic acid series standard working solution (11.2.7), measure according to the chromatographic reference conditions (11.4.1), and draw a standard curve with the ratio of the concentration of acetic acid series standard working solution to the concentration of 2-ethylbutyric acid internal standard (ethanol) solution as the horizontal axis and the ratio of the peak area of acetic acid series standard working solution to the peak area of 2-ethylbutyric acid internal standard solution as the vertical axis.

11.4.3 Sample determination

Pipette an appropriate amount of sample into a 10mL volumetric flask, add 0.1mL of 2-ethylbutyric acid internal standard (ethanol) solution (11.2.6), use the same sample to make up to volume, and mix thoroughly. Determine according to the chromatographic reference conditions (11.4.1), and perform qualitative analysis based on the retention time of the acetic acid standard substance and the retention time of acetic acid in the sample to be tested. According to the ratio of the peak area of acetic acid in the test solution to that of the 2-ethylbutyric acid internal standard (ethanol) solution, obtain the ratio of the concentration of acetic acid in the test solution to that of the 2-ethylbutyric acid internal standard solution from the standard working curve, and then calculate the content of acetic acid in the sample based on the concentration of the 2-ethylbutyric acid internal standard (ethanol) solution.

11.5 Result calculation

Same as 10.5.1.

11.6 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 10% of their arithmetic mean.

12 caproic acid

12.1 Gas chromatography

12.1.1 Principle

Same as 10.1.

12.1.2 Reagents

12.1.2.1 Ethanol: chromatographic grade.

12.1.2.2 Hexanoic acid standard substance: purity ≥ 99.5%, or standard substance certified by the state and awarded a standard substance certificate.

12.1.2.3 2-ethylbutyric Acid Standard Material: purity ≥ 99%, or standard material certified by the state and awarded with a standard material certificate, used as internal standard.

12.1.2.4 Ethanol solution (50%, volume fraction): Same as 10.2.4.

12.1.2.5 Hexanoic acid standard substance stock solution (10000 mg/L): Weigh 1.0 g (accurate to 1 mg) of hexanoic acid standard substance (12.1.2.2) and add appropriate amount of ethanol solution (50%, volume fraction) (11.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

12.1.2.6 2-ethylbutyric acid internal standard (ethanol) solution (20000 mg/L): same as 11.2.6.

12.1.2.7 Hexanoic acid series standard working solutions: Accurately pipette 0.2 mL, 0.4 mL, 0.6 mL, 0.8 mL, and 1.0 mL of the hexanoic acid standard stock solution (12.1.2.5) into a 10 mL volumetric flask, then add 0.1 mL of the 2-ethylbutyric acid internal standard (ethanol) solution (12.1.2.6) respectively, and use ethanol solution (50%, volume fraction) (12.1.2.4) to make up to volume, and mix thoroughly. Prepare 200 mg/L, 400 mg/L, 600 mg/L, 800 mg/L, and 1000 mg/L of the hexanoic acid series standard working solutions, and use them immediately after preparation.

12.1.3 Instruments and equipment

Same as 10.3.

12.1.4 Test procedures

12.1.4.1 Chromatographic reference conditions

Same as 10.4.1.1.

12.1.4.2 Drawing a standard curve

Except that the standard substance is changed to hexanoic acid series standard working solution (12.1.2.7), other operations are the same as 11.4.2.

12.1.4.3 Sample determination

Except that the standard substance is changed to hexanoic acid series standard working solution (12.1.2.7), other operations are the same as 11.4.3.

12.1.5 Result calculation

Same as 10.5.1.

12.1.6 Precision

Same as 11.6.

12.2 Ion Chromatography

12.2.1 Principle

After the sample was diluted with water, it was separated by anion exchange chromatography column and measured by conductivity detector. The qualitative analysis was performed by retention time and the quantitative analysis was performed by external standard method.

12.2.2 Reagents and materials

Unless otherwise specified, all reagents were of analytical grade and water was grade 1 water as specified in GB/T 6682-2008.

12.2.2.1 Ethanol: chromatographic grade.

12.2.2.2 Sodium carbonate.

12.2.2.3 Sodium bicarbonate.

12.2.2.4 Sodium hydroxide.

12.2.2.5 Hexanoic acid standard substance: same as 12.1.2.2.

12.2.2.6 Sodium carbonate solution (1 mmol/L): Accurately weigh 0.106 g of sodium carbonate solution, dissolve it in appropriate amount of water, make up to 1000 mL, and mix well.

