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Animal and vegetable fats and oils — Determination of dilatation

Corps gras d'origines animale et végétale — Détermination de la dilatation

Reference number
ISO 8293:1990(E)

Foreword

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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 8293 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

Annex A of this International Standard is for information only.

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Introduction

Dilatation is an increase in volume. The change in volume between liquid and solid phases is a measure of the solid to liquid ratio. The measurement of dilatation has been used for many years in the fat-processing industry as an indication of the solid fats content at temperatures at which both solid and liquid fats are present. The dilatation value cannot be related to the solid fats content determined using nuclear magnetic resonance.

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Animal and vegetable fats and oils — Determination of dilatation

1 Scope

This International Standard specifies a method for the determination of the dilatation of fats.

It is applicable to animal and vegetable fats and oils (referred to as fats hereinafter), except palm stearin, having a melting point above 0 °C and not more than 60 °C. The method is empirical and therefore any variation in the prescribed operational details will modify the results obtained.

A special procedure is given for the stabilization of cocoa butter and other polymorphic fats.

NOTE 1 Fats which consist principally of 2-unsaturated 1,3-saturated triglycerides display a characteristic polymorphism and need to be stabilized in a particular way before the dilatations are measured. Nevertheless, for cocoa butter substitutes based on lauric acid derivatives, whether hydrogenated or not, the method for non-polymorphic fats (9.5.1) may be applied. It should then be stated in the test report whether the fat has been subjected to a pre-treatment (as in 9.5.2) or not.

Fats very rich in glycerol 2-oleate, 1,3-distearate, such as shea fat fractions, kokum butter and the allanblackia group of fats, sometimes behave unsatisfactorily if occluded air is retained; it is then advisable to heat the dilatometer before filling it.

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:—[1], *Animal and vegetable fats and oils — Sampling.*

3 Definition

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

dilatation of a fat: The isothermal expansion, due to a change in state from solid to liquid, of a fat which has been previously solidified under precisely prescribed conditions.

The dilatation of a fat is expressed in millilitres per kilogram of fat (or microlitres per gram of fat) and represents the difference between the volume the fat would occupy at the temperature in question if it were completely liquid (supercooled) and the actual volume of the fat at this temperature. It affords an empirical indication of the solid fats content at this temperature.

NOTE 2 Care should be taken to avoid confusion with the expression of dilatation in microlitres per 25 g of fat which is sometimes used.

4 Principle

Measurement of the volume of a known mass of fat at 60 °C and at various temperatures below 60 °C. Calculation of the volume of the liquid supercooled fat at other temperatures as required from the volume at 60 °C.

1) To be published. (Revision of ISO 5555:1983.)

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5 Reagent

5.1 Sealing liquid, consisting of distilled water boiled out under vacuum and coloured using a 10 g/l solution of congo-red, methyl orange or potassium dichromate.

6 Apparatus

Usual laboratory and, in particular, the following.

6.1 Round-bottomed flask, of 100 ml capacity.

6.2 Burette, graduated in divisions of 0,05 ml or less.

6.3 Dilatometer, (see figure 1).

The bulb of the dilatometer shall have a volume of (7 ± 0,5) ml, and the graduated part of the capillary tube shall have a volume of 900 µl. The zero mark on the capillary tube shall be level with the top of the neck of the dilatometer bulb. The stopper shall be filled with a sufficient quantity of lead-shot (or other suitable material) to prevent the dilatometer from floating in the water-bath.

The dilatometer shall be carefully cleaned before use. It shall have a regularity of the graduated scale within 2 µl and an accuracy of ± 0,25 %, calculated in accordance with 6.3.1 and 6.3.2 respectively.

6.3.1 Regularity of the graduated scale.

Introduce through the bulb of the dilatometer (6.3) into the capillary tube approximately 250 µl of mercury. Place the dilatometer in a horizontal position on two wooden blocks and place a small mirror under the capillary tube to avoid errors in reading due to parallax. When the dilatometer and mercury have attained room temperature, read the volume of mercury in scale units. Slightly displace the mercury column along the capillary tube and again read the volume of mercury in scale units. Repeat this procedure for various positions along the capillary.

