INTERNATIONAL STANDARD

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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

Reference number
ISO 6321:1991(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 6321 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 11, *Animal and vegetable fats and oils*.

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Animal and vegetable fats and oils — Determination of melting point in open capillary tubes (slip point)

1 Scope

This International Standard specifies two methods for the determination of the melting point in open capillary tubes, commonly known as the slip 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.

NOTES

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

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 661:1989, *Animal and vegetable fats and oils — Preparation of test sample.*

ISO 5555:1991, *Animal and vegetable fats and oils — Sampling.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 melting point (in open capillary tubes); slip point: The temperature at which a column of fat in an open capillary tube commences to rise under the conditions specified in this International Standard.

4 Principle

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

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 1,0 mm to 1,2 mm, external diameter 1,3 mm to 1,6 mm, wall thickness 0,15 mm to 0,20 mm and length 50 mm to 60 mm.

Check the internal and external diameters of the capillary tubes using a test gauge such as that shown in figure 1.

Before use, clean the tubes thoroughly by washing them successively with a mixture of chromic acid,

water and acetone, and then dry them in an oven. It is recommended that new tubes be 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 2;

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 [a)].

An example of a suitable heating apparatus is shown in figure 3.

NOTE 3 Other types of heating apparatus, such as a water-bath with magnetic stirrer, capable of being controlled to produce the specified temperature rise may also be used.

6 Sampling

Sampling shall have been carried out in accordance with ISO 5555.

7 Preparation of the 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, e.g. 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.

