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

GB/T 14454.13-2008

**Fragrance/Flavor substances -- Determination of carbonyl value
and carbonyl compounds content
(English Translation)**

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1 Scope

Method I of this Part of GB/T 14454 specifies the method for determination of the carbonyl value of essential oils and the carbonyl compounds content in isolated and synthetic fragrance/flavor substances using hydroxylammonium chloride.

A cold oximation reaction with hydroxylammonium chloride of this method applies to fragrance/flavor substances whose main constituents are easily oximable aldehydes and ketones, with the exception of citronellal which needs a low temperature to avoid cyclization phenomena and acetalization. A hot oximation reaction with hydroxylammonium chloride applies to fragrance/flavor substances whose main constituents are ketones which are in general oximable only with difficulty.

Note 1: For the determination of essential oil containing citronellal, use free hydroxylamine method. See Method II.

Note 2: Examples of essential oils concerned containing oximable aldehydes or ketones are lemongrass, hesperidus and rue oils.

Note 3: Examples of essential oils concerned are vetiver, Dalmation sage and white artemisia oils which contain methylketones oximable only with difficulty.

Method II of this Part of GB/T 14454 specifies the method for determination of the carbonyl value of essential oils and the carbonyl compounds content in isolated and synthetic fragrance/flavor substances using free hydroxylamine method.

This method applies to fragrance/flavor substances which contain carbonyl compounds (especially ketones, excluding methylketones) which are difficult to convert to oximes by the method specified in Method I. The method is not applicable to fragrance/flavor substances which contain substantial amounts of esters or other alkali-sensitive constituents.

Method III of this Part of GB/T 14454 specifies the method for determination of the carbonyl compounds content in fragrance/flavor substances using the neutral sodium sulfite method. It is applicable to the determination of the content of carbonyl compounds in aldehyde and certain ketone fragrance/flavor

Conversion of the carbonyl compounds to oximes by reaction with hydroxylammonium chloride.

USE sodium hydroxide standard solution to titrate the hydrochloric acid released by this reaction. [Translator note: "potassium hydroxide standard solution"?]

4.2 Reagents

The reagents used are all analytically pure reagents. The water is distilled water or water of equivalent purity. Unless otherwise specified, the preparation method shall be carried out in accordance with GB/T 14454.14.

4.2.1

Sodium hydroxide standard solution, $c(\text{KOH})=0.5 \text{ mol/L}$.

4.2.2

Bromophenol blue indicator solution.

4.2.3

Hydroxylammonium chloride solution.

4.3 Apparatus

Usual laboratory equipment and, in particular, the following.

4.3.1

Beaker, of capacity 100 mL, tall form.

4.3.2

Conical flask, of capacity 250 mL.

4.3.3

pH meter.

4.3.4

Glass electrode.

4.3.5

Burette, with a capacity of 50 mL and a scale of 0.1 mL.

4.4.1 Preparation of test sample

According to the requirements of GB/T 14454.1.

4.4.2 Test portion

WEIGH 1 g~1.5 g of fragrance/flavor substance in a tall-form beaker (4.3.1) or conical flask (4.3.2), accurate to 0.0002 g.

Note: If the test sample should be larger, this will be stated in the appropriate product standard for the fragrance/flavor substance concerned.

4.4.3 Determination

The meanings of V, c, and m are the same as formula (1).

The tolerance of the parallel test results:

1.0 for the carbonyl value; 0.5% for the carbonyl compounds content.

5 Method of hot oximation with hydroxylammonium

chloride

5.1 Principle

Conversion of the carbonyl compounds to oximes by reaction with hydroxylammonium chloride.

USE sodium hydroxide solution to titrate the hydrochloric acid released by this reaction.

[Translator note: "potassium hydroxide standard solution"?]

5.2 Reagents

Same as 4.2.

5.3 Apparatus

Usual laboratory equipment and, in particular, the following.

5.3.1

Burette, with a capacity of 50 mL and a scale of 0.1 mL.

5.3.2

Beaker, of capacity 100 mL, tall form.

5.3.3 Saponification bottle, made of alkali-resistant glass, with frosted bottle

mouth. The capacity is 100 mL~250 mL. It is fitted with a frosted-mouth glass air condenser with a length of at least 1 m and an inner diameter of 1 cm~1.5 cm.

5.3.4 pH meter.

5.3.5 Glass electrode.

5.3.6 Heater with magnetic stirrer.

5.4.1 Preparation of test sample

According to the requirements of GB/T 14454.1.

5.4.2 Test portion

WEIGH 2 g~2.5 g of fragrance/flavor substance into the saponification bottle (5.3.3), accurate to 0.0002 g. See the note to 4.4.2.

5.4.3 Determination

ADD 50 mL of hydroxylammonium chloride solution (4.2.3) to the test portion (5.4.2). ADD 3 drops of bromophenol blue indicator solution (4.2.2) and mix thoroughly. USE sodium hydroxide standard solution (4.2.1) to titrate to the same yellow-green color as hydroxylammonium chloride solution (4.2.3).

Connect the saponification bottle to the reflux tube. PUT the saponification bottle on the heater (5.3.6) and heat with stirring, to make the temperature appropriate enough to maintain a constant reflux. After 10 min, cool, add 3 drops of bromophenol blue indicator solution (4.2.2); USE sodium hydroxide standard solution (4.2.1) to titrate slowly to the same yellow-green color as hydroxylammonium chloride solution (4.2.3). PUT the saponification bottle on the heater. REPEAT this operation every 10 min, until the sodium