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GB/T 14455.6-2008

Replacing GB/T 14455.6-1993, GB/T 14457.5-1993

Fragrance/Flavor substances Determination of ester value or ester content

香料 酯值或含酯量的测定

(ISO 709:2001, Essential oils - Determination of ester value, MOD)

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Standardization Administration of the People's Republic of China.

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Foreword

GB/T 14455, Fragrance/Flavor substances, consists of the following parts:

- -- Part 1: Essential oils Principles of nomenclature;
- -- Part 3: Fragrance/Flavor substances Evaluation of solubility (miscibility) in ethanol
- -- Part 5: Fragrance/Flavor substances Determination of acid value or acid content:
- -- Part 6: Fragrance/Flavor substances Determination of ester value or ester content;
- -- Part 7: Fragrance/Flavor substances Determination of ester value after acetylation and evaluation of free alcohols and total alcohols content.

This Part is Part 6 of GB/T 14455.

This Part modifies and adopts ISO 709:2001, "Essential oils - Determination of ester value". Compared with ISO 709:2001, the main technical differences are as follows:

- -- Add the determination method and calculation formula of the ester content of isolate and synthetic fragrance;
- -- Delete the sampling method and appendix A of ISO 709: 2001.

This Part is the combination and revision of GB/T 14455.6-1993, "Essential oils - Determination of ester value" and GB/T 14457.5-1993 "Isolate and synthetic aroma chemicals - Determination of ester content". Compared with GB/T 14455.6-1993 and GB/T 14457.5-1993, the main changes in this Part are as follows:

- -- Add the use of potentiometer to judge the end point;
- -- Add sample preparation;
- -- Add relevant contents of the test report.

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.

Fragrance/Flavor substances -

Determination of ester value or ester content

1 Scope

This Part of GB/T 14455 specifies the method for determining ester value or ester content of fragrance/flavor substances.

This Part applies to the determination of ester content of single esters AND ester value of essential oils in isolate and synthetic fragrance.

This Part does not apply to the determination of the ester content of mixed esters and esters that are difficult to be saponified, and the ester value of essential oils that contain lactones or more aldehydes.

2 Normative references

The terms in the following documents become the terms of this Part by reference to this Part of GB/T 14455. For dated references, all subsequent amendments (not including errata content) or revisions do not apply to this Part. However, parties to agreements that are based on this Part are encouraged to study whether the latest versions of these documents can be used. For undated references, the latest edition applies to this Part.

GB/T 14454.1, Fragrance/Flavor substances - Preparation of test samples (GB/T 14454.1-2008, ISO 356:1996, MOD)

GB/T 14454.14, Fragrance/Flavor substances - Preparation of standard solution, test solution and indicator solution

GB/T 14455.5, Fragrance/Flavor substances - Determination of acid value or acid content (GB/T 14455.5-2008, ISO 1242:1999, MOD)

3 Terms and definitions

The following terms and definitions are applicable to this Part of GB/T 14455.

3.1 Ester value

- **6.4** Pipette, whose capacity is 25 mL.
- 6.5 Boiling water bath.
- **6.6** Analytical balance.
- **6.7** Potentiometer.

7 Sample preparation

In accordance with the provisions of GB/T 14454.1.

8 Operating procedures

8.1 Sample

Weigh about 2 g of essential oil or an appropriate amount of isolate and synthetic fragrance (accurate to 0.0002 g).

If the sample size is different from the above, it is specified in the relevant fragrance product standard.

8.2 Blank test

Use the same reagent as sample measurement (8.3) at the same time under the same conditions: see 8.3.3.

8.3 Determination

8.3.1 Put the sample (8.1) into the saponification bottle (6.1). Use a pipette (6.4) to add 25 mL of potassium hydroxide ethanol solution (5.2) and some pumice or porcelain pieces.

For fragrance of a high ester value, increase the amount of potassium hydroxide ethanol solution (5.2), so that V_0 - V_1 (see Chapter 9) is at least 10 mL.

For fragrance of a low ester value, increase the sample volume.

Connect the air condenser; place the saponification bottle in the boiling water bath (6.5), to reflux for 1h (or reflux at the time that is specified in the relevant fragrance product standard).

Cool down; remove the air condenser. Add 20 mL of water and 5 drops of phenolphthalein indicator solution or phenol red indicator solution (5.4) (if the fragrance contains components with phenol groups).

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$$w = \frac{M_{\rm r} \times {\rm EV}}{561} \qquad \qquad (2)$$

Where:

M_r -- the relative molecular mass that is usually used to indicate the specified ester:

EV -- the ester value that is calculated by Formula (1).

When the ester value is less than 100, retain two significant digits; when the ester value is equal to or greater than 100, retain three significant digits.

9.1.2 Ester value after determination of acid value

When the sample is determined in a solution that has been determined for the acid value, calculate the ester value EV according to Formula (3):

$$EV = \frac{56.1 \times c \times (V_0 - V_1')}{m} \qquad \dots (3)$$

Where:

V'₁ -- the volume of hydrochloric acid standard solution (5.3) that is consumed in the new determination process, in milliliters (mL).

9.2 Ester content

9.2.1 Calculate ester content E according to Formula (4); express in %:

$$E = \frac{(V_0 - V_1') \times c \times M_r}{10m}$$

Retain the result to one decimal place.

9.2.2 Allowable difference of parallel test results: ester value below 10 is 0.2; ester content below 10% is 0.2%; ester value at $10 \sim 100$ is 0.5; ester content above 10% is 0.5%; ester value above 100 is 1.0.

10 Test report

The test report shall include:

- -- the used test method;
- -- the obtained test result;
- -- the finally-obtained result, if repeatability has been verified.

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