12.2.2.7 Mixed solution of sodium carbonate and sodium bicarbonate (sodium carbonate 13 mmol/L, sodium bicarbonate 2 minol/L): Accurately weigh 1.378 g sodium carbonate solution (12.2.2.6) and 0.168 g sodium bicarbonate (12.2.2.3), dissolve in appropriate amount of water, make up to 1000 mL, and mix thoroughly.

12.2.2.8 Sodium hydroxide solution (100 mmol/L): weigh 40 g of sodium hydroxide (12.2.2.4), dissolve it in appropriate amount of water, make up to 1000 mL, and mix well. You can also use an automatic eluent generator (H type

12.2.2.9 Ethanol solution (50%, volume fraction, same as 10

12.2.2.10 Caproic acid standard stock solution (1000 mg/L, take 0.1 g accurate to 1 mg) Add an appropriate amount of ethanol solution (50%, volume fraction) (12.2.2.9) to the caproic acid standard substance (12.2.2.5) to dissolve it, transfer it to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

12.2.2.11 Caproic acid series standard working solutions: pipette appropriate amounts of caproic acid standard stock solutions (12.2.2.10) respectively and prepare a series of standard working solutions of 5.0 mg/L, 10.0 mg/L, 15.0 mg/L, 20.0 mg/L and 30.0 mg/L with water for immediate use.

12.2.3 Instruments and equipment

12.2.3.1 Ion chromatograph: equipped with conductivity detector and gradient elution system.

12.2.3.2 Analytical balance: sensitivity 0.1 mg.

12.2.3.3 Organic microporous membrane: 0.45 μm.

12.2.4 Test steps

12.2.4.1 Sample preparation

Accurately transfer 1.0 mL of sample into a 50 mL volumetric flask, dilute to volume with water, and mix well; filter the diluted sample through an organic microporous filter membrane, collect 1 mL of filtered sample in a sample bottle for testing.

12.2.4.2 Chromatographic reference conditions

12.2.4.2.1 Carbonate/bicarbonate elution system

The chromatographic reference conditions for the carbonate/bicarbonate elution system are as follows:

————Anion exchange column: polystyrene and divinylbenzene copolymer filler, quaternary ammonium salt active group 4.0mm×250mm (with the same type of guard column 4.0mm×5mm), or ion chromatography column with equivalent performance;

- Eluent: A is sodium carbonate solution (12.2.2.6), B is a mixed solution of sodium carbonate and sodium bicarbonate (12.2.2.7);

-Suppressor: Ultra-micro filled inlay structure;

Flow rate: 0.7 mL/min;

- Column oven temperature: room temperature;

Injection volume: 20 μL;

Detector: Conductivity detector;

Detection cell temperature: 35 °C;

——Gradient elution program is shown in Table 1.

Table 1 Gradient elution program

序号	时间/min	A/%	B/%
1	0	100	0
2	18	100	0
3	18	0	100
4	23	0	100
5	23	100	0
6	45	100	0

12.2.4.2.2 Hydroxide elution system

The chromatographic reference conditions for the hydroxide elution system are as follows:

- Anion chromatography column: divinylbenzene-ethylvinylbenzene copolymer filler, alkyl quaternary ammonium salt exchange functional group, 4.0mm×250mm (with the same type of guard column 4.0mm×5mm), or ion chromatography column with equivalent performance;

-Eluent: generated by an online generator of hydroxide eluent;

-Suppressor: Self-circulation mode or equivalent performance suppressor, suppression current 93mA;

- Flow rate: 1.5 mL/min;

- Column oven temperature: 35°C;

Injection volume: 20 μL;

-Detector: conductivity detector;

- Detection cell temperature: 35 ℃;

—Gradient elution program is shown in Table 2.

Table 2 Gradient elution program

序号	时间/min	淋洗浓度/(mmol/L)
1	0	5
2	15	5
3	20	60

Table 2 Gradient elution program (continued)

表 2　梯度洗脱程序（续）

序号	时间/min	淋洗浓度/(mmol/L)
4	25	60
5	26	5
6	33	5

12.2.4.3 Standard curve drawing

Take the caproic acid series standard working solution (12.2.2.11) and measure it according to the chromatographic reference conditions (12.2.4.2). Perform qualitative analysis based on the retention time of the caproic acid chromatographic peak. Draw a standard curve with the concentration of the caproic acid series standard working solution as the horizontal axis and the peak area as the vertical axis.