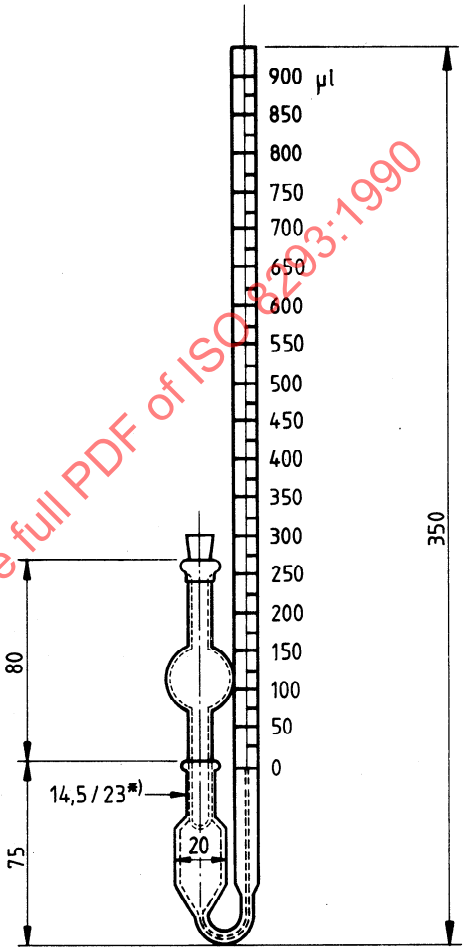
If the differences between the readings exceed 2 µl, or if they regularly increase or decrease, reject the tube.

6.3.2 Accuracy of the graduated scale.

Ensure that the capillary tube is empty. Introduce about 750 µl of mercury into the capillary tube via the bulb. Place the dilatometer in a horizontal position on two wooden blocks alongside a thermometer, and position a small mirror under the capillary tube.

Read the volume of the mercury and note the temperature. Slightly displace the mercury column along the capillary and again read the volume and note the temperature. Repeat this process several times. Pour the mercury from the 900 µl graduated end of the capillary tube into a tared flask and weigh.

Dimensions in millimetres



*) In accordance with ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints.*

Figure 1 — Dilatometer

Calculate the volume of mercury from its mass and apparent density at the measuring temperature. Deduct 1 µl from the observed volume as a correction for the meniscus reading, and compare the corrected observed volume with the true volume.

Reject dilatometers showing differences of 0,5 % or more.

6.4 Water-baths.

6.4.1 Each water-bath shall be fitted with

a) a thermostat, capable of maintaining the water

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at any selected temperature between 10 °C and 60 °C to within ± 0,1 °C,

b) a vigorous stirrer, and

c) a thermometer (see 6.6).

6.4.2 Depending on whether one or two water-baths are used for the determination, the heating capacity of the water-baths shall be sufficient to increase the temperature of the water

a) at a rate of at least 1 °C/min, if one water-bath is used;

b) by 10 °C within a maximum of 30 min, in the case of two water-baths.

6.4.3 The water-baths shall be capable of producing a constancy of temperature of approximately 0,05 °C throughout the water-bath.

NOTE 3 The efficiency of stirring and possible deviation of the temperature of the water in the immediate neighbourhood of the dilatometer can be checked by inserting a dilatometer filled with hard fat (e.g. hardened palm oil, melting point 42 °C to 48 °C) and fitted with a thermometer.

The reading of this thermometer should not deviate more than 0,05 °C from that of the other thermometer in the water-bath.

6.4.4 The water-baths shall be equipped with an arrangement for suspending the dilatometers in the water and which allows individual vertical adjustment and easy transfer from one water-bath to another.

6.5 Bath at 0 °C.

If crushed ice is used for the bath at 0 °C, the vessel shall have drain holes, otherwise water at a temperature above 0 °C will collect at the bottom of the vessel. The ice shall be stirred and repacked frequently, especially if several dilatometers are being cooled from 60 °C to 0 °C in the same bath. A cooling unit that can be maintained at 0 °C is recommended, but this will need to be filled with a liquid having a freezing point below 0 °C.

6.6 Thermometers, graduated in divisions of 0,05 °C, covering the required range of temperatures and calibrated against an official standard thermometer.

