EAST AFRICAN COMMUNITY

EDICT OF GOVERNMENT

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EAS 319 (2002) (English): Animal and vegetable fats and oils — Determination of melting point in open capillary tubes (slip point)

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EAST AFRICAN STANDARD

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

EAST AFRICAN COMMUNITY

First Edition 2002

Foreword

Development of the East African Standards has been necessitated by the need for harmonizing requirements governing quality of products and services in East Africa. It is envisaged that through harmonized standardization, trade barriers which are encountered when goods and services are exchanged within the Community will be removed.

In order to achieve this objective, the Partner States in the Community through their National Bureaux of Standards, have established an East African Standards Committee.

The Committee is composed of representatives of the National Standards Bodies in Partner States, together with the representatives from the private sectors and consumer organizations. Draft East African Standards are circulated to stakeholders through the National Standards Bodies in the Partner States. The comments received are discussed and incorporated before finalization of standards, in accordance with the procedures of the Community.

East African Standards are subject to review, to keep pace with technological advances. Users of the East African Standards are therefore expected to ensure that they always have the latest versions of the standards they are implementing.

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INTERNATIONAL STANDARD

ISO 6321

Second edition
2002-02-15

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
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 6321 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This second edition cancels and replaces the first edition (ISO 6321:1991), of which it constitutes a minor revision to incorporate Amendment 1:1998.

Annex A forms a normative part of this International Standard. Annex B is for information only.

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.

A method for the determination of the melting point of palm oil samples is given in annex A.

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 reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

3 Term and definition

For the purposes of this International Standard, the following term and definition apply.

3.1
melting point (in open capillary tubes)
slip point
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

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 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.

Dimensions in millimetres

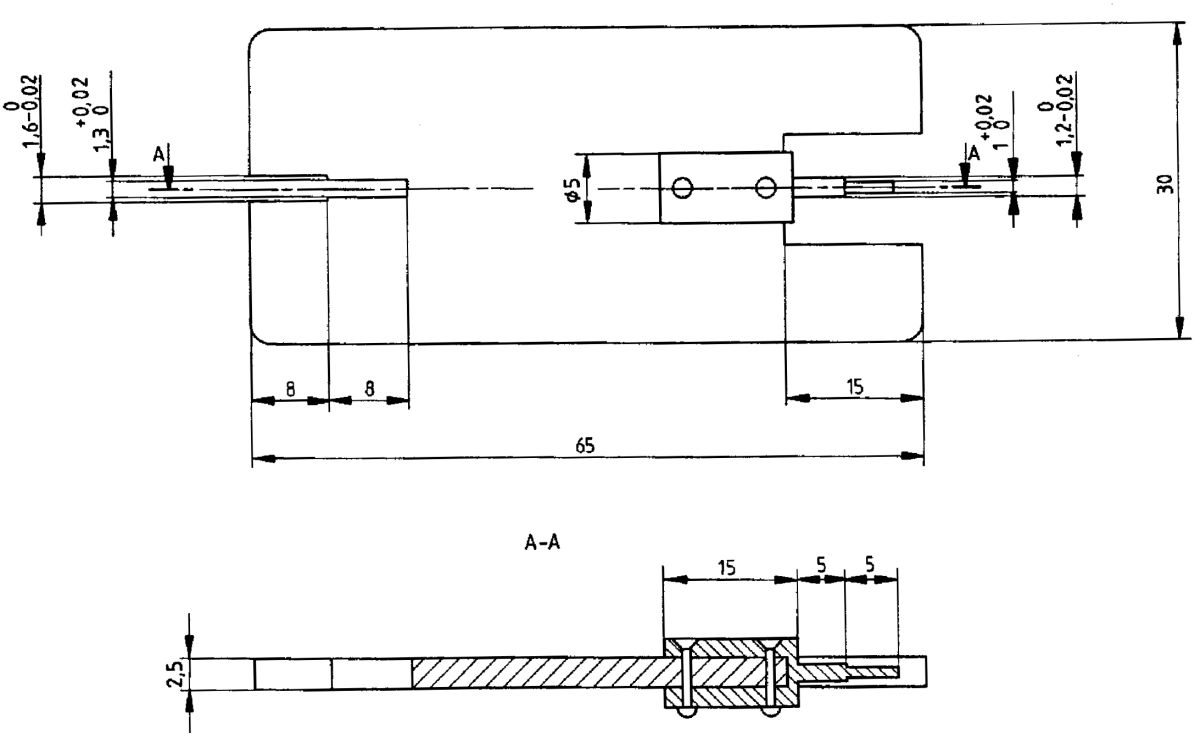


Figure 1 — Test gauge for capillary tubes

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:

