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GB/T 14454.14-2008

Replacing GB/T 14454.14-1993

Fragrance/Flavor substances - Preparation of standard solution, test solution and indicator solution

香料 标准溶液、试液和指示液的制备

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Foreword

GB/T 14454, Fragrance/Flavor substances, consists of the following parts:

-- Part 1: Fragrance/Flavor substances - Preparation of test samples;

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-- Part 4: Fragrance/Flavor substances - Determination of refractive index;

-- Part 5: Fragrance/Flavor substances - Determination of optical rotation;

-- Part 6: Fragrance/Flavor substances - Quantitative evaluation of residue on evaporation;

-- Part 7: Fragrance/Flavor substances - Determination of freezing point;

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-- Part 13: Fragrance/Flavor substances - Determination of carbonyl value and carbonyl compounds content;

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-- Part 15: Oil of sassafras - Determination of safrole and isosafrole content - Gas chromatographic method on packed columns.

This Part is Part 14 of GB/T 14454.

This Part is a revision of GB/T 14454.14-1993 "Aromatics - Preparation of standard solution, test solution and indicator solution". The technical content is the same as GB/T 14454.14-1993; the compiling method is in accordance with GB/T 1.1-2000.

This Part was proposed by China National Light Industry Council.

This Part shall be under the jurisdiction of National Technical Committee 257 on Fragrance and Flavor Cosmetic of Standardization Administration of China.

This Part is drafted by Shanghai Research Institute of Fragrance & Flavor Industry.

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Fragrance/Flavor substances - Preparation of standard solution, test solution and indicator solution

1 Scope

This Part of GB/T 14454 stipulates the preparation and calibration methods of standard solution, test solution and indicator solution for fragrance titration analysis.

This Part applies to the preparation of solutions of accurate concentration, as well as the preparation of test solution and indicator solution.

2 General provisions

2.1 The water that is used in this Part of GB/T 14454 shall be distilled water or water of equivalent purity unless other requirements are indicated.

2.2 The purity of the reagents that are used in this Part is at least analytically pure.

2.3 Weights of the analytical balance, burettes, volumetric flasks, and pipettes shall be regularly calibrated.

2.4 When calibrating the concentration of the standard solution, perform the parallel test for not less than eight times; two persons shall do four parallels. The extreme difference between the four parallel determination results per person, and the ratio of the difference between the averages of the determination results of the two persons to the average value cannot be greater than 0.1% for the standard solution of 0.5 mol/L concentration, and cannot be greater than 0.5% for the standard solution of 0.1 mol/L concentration (that is, the difference of the fourth significant digit cannot be greater than 5).

2.5 The relative error between the concentration of the prepared standard solution and the specified concentration cannot be greater than 5%.

2.6 The standard solution for titration analysis shall be calibrated and used at normal temperature of 15 °C ~ 25 °C; the storage time cannot exceed three months in general.

Where:

c(NaOH) -- the concentration of sodium hydroxide standard solution, in moles per liter (mol/L);

m -- the mass of potassium hydrogen phthalate, in grams (g);

V_1 -- the volume of sodium hydroxide standard solution for titration, in milliliters (mL);

V_2 -- the volume of sodium hydroxide standard solution for blank test, in milliliters (mL);

0.2042 -- the mass of potassium hydrogen phthalate, in grams, that is equivalent to 1.00 mL of sodium hydroxide standard solution.

3.2 Hydrochloric acid standard solution

c(HCl) = 0.5 mol/L, c(HCl) = 0.1 mol/L.

3.2.1 Preparation

Measure hydrochloric acid (relative density of 1.19) of the volume that is specified in Table 3; inject it into 1 000 mL of water; shake well.

Table 3

c(HCl)/(mol/L)	Hydrochloric acid/ mL
0.5	45
0.1	9

3.2.2 Calibration

3.2.2.1 Determination method

Weigh standard anhydrous sodium carbonate, of the amount that is specified in Table 4, that is burned to a constant mass at 270 °C ~ 300 °C; weigh to 0.000 2 g; place it in a 250 mL conical flask; use 50 mL of distilled water to dissolve; add 10 drops of bromocresol green-methyl red mixed indicator solution; use the prepared hydrochloric acid standard solution to titrate until the solution changes from green to dark red; boil for 2 minutes; after cooling, continue to titrate until the solution is dark red again. Do a blank test at the same time.

Table 4

c(HCl)/(mol/L)	Standard anhydrous sodium carbonate/g
0.5	0.8
0.1	0.2

solution; use the prepared potassium hydroxide ethanol solution to titrate until the solution is pink.

3.4.2.2 Calculation

The concentration of potassium hydroxide ethanol standard solution is calculated according to Formula (4):

$$c(KOH \cdot C_2H_5OH) = \frac{V_1 \times c_1}{V} \qquad \cdots\cdots(4)$$

Where:

$c(KOH \cdot C_2H_5OH)$ -- the concentration of potassium hydroxide ethanol standard solution, in moles per liter (mol/L);

V_1 -- the volume of hydrochloric acid standard solution, in milliliters (mL);

c_1 -- the concentration of hydrochloric acid standard solution, in moles per liter (mol/L);

V -- the volume of potassium hydroxide ethanol standard solution for titration, in milliliters (mL).

4 Preparation of test solution

4.1 Refined ethanol (95%)

Add 1 g of silver nitrate ($AgNO_3$) to every 1 000 mL of 95% ethanol for industrial use; dissolve in 5 mL of water; mix thoroughly. Dissolve another 5 g of potassium hydroxide in 25 mL of warm ethanol; after cooling, slowly add to the above solution; reflux for 4 h; use it after distillation.

4.2 Neutral analytically pure ethanol or neutral refined ethanol (95%)

Take an appropriate amount of analytically pure ethanol or refined ethanol; heat it; add a few drops of phenolphthalein indicator solution; use 0.1 mol/L sodium hydroxide solution to neutralize while it is hot.

4.3 Potassium hydroxide ethanol solution (0.5 mol/L)

Dissolve 30 g of potassium hydroxide in 1 000 mL of 95% ethanol; let it stand to clarify; carefully pour the upper transparent solution; or filter and store in a glass bottle. The solution cannot be used if it appears yellowish.

4.4 Hydroxylamine solution

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