12.2.4.4 Sample determination

Inject the prepared sample (12.2.4.1) into the ion chromatograph, determine the hexanoic acid chromatographic peak area according to the chromatographic reference conditions (12.2.4.2), and obtain the hexanoic acid content in the test solution according to the standard working curve.

12.2.4.5 Result calculation

The content of hexanoic acid in the sample is calculated according to formula (6).

In the formula:

$$X_4 = \rho_1/1000*n \quad (6)$$

X_4- the content of caproic acid in the sample, expressed as mass concentration, in grams per liter (g/L);

ρ_1 - the mass concentration of caproic acid in the test solution obtained from the standard curve, in milligrams per liter (mg/L)

n - sample dilution multiple.

The calculation result is expressed to two decimal places.

12.2.5 Precision

Same as 11.6.

13 Ethyl propionate

13.1 Principle

Same as 10.1.

13.2 Reagents and Materials

13.2.1 Ethanol; chromatographic grade.

13.2.2 Ethyl propionate standard substance: purity ≥ 99%, or standard substance certified by the state and awarded a standard substance certificate.

13.2.3 n-amyl acetate standard (ethanol) substance: purity ≥ 99%, or standard substance certified by the state and awarded with standard substance certificate, used as internal standard (ethanol) .

13.2.4 Ethanol solution (50%, volume fraction): Same as 10.2.4.

13.2.5 n-amyl acetate internal standard (ethanol) solution (10000 mg/L): Weigh 0.5 g (accurate to 1 mg) of n-amyl acetate standard substance (13.2.3), add an appropriate amount of ethanol solution (50%, volume fraction) (13.2.4) to dissolve, transfer to a 50 mL volumetric flask, make up to volume, and mix thoroughly.

13.2.6 Ethyl propionate standard substance stock solution (1000 mg/L): Accurately weigh 0.1 g (accurate to 1 mg) of ethyl propionate standard substance (13.2.2), add an appropriate amount of ethanol solution (50%, volume fraction) (13.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume, and mix thoroughly.

13.2.7 Ethyl propionate series standard working solutions: pipette 0.05 mL, 0.1 mL, 0.2 mL, 0.3 mL, and 0.4 mL of ethyl propionate stock solution (13.2.6) into five 10 mL volumetric flasks, add 0.1 mL of n-amyl acetate internal standard (ethanol) solution (13.2.5) to each flask, make up to volume with ethanol solution (50%, volume fraction) (13.2.4), mix thoroughly, and prepare 5.0 mg/L, 10.0 mg/L, 20.0 mg/L, 30.0 mg/L, and 40.0 mg/L series standard working solutions in sequence for immediate use.

13.3 Instruments and Equipment

Same as 10.3.

13.4 Test procedures

13.4.1 Chromatographic reference conditions

13.4.1.1 Medium polarity stationary phase: 6% cyanopropylbenzene, 94% dimethylsiloxane (30m×0.53mm×3.0 μm) or other chromatographic columns with equivalent analytical performance.

13.4.1.2 Column temperature: Initial temperature 35°C, maintain for 8 min, increase to 160°C at 10.0°C/min, maintain for 5 min.

13.4.1.3 Detector temperature: 230 °C.

13.4.1.4 Inlet temperature: 230 °C.

13.4.1.5 Carrier gas flow rate: 2.0 mL/min.

13.4.1.6 Injection volume: 1.0 μL.

13.4.1.7 Split ratio: 30:1.

13.4.2 Drawing a standard curve

Take an appropriate amount of ethyl propionate series standard working solution (13.2.7), inject and measure according to the chromatographic reference conditions (13.4.1), and perform qualitative analysis based on the retention time of the chromatographic peak of the ethyl propionate standard. Draw the standard working curve with the ratio of the concentration of ethyl propionate to the concentration of n-amyl acetate internal standard solution as the horizontal axis and the ratio of the peak area of ethyl propionate to the peak area of n-amyl acetate internal standard (ethanol) solution as the vertical axis.