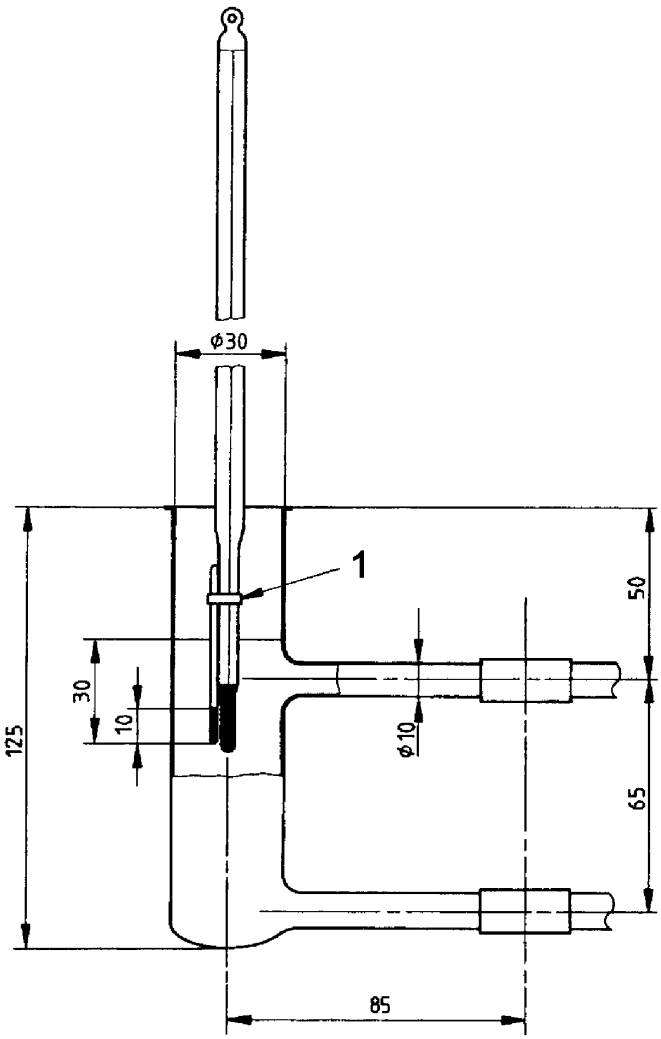
a) **water jacket**, made of glass, provided with inlet and outlet tubes, and having the shape and dimensions shown in Figure 2;

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.

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.

