INTERNATIONAL STANDARD

**ISO 6321**

Third edition
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# Animal and vegetable fats and oils — Determination of melting point in open capillary tubes — Slip point

*Corps gras d'origines animale et végétale — Détermination du point de fusion en tube capillaire ouvert — Point de glissement*



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# Contents

Page

# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 34, *Food products,* Subcommittee SC 11, *Animal and vegetable fats and oils,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 307, *Oilseeds, vegetable and animal fats and oils and their by-products — Methods of sampling and analysis,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 6321:2002), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the requirement to measure the diameters of each capillary tube has been removed, and

— a footnote stating suggested suppliers of suitable capillary tubes has been included.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.



# Animal and vegetable fats and oils — Determination of melting point in open capillary tubes — Slip point

## 1 Scope

This document specifies two methods for the determination of the melting point in open capillary tubes, commonly known as the slip melting point, of animal and vegetable fats and oils (referred to as fats hereinafter).

— Method A is only applicable to animal and vegetable fats which are solid at ambient temperature and which do not exhibit pronounced polymorphism.

— Method B is applicable to all animal and vegetable fats which are solid at ambient temperature and is the method to be used for fats whose polymorphic behaviour is unknown.

For the determination of the slip melting point of palm oil samples the method given in Annex A shall be used.

NOTE 1 If applied to fats with pronounced polymorphism, method A will give different and less satisfactory results than method B.

NOTE 2 Fats which exhibit pronounced polymorphism are principally cocoa butter and fats containing appreciable quantities of 2-unsaturated, 1,3-saturated triacylglycerols.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

**3.1**
**slip melting point in open capillary tube**
temperature at which a column of fat in an open capillary tube commences to rise under the conditions specified in this document

## 4 Principle

A capillary tube containing a column of the fat which has been crystallized under controlled conditions is immersed to a specified depth in water, the temperature of which is increased at a specified rate. The temperature at which the column is observed to start rising in the capillary tube is recorded.

## 5 Apparatus

Usual laboratory apparatus and, in particular, the following.

**5.1 Capillary tubes**, having uniform walls and which are open at both ends, of internal diameter 0,9 mm to 1,2 mm, external diameter 1,2 mm to 1,6 mm, wall thickness 0,15 mm to 0,30 mm and length 50 mm to 70 mm[1)].

Before use, clean the tubes thoroughly by washing them successively with a mixture of chromic acid, water and acetone or an alternative suitable cleaning solution, for example, hydrogen peroxide can be used. Dry the capillary tubes in an oven. However, it is recommended that new tubes are used.

**5.2 Thermometer**, graduated in divisions of 0,1 °C, calibrated over the range of melting points expected.

**5.3 Stirrer**, electrical.

**5.4 Cooling bath**, filled with brine or other non-freezing liquid, thermostatically maintained at a temperature of −10 °C to −12 °C, or filled with a mixture of flaked ice and salt (in the proportions 2 to 1 by mass) at a temperature of −10 °C to −12 °C.

**5.5 Heating apparatus**, consisting of the following elements:

a) **water jacket**, made of glass, provided with inlet and outlet tubes, and having the shape and dimensions shown in Figure 1;

b) **water heater**, capable of delivering a slow stream of water, the temperature of which can be controlled to increase at a rate of between 0,5 °C/min and 4 °C/min, through the water jacket [5.5 a)].