6.7 Water-bath, containing boiling water.

6.8 Boiling aids.

7 Sampling

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

8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Types of procedure

There are three types of procedure as follows:

a) normal short range: measurements are made at temperatures of 20 °C, 25 °C, 30 °C and 35 °C, and at 5 °C intervals thereafter up to 60 °C if required;

b) normal long range: measurements are made at temperatures of 0 °C, 10 °C, 15 °C, 20 °C and 25 °C, and at 5 °C intervals thereafter up to 60 °C if required;

c) rapid method for factory control: measurements are made at temperatures of 20 °C and 30 °C, and at any higher temperature if required.

9.2 Test portion

Transfer 15 ml to 20 ml of the test sample (clause 8) into a 100 ml round-bottomed flask (6.1), add some boiling aids (6.8), heat to 80 °C to 100 °C in a boiling water-bath (6.7) and evacuate in a sufficient vacuum, shaking vigorously at intervals until air bubbles no longer escape from the boiling aids (this may take 10 min). Store the test portion under vacuum at 70 °C until required (see 9.3).

9.3 Filling the dilatometer

Care shall be taken not to heat the capillary tube by the hands or breath.

By means of a burette (6.2) introduce 1,5 ml of the sealing liquid (5.1) into the dilatometer (6.3). Weigh the dilatometer with the stopper to the nearest 10 mg.

Fill the dilatometer bulb to half-way up the neck with the prepared test portion (9.2), if necessary after warming the dilatometer to prevent the fat from solidifying. Insert the stopper carefully to avoid trapping air bubbles and at the same time to drive the sealing liquid up the capillary tube. When the sealing liquid in the capillary tube rises to the 600 µl to 700 µl mark, close the capillary tube almost completely with the tip of the finger. Then place the stopper securely in position and remove the finger.

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Check that no air has been trapped under the stopper. Clean the outside of the bulb with solvent, dry and weigh the dilatometer again to determine the mass of the fat.

9.4 Volume of liquid fat

Immerse the dilatometer to the zero mark in a water-bath (6.4) maintained at (60 ± 0,1) °C. Read the level of the sealing liquid in the capillary tube after not less than 30 min.

NOTE 4 A burette magnifier increases the precision with which the reading can be made.

9.5 Maturing

9.5.1 All fats

Remove the dilatometer from the water-bath (6.4) and immerse it to the zero mark in the bath at 0 °C (6.5) for 90 min.

For non-polymorphic fats, for which the dilatation D_0 is required at 0 °C, record the level of the meniscus in the capillary tube and proceed to 9.6. For polymorphic fats, proceed to 9.5.2.

9.5.2 Further maturing for polymorphic fats

Remove the dilatometer from the bath at 0 °C and immerse it to the zero mark in a water-bath (6.4) fitted with a stirrer and maintained at (26 ± 0,2) °C for (40 ± 0,5) h. During this time, the capillary tube shall be capped in order to avoid evaporation of the sealing liquid.

Immerse the dilatometer again to the zero mark in the bath at 0 °C (6.5) for 90 min. Note the level of the meniscus in the capillary tube if the value D_0 is required.

9.6 Reading the dilatometer

If, by error, the temperature of a water-bath exceeds the intended temperature, the water-bath shall not be allowed to cool (since the melted part of the fat will not recrystallize) and the dilatation of the fat shall be measured and reported at this temperature.

9.6.1 Remove the dilatometer from the bath at 0 °C and immerse it to the zero mark in a water-bath (6.4) maintained at the lowest temperature at which the dilatation is to be measured. Read the level of the meniscus 30 min after the water-bath has reached the required measuring temperature. Repeat the reading at 5 min intervals until it is constant.

Similarly, take readings of the meniscus level at successively higher temperatures, 30 min after the water-bath has attained the measuring temperature in each case.

9.6.2 Repeat the reading at (60 ± 0,1) °C; this value should agree to within 2 µl with the value found in 9.4. A difference greater than 2 µl could be the consequence of a leak in the dilatometer or of the presence of air in the fat.

9.7 Number of determinations

Carry out two determinations as in 9.3 to 9.6 on test portions taken from the same test sample.