13.4.3 Sample determination

Pipette an appropriate amount of sample into a 10 mL volumetric flask, add 0.1 mL of n-amyl acetate internal standard solution (13.2.5), use the same sample to make up to volume, and mix thoroughly. Determine the peak area of ethyl propionate in the test solution and the peak area of n-amyl acetate internal standard (ethanol) solution according to the chromatographic reference conditions (13.4.1). According to the ratio of the peak area of ethyl propionate in the test solution to that of n-amyl acetate internal standard (ethanol) solution, obtain the ratio of the concentration of ethyl propionate in the test solution to that of n-amyl acetate internal standard (ethanol) solution I from the standard working curve, and then calculate the content of ethyl propionate in the sample according to the mass concentration p2 of n-amyl acetate internal standard (ethanol) solution.

13.5 Result calculation

The content of ethyl propionate in the sample was calculated according to formula (7).

In the formula:

$$X_5 = I * \rho_2 \quad (7)$$

X_5 - the content of ethyl propionate in the sample, expressed as mass concentration, in milligrams per liter (mg/L);

I - obtain the ratio of the mass concentration of ethyl propionate in the test solution to the mass concentration of n-pentyl acetate internal standard (ethanol) from the standard curve;

ρ_2 - mass concentration of n-pentyl acetate internal standard, in milligrams per liter (mg/L).

The calculation result is expressed to one decimal place.

13.6 Precision

Same as 10.6.

14 Dibasic acid (pimelic acid, suberic acid, azelaic acid) diethyl ester

14.1 Principle

Same as 10.1.

14.2 Reagents and Materials

14.2.1 Ethanol: chromatographic grade.

14.2.2 Standard substances of diethyl pimelate, diethyl suberate and diethyl azelate: purity ≥ 99%, or standard substances certified by the state and awarded with standard substance certificates.

14.2.3 Tetradecanol standard substance: purity ≥ 99%, or standard substance certified by the state and awarded with standard substance certificate, used as internal standard (ethanol)

14.2.4 Ethanol solution (30%, volume fraction): Measure 30 mL of ethanol, add 70 mL of water, and mix thoroughly.

14.2.5 Stock solution of mixed standard substance of diethyl pimelate, diethyl suberate and diethyl azelaic acid (200 mg/L): Weigh 0.02 g (accurate to 1 mg) of diethyl pimelate, diethyl suberate and diethyl azelaic acid standard substance (14.2.2) respectively, add ethanol solution (30%, volume fraction) (14.2.4) to dissolve, transfer to a 100 mL volumetric flask, make up to volume and mix thoroughly.

14.2.6 Tetradecanol internal standard (ethanol) solution (1000 mg/L): Weigh 0.1 g (accurate to 1 mg) of tetradecanol standard substance (14.2.3), add appropriate amount of ethanol (14.2.1) to dissolve, transfer to a 100 mL constant volume flask, adjust to volume, and mix thoroughly.

14.2.7 Intermediate solution of mixed standard substance of diethyl pimelate, diethyl suberate and diethyl azelaic acid (20 mg/L): Pipette 1 mL of mixed standard substance stock solution of diethyl pimelate, diethyl suberate and diethyl azelaic acid (14.2.5) into a 10 mL volumetric flask, make up to volume with ethanol solution (30%, volume fraction) (14.2.4), and mix thoroughly.

14.2.8 Working solutions of mixed standard substances of diethyl pimelate, diethyl suberate and diethyl azelaic acid: take 0.2 mL, 0.4 mL, 0.6 mL, 1.0 mL and 2.0 mL of the intermediate solutions of mixed standard substances of diethyl pimelate, diethyl suberate and diethyl azelaic acid (14.2.7) respectively, and add 0.1 mL of tetradecanol internal standard solution (14.2.6) into five 10 mL volumetric flasks respectively. Make up to volume with ethanol solution (30%, volume fraction) (14.2.4), mix thoroughly and prepare 0.4 mg/L, 0.8 mg/L, 1.2 mg/L, 2.0 mg/L and 4.0 mg/L mixed standard working solutions in sequence. Prepare and use immediately.

14.3 Instruments and Equipment

Same as 10.3.

14.4 Test procedures

14.4.1 Chromatographic reference conditions

14.4.1.1 Chromatographic reference conditions 1

Same as 10.4.1.1.