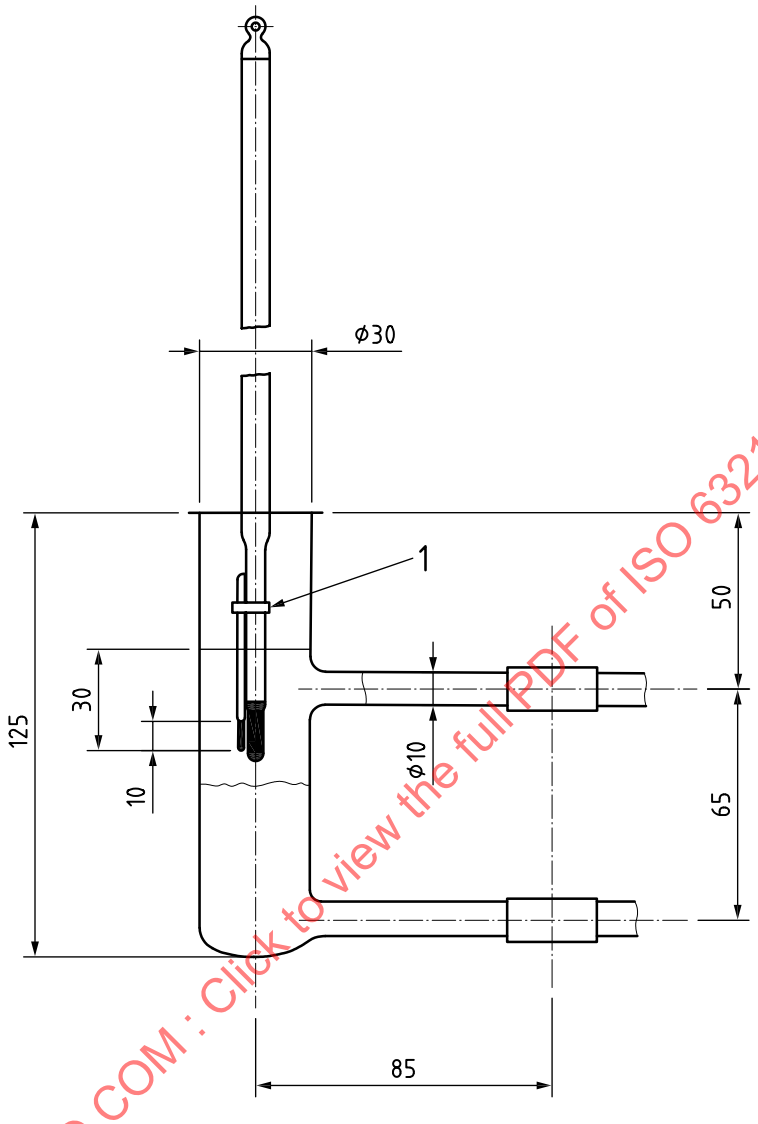
An example of a suitable heating apparatus is shown in Figure 2.

Other types of heating apparatus, such as a water bath with magnetic stirrer, capable of being controlled to produce the specified temperature rise [5.5 b)], may also be used.



1) Reference 1411022 from Hilgenberg (https://www.hilgenberg-gmbh.de/innovative-glasprodukte/), Reference 2930201 from Marienfeld (https://www.marienfeld-superior.com/home.html) and Reference 9201570 from Hirschmann (http://www.hirschmann-laborgeraete.de/) are examples of suitable products from providers of capillary tubes. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of products by these providers.