10 Expression of results

The dilatation D_t of the fat at temperature t °C is given by the formula

$$D_t = \frac{A_{60} - A_t - W_t}{m} - V_t$$

where

A_{60} is the reading at 60 °C, in microlitres;

A_t is the reading at t °C, in microlitres;

m is the mass, in grams, of the fat in the dilatometer;

V_t is the expansion of the oil, i.e. the difference between the volumes, in microlitres, of 1 g of melted fat at 60 °C and of the same fat supercooled at t °C (see table 2);

W_t is the correction, in microlitres, for the sealing liquid, i.e. the correction for the expansion of the sealing liquid and glass between 60 °C and t °C (see table 1).

Report the result to the nearest half-unit.

Take as the result the arithmetic mean of the two determinations provided that the requirements for repeatability (see clause 11) are met. Otherwise repeat the procedure as in 9.2 to 9.7, and take as the result the median of the four determinations; state this incident in the test report.

A specimen dilatation report for the calculation of the dilatation is shown in annex A; the use of this form is optional but recommended.

To find the correction W_t, in microlitres, for the expansion of the sealing liquid and glass at various temperatures and various observed levels of the meniscus (from the zero of the capillary tube to the level of the water in the water-bath), round off the readings of the position of the meniscus to multiples of 100, and read the correction W_t from table 1.

Values of V_t are given in table 2.

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Table 1 — Correction W_t as a function of temperature t

Temperature t °C	Correction W_t, µl, for a meniscus level of									
	0	100	200	300	400	500	600	700	800	900
0	24	22	20	18	16	14	12	11	9	7
10	22	21	19	17	15	14	12	11	9	7
15	21	20	18	16	15	13	12	10	8	7
20	20	18	17	15	14	12	11	9	8	6
25	18	17	16	14	13	11	10	9	7	6
30	17	15	14	13	12	10	9	8	6	5
35	15	13	12	11	10	9	8	7	6	5
40	12	11	10	9	9	8	7	6	5	4
45	10	9	8	7	7	6	5	5	4	3
50	7	6	6	5	5	4	4	3	3	2
55	3	3	3	3	2	2	2	2	1	1
60	0	0	0	0	0	0	0	0	0	0

Table 2 — Values of V_t

Temperature t °C	V_t µl/g
0	50,6
10	42,4
15	38,3
20	34,1
25	29,9
30	25,7
35	21,5
40	17,2
45	13,0
50	8,6
55	4,3
60	0

NOTE 5 Corrections for the expansion of glass, of the sealing liquid and of the oil are as follows.

a) Glass correction

For a dilatometer of volume 7 ml, the glass correction, based on a coefficient of linear expansion of 33×10^{-7} cm/°C, is 0,07 µl/°C.

b) Correction for the expansion of water and glass between t °C and 60 °C

The equation giving the variation in the coefficient of expansion of water between 0 °C and 60 °C may be taken to be linear. Using the known values above 20 °C and extrapolating to 0 °C the equation becomes

$$E_t = (60 - t)\frac{2{,}3 + 0{,}09(60 + t)}{25}$$

This quantity is multiplied by the volume of water remaining in the bulb at each temperature

$$1{,}5 - 0{,}001 \times \text{reading at } t\ °C$$

to give the water correction. The mean glass correction, i.e. $0{,}07(60 - t)$, is subtracted from this to yield the values shown in table 1.

c) Correction for the expansion of the oil

The correction V_t, in microlitres per gram, for the expansion of the oil is given by the following equation:

$$V_t = \frac{1}{25}\int_t^{60}(20{,}5 + 0{,}02t)\mathrm{d}t$$

11 Repeatability

The results of two determinations (see 9.7) carried out in rapid succession or simultaneously by the same operator shall not differ by more than 1,6 µl/g (absolute).

NOTE 6 The value of 1,6 µl/g is taken from the Netherlands standard NEN 6317:1981, *Onderzoekingsmethoden voor plantaardige en dierlijke oliën en vetten. Bepaling van de dilatatie van vetten.*

12 Test report

The test report shall mention whether the "general method" (9.5.1 and 9.6) or the "polymorphic method" (9.5.2 and 9.6) was used and whether the "normal short-range procedure", the "normal long-range procedure" or the "rapid method for factory control" was used (since the number of temperature steps used influences the dilatation values).

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.

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