

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

## ISO RECOMMENDATION R 1041

DETERMINATION OF FREEZING POINT  
OF ESSENTIAL OILS

1st EDITION

April 1969

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Printed in Switzerland

Also issued in French and Russian. Copies to be obtained through the national standards organizations.

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## BRIEF HISTORY

The ISO Recommendation R 1041, *Determination of freezing point of essential oils*, was drawn up by Technical Committee ISO/TC 54, *Essential oils*, the Secretariat of which is held by the Repartição de Normalização (IGPAI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In August 1965, this Draft ISO Recommendation (No. 831) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Germany	Portugal
Australia	Greece	Romania
Austria	India	Spain
Belgium	Italy	Sweden
Canada	Morocco	United Kingdom
Chile	Netherlands	U.S.S.R.
France	New Zealand	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in April 1969, to accept it as an ISO RECOMMENDATION.

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## DETERMINATION OF FREEZING POINT OF ESSENTIAL OILS

### 1. SCOPE

This ISO Recommendation describes a method to be used in determining the freezing points of essential oils. It is not applicable to essential oil of rose.

### 2. DEFINITION

The *freezing point* of an essential oil is either the constant temperature, or the maximum temperature, observed when the oil, in a supercooled liquid state, liberates its latent heat of fusion.

### 3. PRINCIPLE OF THE METHOD

Slow and progressive cooling of the essential oil.

Observation of the variations in temperature as the oil passes from the liquid to the solid state.

### 4. APPARATUS

#### 4.1 Calibrated thermometers, fulfilling the following requirements :

- |                    |             |
|--------------------|-------------|
| — length of bulb   | 10 to 20 mm |
| — diameter of bulb | 5 to 6 mm   |
| — graduations      | 0.1 °C      |

It is essential that the set of thermometers used (see ISO Recommendation R 653, *Long solid-stem thermometers for precision use*), should permit the measurement of any temperature between - 20 and + 50 °C.

The relevant ISO Recommendations for the essential oils indicate which thermometer should be used.

#### 4.2 Test tube, about 20 mm in diameter and 100 mm long.

#### 4.3 Stout-walled test tube. About 30 mm in diameter and 125 mm long.

#### 4.4 Apparatus assembly for determination of freezing point (see the Figure on page 7, which is illustrative only). It consists of a wide-mouthed container of about 500 cm<sup>3</sup> capacity, provided with a bored cork or rubber stopper into which the stout-walled test tube (4.3) is inserted. The test tube (4.2) is fitted into the stout-walled test tube (4.3) by means of another bored cork or rubber stopper. Into the test tube (4.2) a thermometer (4.1) is inserted, so that the centre of its mercury bulb is located at the centre of the liquid.

## 5. SAMPLING

See ISO Recommendation R 212, *Essential oils – Sampling*.

## 6. PROCEDURE

### 6.1 Preparation of test sample

See ISO Recommendation R 356, *Essential oils – Methods of test – Preparation of sample*.

### 6.2 Preliminary test

If necessary, first liquefy the essential oil by warming. Cool a few cubic centimetres of the oil to be tested in a small test tube, and stir with a thermometer until solidification takes place.

Note the temperature and set aside in a cool place.

### 6.3 Determination

Fill the container (see clause 4.4) with water, melting ice or any suitable freezing mixture, so as to obtain a temperature of 5 °C lower than that noted in the preliminary test. Fit the stout-walled test tube (4.3) in its place.

Into the test tube (4.2), place 10 cm<sup>3</sup> of the essential oil, insert the thermometer and carefully cool the oil to the temperature indicated in the preliminary test. Now insert the test tube (4.2) in the stout-walled tube (4.3) of the apparatus and allow the temperature to fall a further 2 °C.

Seed the oil with a trace of the solidified oil, obtained in the preliminary test, and stir vigorously with the thermometer, taking care to avoid the adhesion of particles to the walls of the tube. Observe the temperature variations as carefully as possible.

Note the observed temperature when the temperature curve in relation to time shows a maximum value, or a constant level for at least 1 minute.

Repeat the determination until two consecutive results do not differ by more than 0.2 °C. Average the two last readings to obtain the final figure.

## 7. EXPRESSION OF RESULTS

The freezing point is the maximum temperature observed at the end of the test. It is expressed in degrees Celsius to one decimal place.

## 8. TEST REPORT

The test report should include the following information :

- (a) a reference to the method used;
- (b) the results obtained;
- (c) any operation not included in this ISO Recommendation or any operation regarded as optional which may have influenced the results;
- (d) all details necessary for the complete identification of the sample.