Dimensions in millimetres

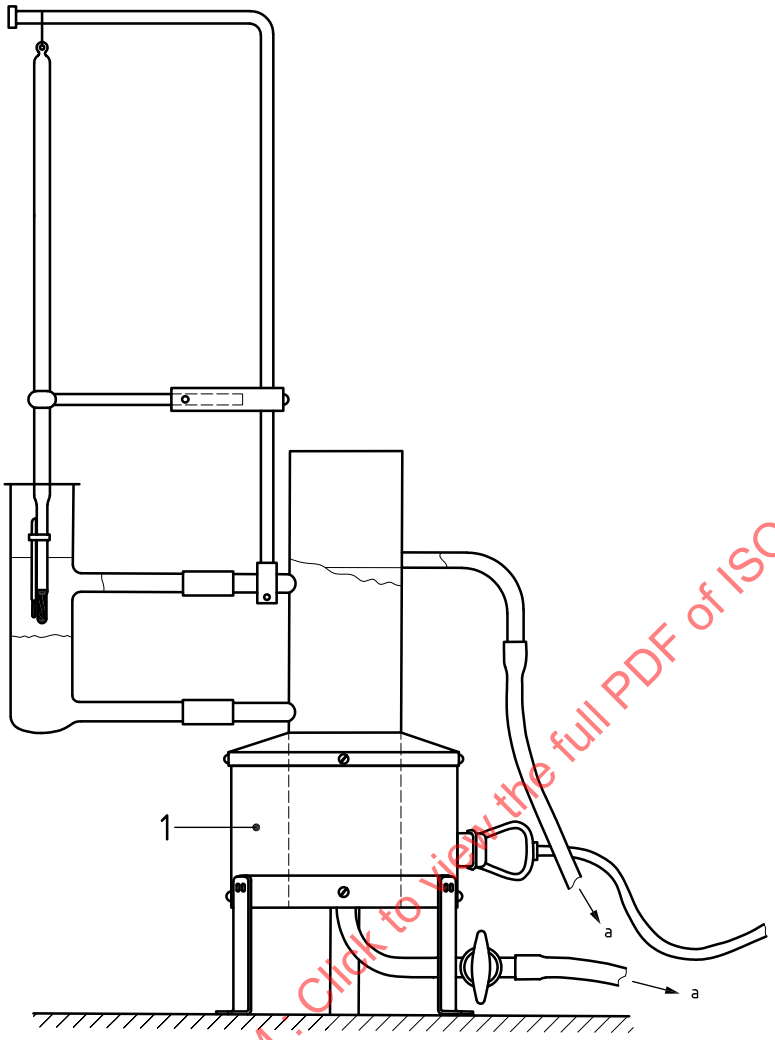



**Key**

1 rubber band

**Figure 1 — Water jacket**

Dimensions in millimetres




**Key**

1 heating element (coil 220 W)

a To drain.

**Figure 2 — Example of heating apparatus (heating by natural convection)**



## 6 Sampling

It is important the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 5555[1].

## 7 Preparation of test sample

Prepare the test sample in accordance with ISO 661.

# 8 Procedure

## 8.1 Preparation of the capillary tubes for method A

Melt a portion of the test sample as rapidly as possible to at least 5 °C, but not more than 10 °C, above the temperature at which it is completely melted.

Dip two capillary tubes (5.1) into the melted test sample until columns of fat 10 mm ± 2 mm long are obtained. Immediately after filling the tubes, wipe them quickly with absorbent tissue to remove any fat adhering to the outer surfaces of the tubes. Immediately place the filled capillary tubes for a few seconds against a beaker filled with ice so that the fat solidifies.

Place the tubes in the cooling bath (5.4) for 5 min.

Continue in accordance with 8.3.

## 8.2 Preparation of the capillary tubes for method B

Melt a portion of the test sample as rapidly as possible to at least 5 °C, but not more than 10 °C, above the temperature at which it is completely melted.

Cool the melted test sample, with occasional stirring, until its temperature is 32 °C to 34 °C and then stir continuously with the stirrer (5.3), allowing the fat to cool until the first signs of cloudiness appear.

Continue stirring by hand until the fat has a pasty consistency and then transfer the fat to a 100 ml beaker at 17 °C ± 2 °C.

Store the fat at this temperature for a minimum of 24 h.

Push four capillary tubes (5.1) into the conditioned fat until a column of fat 10 mm ± 2 mm long is obtained in each tube. Wipe the tubes quickly with absorbent tissue to remove any fat adhering to the outer surfaces of the tubes.

Store the tubes at 17 °C ± 2 °C until required.

## 8.3 Determination

**8.3.1** Avoiding transfer of body heat to the fat, attach two capillary tubes prepared for method A (8.1) or for method B (8.2) to the thermometer (5.2) using small rubber bands (or by any other suitable means such as a rubber ring) so that the columns of fat are located at the lower ends of the tubes and lie adjacent to the bulb of the thermometer.

**8.3.2** Fill the water jacket [5.5 a)] and the water heater [5.5 b)] with previously boiled water cooled to 15 °C. Clamp or suspend the thermometer with the attached capillary tubes centrally in the water jacket so that the lower ends of the capillary tubes are 30 mm below the surface of the water.

**8.3.3** Operate the heating apparatus (5.5) so that a slow stream of water passes through the water jacket, regulating the heating so that the rise in temperature of the water, as measured by the thermometer in the water jacket, is about 3 °C/min to 4 °C/min for method A and 1 °C/min for method B.

**8.3.4** For each of the two capillary tubes, note the temperature value indicated by the thermometer immediately as the fat starts to rise in the tube.

**8.3.5** Note the arithmetic mean of the two readings obtained. For method A, take this arithmetic mean as the result of one determination.

**8.3.6** For method B, repeat the operations described in 8.3.1 to 8.3.3 using the remaining two capillary tubes (8.2), decreasing the rate of temperature rise to 0,5 °C/min when the water temperature is within 5 °C of the mean reading determined in 8.3.5. For each of the two capillary tubes, note the temperature value indicated by the thermometer immediately the fat starts to rise in the tube. Record the arithmetic mean of the two readings obtained and take this as the result of one determination.

### 8.4 Number of determinations

Carry out two determinations on the same test sample [i.e. to obtain two mean readings for method A (8.3.5) and two final mean readings for method B (8.3.6)].

