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

GB/T 14454.7-2008

**Fragrance/Flavor substances -- Determination of freezing point
(English Translation)**

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1 Scope

This Part of GB/T 14454 specifies the methods for determining the freezing point of fragrance/flavor substances, determining the content of safrole in fragrance/flavor substances by freezing point method, and determining the content of cineol in fragrance/flavor substances by o-Cresol freezing point method.

This Part is applicable to the determination of the freezing point of fragrance/flavor substances; as well as to the determination of the content of safrole in essential oils and isolate fragrance/flavor substances with safrole content above 69%, and the determination of the content of cineol in essential oils and isolate fragrance/flavor substances with cineol content above 45.6%.

2 Normative references

The following documents contain provisions which, through reference in this

Part of GB/T 14454, constitute provisions of this Part. For the dated references, their subsequent amendments (excluding corrections) or revisions do not apply to this Part. However, the parties who enter into agreement based on this Part are encouraged to investigate whether the latest editions of these documents are applicable. For undated reference documents, the latest editions apply to this Part.

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

3 Terms and definitions

The following terms and definitions apply to this Part of GB/T 14454.

3.1

Freezing point

The constant temperature or the highest temperature observed when the fragrance/flavor substance is transformed from a liquid state to a solid state under supercooling to release its latent heat of fusion.

4 Principle

COOL the sample slowly and gradually. When the sample changes from liquid to solid, observe the temperature change.

5 Reagents (for the determination of cineol content

only)

The reagents used are all analytically pure reagents. The water is distilled water or water of equivalent purity.

5.1

O-Cresol. The freezing point is not less than 30 °C. When used, it needs to be distilled. TAKE the fraction between 190 °C~192 °C; PLACE in a dry bottle with glass stopper for use. Before use, it must be mixed with pure cineol (100%) according to the relative molecular mass ratio of 108.13 : 154.24 (o-Cresol :

cineol), to check its freezing point, which shall be 55.2 °C.

5.2

Anhydrous sodium sulfate.

5.3

Cineol (100%), which is dried using anhydrous sodium sulfate and set aside.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1

Calibrated thermometer. It shall meet the following requirements:

Length of mercury bulb: 10 mm~20 mm;

Diameter of mercury bulb: 5 mm~6 mm;

Scale: 0.1 °C.

This set of thermometer shall allow any temperature between -20 °C~+60 °C to be measured.

Specially required thermometers will be indicated in the product standards for the fragrance/flavor substances concerned.

6.2

Test tube. The diameter is about 20 mm. The length is about 100 mm.

6.3

Thick-walled test tube. The diameter is about 30 mm. The length is about 125 mm.

7.1.1 Preparation of test sample

According to the requirements of GB/T 14454.1.

7.1.2 Preliminary test

If necessary, first melt the sample warmly. COOL a few milliliters of the sample to be tested in the test tube (6.2); USE a stirrer to stir until it freezes.

Write down the temperature at this time; KEEP in a cold place.

7.1.3 Determination of freezing point

In order to achieve a temperature requirement that is 5 °C lower than the temperature observed in the preliminary test, add water, melting ice, or any suitable mixed refrigerant to the wide-mouth bottle (see 6.4). Install the thick-walled test tube (6.3).

ADD 10 mL of sample (melt if necessary) to the test tube (6.2). INSERT the thermometer and carefully cool the sample to the temperature obtained in the preliminary test. Immediately insert the test tube (6.2) into the thick-walled test tube (6.3), to reduce the temperature by another 2 °C.

If necessary, a small amount of solid sample, obtained from the preliminary test, may be added to the sample as a seed crystal. USE a stirrer to continuously stir, to cause the sample to crystallize. When the temperature rises, stop stirring.

TAKE care to prevent the precipitated particles from adhering to the tube wall.

Carefully observe the temperature changes.

When the temperature on the temperature-time curve shows the maximum value, or

remains unchanged at least 1 min, note the observed temperature.

REMOVE the test tube (6.2) from the device; re-liquefy the sample. REPEAT the determination, until the difference between the two consecutive test results does not exceed 0.2 °C.

TAKE the average of the last two readings as the final value.

Note 1: When determining the freezing point of individual products, when the temperature starts to rise, the stirring shall still be continued and shall not be stopped. Otherwise, the freezing point will be low, such as benzyl benzoate, which will be pointed out in the product standard for the fragrance/flavor substance concerned.

REMOVE the test tube (6.2) and stir in hot water³). After the sample melts, insert the test tube (6.2) into the thick-walled test tube (6.3); PLACE the thick-walled test tube (6.3) in a wide-mouth bottle (see 6.4). The bottle contains water that is 5 °C~10 °C below the expected freezing point. When cooling to some microcrystals in the test tube (6.2), or when it has cooled to the expected freezing point, the stirring is accelerated, to rapidly solidify. RECORD the temperature that rises to the highest point.

REMOVE the test tube (6.2) from the device; re-liquefy the sample. REPEAT the determination, until the difference between the two consecutive test results does not exceed 0.1 °C.

7.3.2 Samples with cineol content below 50%

Respectively, accurately weigh 5 ± 0.001 g of the sample dried with anhydrous sodium sulfate and of cineol (5.3); and, mix in a conical flask (6.6). WEIGH 2.1 ± 0.001 g of o-Cresol (5.1) and 3 ± 0.001 g of the above mixed sample;

determine the freezing point according to 7.3.1.

8.1 Freezing point

The highest temperature observed at the end of the test is the freezing point, expressed in °C. It is accurate to 0.1 °C.

The tolerance of the parallel test results is 0.2 °C.

8.2 Safrole content

After the freezing point is measured, according to Table 1, it is converted into the content of safrole.

The tolerance of the parallel test results is 1%.