Dimensions in millimetres

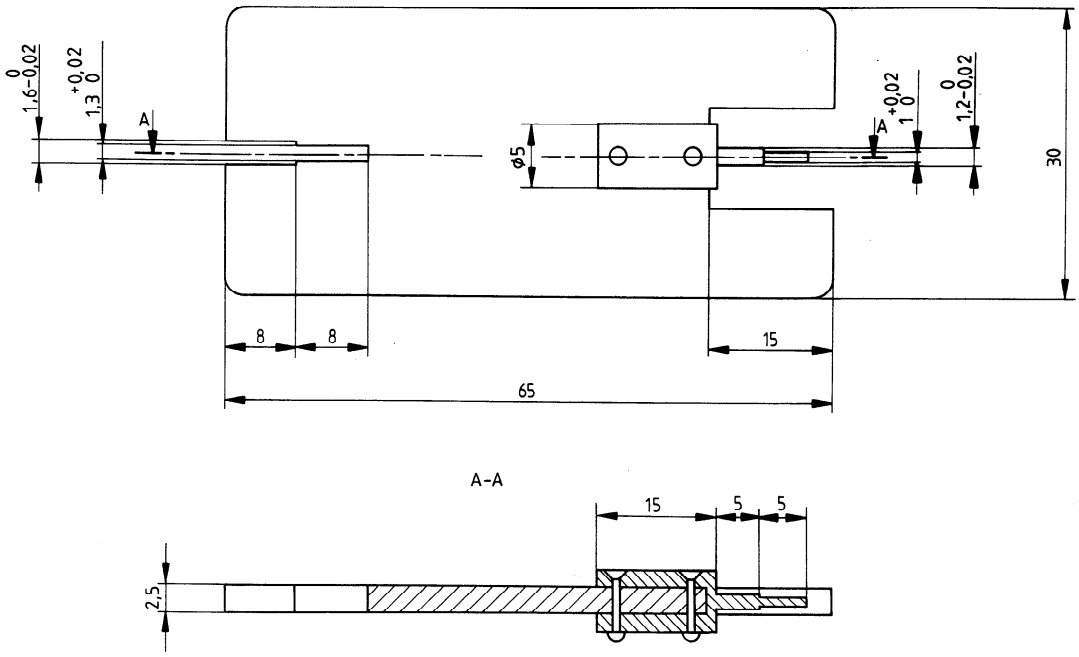

Figure 1 — Test gauge for capillary tubes

Dimensions in millimetres

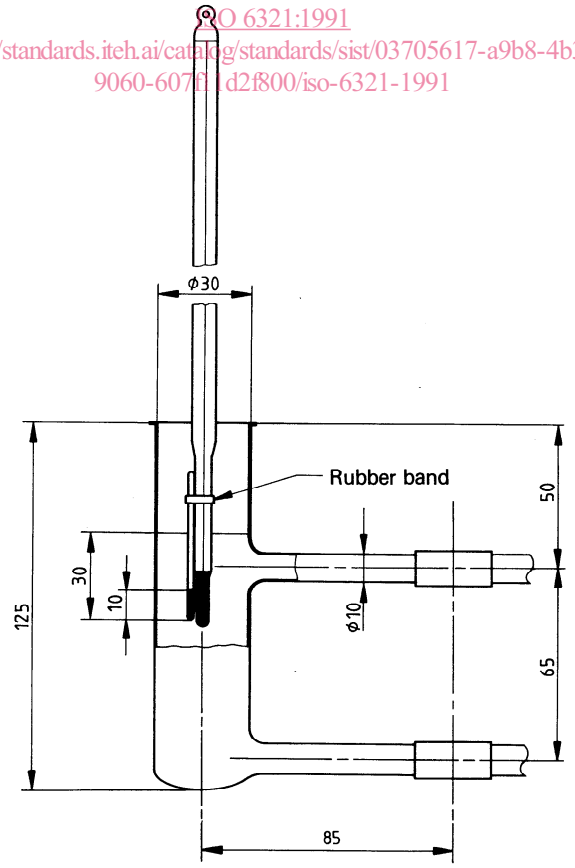

Figure 2 — Water jacket

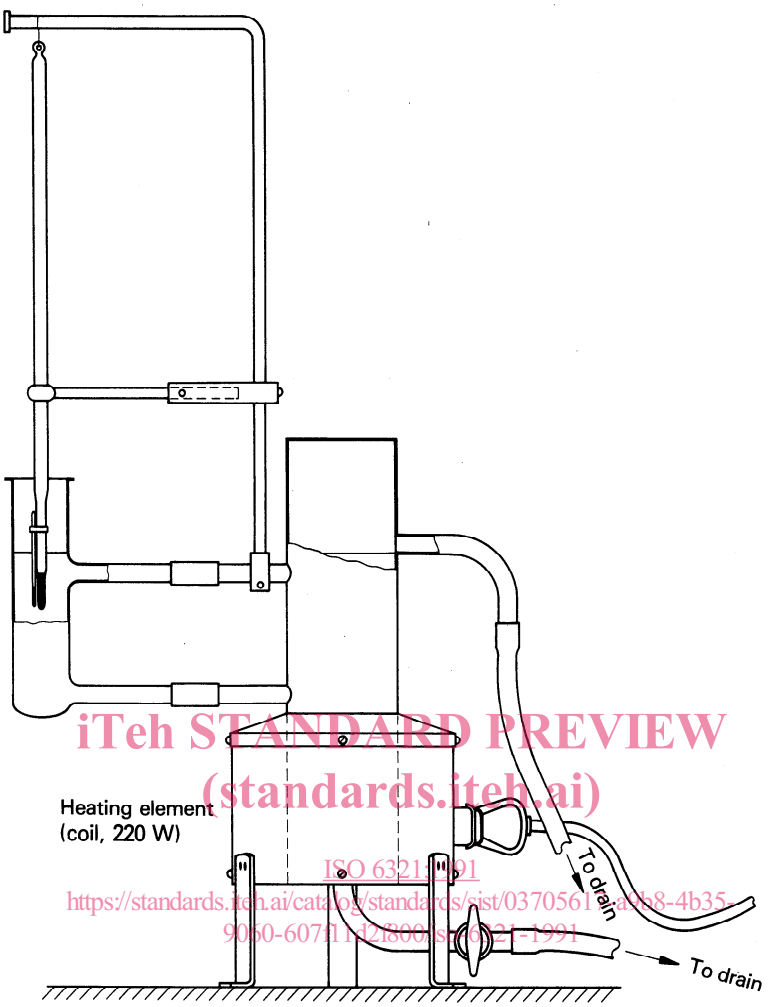

Figure 3 — Example of heating apparatus (heating by natural convection)

8.3.2 Fill the water jacket [5.5a)] and the water heater [5.5b)] 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 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 result to the nearest 0,1 °C.

10 Precision

10.1 Statistical results of inter-laboratory tests

Two inter-laboratory 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[1]) shown in table 1.

10.2 Repeatability

The difference between the values of two determinations, carried out in rapid succession (or simultaneously) by the same operator using the same apparatus on the same test sample, shall not exceed 0,5 °C for method A and 1,0 °C for method B.

11 Test report

The test report shall specify the method used (i.e. ISO 6321, method A or method B) and the result obtained. It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the result.

The test report shall include all information necessary for the complete identification of the sample.

Table 1

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, 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, 2,8 s_R (°C)	0,9	2,1	5,7	7,1	2,6	1,4	2,2

1) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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