## 9 Expression of results

Take as the result the arithmetic mean of the two determinations.

Express the slip melting point in open capillary tube to the nearest 0,1 °C.

## 10 Precision

### 10.1 Interlaboratory tests

Details of interlaboratory tests on the precision of the method are summarized in Annex B. The values derived from these interlaboratory tests may not be applicable to concentration ranges and matrices other than those given.

### 10.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,5 °C for method A and 1,0 °C for method B.

## 11 Test report

The test report shall specify:

a) the sample;

b) this document including year of publication (e.g. ISO 6321:2021);

c) all information necessary for the complete identification of the sample;

d) the sampling method used, if known;

e) the test method used (i.e. ISO 6321, method A or method B);

f) all operating details not specified in this document, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

g) the test result(s) obtained, or, if the repeatability has been checked, the final result obtained;

h) any deviations from the procedure;

i) any unusual features observed;

j) the date of the test.



# Annex A
(normative)

# Method for palm oil samples

Melt the sample and filter through a filter paper. Conduct the filtration in an oven set at 60 °C to avoid any crystallization of the sample. Leave the filtered sample in the oven for 10 min until it is free of air bubbles.

Dip at least three clean capillary tubes into the liquid sample so that columns of fat approximately 10 mm high are obtained in the tubes. Immediately chill the columns of fat by holding and rolling the ends of the tubes containing the sample pressed against a piece of ice, until the fat has solidified. Do not allow the open end of the tube to touch the ice. Wipe the tubes against a piece of tissue paper as quickly as possible. Place the tubes in a test tube which is held in a beaker of water that has been equilibrated at 10 °C ± 1 °C in a thermostated water bath. Transfer the beaker to the water bath and hold for 16 h at 10 °C ± 1 °C.

For the determination, follow the procedure as given in 8.3.1 to 8.3.3. Regulate the rise in temperature in the water jacket to 1 °C/min, slowing down to 0,5 °C/min as the slip point is reached. Note the temperature value indicated by the thermometer as soon as the fat rises in each of the tubes.

Note the arithmetic mean of the three readings obtained and take this as the result of one determination.

# Annex B
(informative)

# Results of interlaboratory tests

Two interlaboratory tests, carried out at the international level in 1982 and 1986 by ISO/TC 34/SC 11, in which 20 laboratories [each of which carried out three determinations on each sample (columns 2, 3 and 8)] and 15 laboratories [each of which carried out three determinations on each sample (columns 4 to 7)] participated, gave the statistical results (evaluated in accordance with ISO 5725:1986[2]) shown in Table B.1.

The results of interlaboratory tests on palm oil samples are given in Tables B.2 and B.3.

**Table B.1 — Statistical results**

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|---|
| | **Method A** | | **Method B** | | | | |
| | Palm kernel oil | Hydrogenated soyabean oil | Cocoa butter | Palm oil | Hydrogenated coconut oil | Hydrogenated palm oil | Hydrogenated palm oil |
| Number of laboratories retained after eliminating outliers | 18 | 18 | 14 | 14 | 13 | 13 | 18 |
| Mean (°C) | 27,6 | 35,4 | 31,4 | 36,3 | 37,1 | 45,5 | 47,5 |
| Standard deviation of repeatability, $s_r$ (°C) | 0,15 | 0,14 | 0,29 | 0,35 | 0,30 | 0,13 | 0,15 |
| Coefficient of variation of repeatability (%) | 0,5 | 0,4 | 0,9 | 1,0 | 0,8 | 0,3 | 0,3 |
| Repeatability limit, $r$ ($2{,}8\ s_r$)(°C) | 0,4 | 0,4 | 0,8 | 1,0 | 0,8 | 0,4 | 0,4 |
| Standard deviation of reproducibility, $s_R$ (°C) | 0,31 | 0,75 | 2,0 | 2,5 | 0,9 | 0,5 | 0,77 |
| Coefficient of variation of reproducibility (%) | 1,1 | 2,1 | 6,4 | 6,9 | 2,5 | 1,1 | 1,7 |
| Reproducibility limit, $R$ ($2{,}8\ s_R$) (°C) | 0,9 | 2,1 | 5,7 | 7,1 | 2,6 | 1,4 | 2,2 |



2) ISO 5725:1986 was used to obtain the precision data and has since been withdrawn.