14.4.1.2 Chromatographic reference conditions 2

14.4.1.2.1 Chromatographic column: polyethylene glycol gas chromatography column (30m×0.32 mm×0.25 μm) or equivalent chromatographic column.

14.4.1.2.2 Column temperature: Initial temperature 50°C, maintain for 3 min, increase to 170°C at 10.0°C/min, then increase to 180°C at 5°C/min, maintain for 5 min, then increase to 210°C at 20°C/min, maintain for 3 min.

14.4.1.2.3 Detector temperature: 250 °C.

14.4.1.2.4 Inlet temperature: 250℃.

14.4.1.2.5 Constant pressure mode - column pressure: 82.74 kPa (12 psi).

14.4.1.2.6 Injection volume: 1.0 μL.

14.4.1.2.7 Split ratio: 20:1.

14.4.2 Drawing a standard curve

Take an appropriate amount of the mixed standard working solution of diethyl pimelate, diethyl suberate and diethyl azelaic acid (14.2.8), inject and measure according to the chromatographic reference conditions (14.4.1), and make qualitative analysis based on the retention time of the chromatographic peaks of the single standard products of diethyl pimelate, diethyl suberate and diethyl azelaic acid. Draw the standard working curve with the ratio of the concentration of diethyl pimelate, diethyl suberate and diethyl azelaic acid to the concentration of the tetradecanol internal standard (ethanol) solution as the horizontal axis and the ratio of the peak area of diethyl pimelate, diethyl suberate and diethyl azelaic acid to the peak area of the tetradecanol internal standard solution as the vertical axis.

14.4.3 Sample determination

Pipette an appropriate amount of sample into a 10 mL volumetric flask, add 0.1 mL of tetradecanol internal standard solution (14.2.6), use the same sample to make up to volume, and shake thoroughly. Determine the peak area of diethyl pimelate, diethyl suberate, diethyl azelaic acid and the peak area of tetradecanol internal standard in the test solution according to the chromatographic reference conditions (14.4.1). According to the ratio of the peak area of diethyl pimelate, diethyl suberate, diethyl azelaic acid to the tetradecanol internal standard in the test solution, obtain the ratio I of the concentration of diethyl pimelate, diethyl suberate, diethyl azelaic acid to the tetradecanol internal standard (ethanol) solution in the test solution from the standard working curve, and then calculate the content of diethyl pimelate, diethyl suberate, diethyl azelaic acid in the sample according to the concentration of the tetradecanol internal standard (ethanol) solution.

14.5 Result calculation

14.5.1 The contents of diethyl pimelate, diethyl suberate and diethyl azelate in the sample shall be calculated according to formula (8).

$$X_6 = I_i * \rho_3 \quad (8)$$

In the formula:

X_6 --- The content of diethyl pimelate, diethyl suberate and diethyl azelate in the sample, expressed as mass concentration in milligrams per liter (mg/L);

I_i - obtain the ratio of the mass concentration of diethyl pimelate, diethyl suberate, and diethyl azelate in the test solution to the mass concentration of the tetradecanol internal standard (ethanol) from the standard curve;

ρ_3 - the mass concentration of tetradecanol internal standard, in milligrams per liter (mg/L).

The calculation result is expressed to two decimal places.

14.5.2 The total amount of diethyl ester of dibasic acid in the sample is calculated according to formula (9).

In the formula:

$$X_7 = \Sigma X_i \quad (9)$$

X_7 - the total amount of diethyl esters of dibasic acids (pimelic acid, suberic acid, azelaic acid) in the sample, expressed as mass concentration in milligrams per liter (mg/L);

X_i - contents of diethyl pimelate, diethyl suberate and diethyl azelate in the sample, in milligrams per liter (mg/L).

The calculation result is expressed to one decimal place.

14.6 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions shall not exceed 10% of the arithmetic mean.

中 华 人 民 共 和 国
国 家 标 准
白酒分析方法
GB/T 10345—2022

*

中国标准出版社出版发行
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)

网址 www.spc.net.cn
总编室:(010)68533533 发行中心:(010)51780238
读者服务部:(010)68523946
中国标准出版社秦皇岛印刷厂印刷
各地新华书店经销

*

开本 880×1230 1/16 印张 1.5 字数 46 千字
2022年7月第一版 2022年7月第一次印刷

*

书号:155066·1-70864 定价 30.00 元

GB/T 10345-2022

码上扫一扫 正版服务到