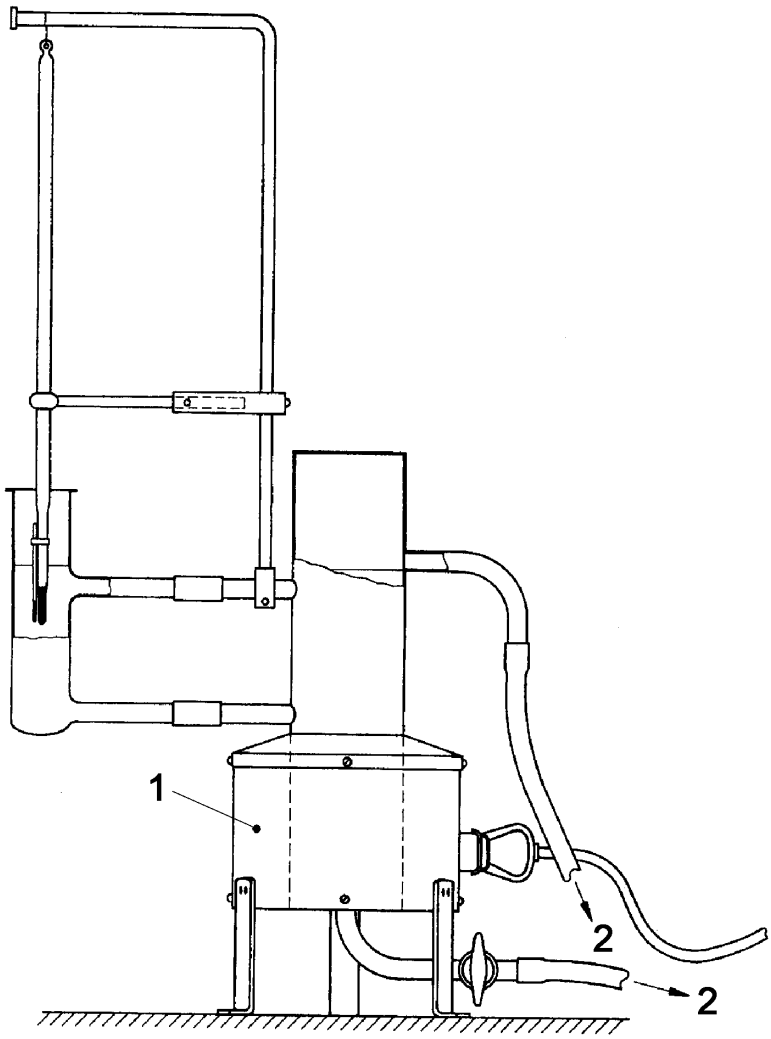
Dimensions in millimetres



Key

1 Rubber band

Figure 2 — Water jacket



Key

1 Heating element (coil 220 W)

2 To drain

Figure 3 — 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 International Standard. 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 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 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:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

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

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

— the test result(s) obtained, or, if the repeatability has been checked, the final result obtained.

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[1)] [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

1) ISO 5725:1986 (now withdrawn) was used to obtain the precision data.

Table B.2 — Comparison of methods using palm oil samples

Samples		Slip point		
		MS 817:1989	ISO 6321	
		AOCS CC 3-25[a]	Method A	Method B
Palm oil, RBD[b]	1	36,8	38,2	36,5
	2	35,3	37,4	35,5
	3	35,2	37,7	35,5
	4	36,6	38,0	36,5
	5	35,6	37,5	35,5
Palm olein, RBD	1	22,3	24,4	25,5
	2	22,2	24,4	25,5
	3	22,5	24,3	25,5
	4	22,5	24,2	24,9
	5	22,3	24,2	24,9
Palm stearin, RBD	1	51,6	51,8	51,5
	2	52,8	51,8	52,8
	3	46,0	44,8	45,0
	4	52,3	52,8	53,4
	5	51,0	51,0	51,5
Crude palm oil	1	35,8	35,6	
	2	35,3	36,6	
	3	35,8	36,4	
	4	35,0	35,8	
	5	35,8	36,8	
Crude palm kernel oil	1	27,8	27,7	27,6
	2	26,6	27,8	27,6
	3	26,7	26,7	27,0
	4	26,8	26,7	27,0
	5	27,0	27,5	27,4
Palm kernel oil, RBD	1	27,8	27,8	28,2
	2	27,8	27,6	27,6
	3	27,7	27,5	28,0
	4	27,8	27,2	28,0
	5	27,6	27,3	27,8
Palm kernel olein, RBD	1	26,2	25,8	26,0
	2	23,4	23,3	23,8
	3	23,5	23,4	23,8
	4	23,4	23,4	23,8
	5	24,6	24,4	24,5
Palm kernel stearin, RBD	1	32,2	32,2	33,0
	2	32,2	32,8	33,0
	3	39,3	38,5	39,4
	4	33,3	33,0	33,2
	5	32,3	33,6	33,2

[a] Malaysian Standard MS 817:1989 [5]. AOCS Official Method CC 3-25, *Slip melting point AOCS standard open tube melting point* (updated 1992).

[b] RBD: refined, bleached, deodorized.

Table B.3 — Statistical results for palm oil samples

	Palm oil	Palm olein	Palm stearin
No. of laboratories retained after eliminating outliers	10	11	11
Mean (°C)	37,4	20,5	52,1
Standard deviation of repeatability, s_r (°C)	0,23	0,15	0,09
Coefficient of variation of repeatability (%)	0,6	0,7	0,2
Repeatability limit, r (2,8 s_r) (°C)	0,64	0,42	0,25
Standard deviation of reproducibility, s_R (°C)	0,78	0,98	0,54
Coefficient of variation of reproducibility (%)	2,1	4,8	1,0
Reproducibility limit, R (2,8 s_R) (°C)	2,2	2,7	1,5

Bibliography

[1] ISO 5555, *Animal and vegetable fats and oils — Sampling*

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

[3] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[4] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

[5] MS 817:1989, *The determination of melting point in open capillary tubes (slip point) for palm oil products*

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