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Animal and vegetable fats and oils — Determination of solid fat content — Pulsed nuclear magnetic resonance method

Corps gras d'origines animale et végétale — Détermination de la teneur en corps gras solides — Méthode par résonance magnétique nucléaire pulsée

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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.

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 8292 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 11, *Animal and vegetable fats and oils*.

Annex A of this International Standard is for information only.

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Animal and vegetable fats and oils — Determination of solid fat content — Pulsed nuclear magnetic resonance method

1 Scope

This International Standard specifies a method for the determination of the solid fat content in animal and vegetable fats and oils (hereinafter referred to as fats) using low-resolution pulsed nuclear magnetic resonance. Alternative thermal pretreatments are specified according to whether or not the fat exhibits pronounced polymorphism.

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

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was 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 edition of the standard 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.*

3 Definition

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

3.1 solid fat content: The percentage by mass of fat in the solid state at a specified temperature when measured by pulsed nuclear magnetic resonance under the conditions specified in this International Standard.

4 Principle

Preparation of test portions at specified temperatures. Measurement of the magnetization decay signals from the solid and liquid fat protons using pulsed nuclear magnetic resonance, with automatic calculation and display of the solid fat content.

5 Material

Calibration material, of known instrument response.

NOTE 2 Calibration materials with known responses are supplied by the instrument manufacturer. Materials giving responses of 0 % (m/m) and of about 35 % (m/m) and 70 % (m/m) are suitable. These response values are constant for all measurement temperatures.

6 Apparatus

6.1 Measuring tubes, suitable for use with the nuclear magnetic resonance instrument.

6.2 Metal blocks, preferably aluminium, with holes. The diameter of the holes shall not be more than 0,4 mm greater than the average diameter of the measuring tubes. The depth of the holes shall be such that the level of the fat is approximately 10 mm below the upper surface of the block. The thickness of the metal under the holes and the distance between the edge of a peripheral hole and the nearest side face shall be 10 mm. The distance between the axes of two adjacent holes shall be 7 mm greater than the diameter of the holes. One block is required for each water-bath used.

6.3 Metal racks, of a depth such that the surface of the fat is 5 mm below the surface of the water-bath in which the rack is placed.

6.4 Water-baths, capable of being maintained at 80 °C ± 0,1 °C, 60 °C ± 0,1 °C, 26 °C ± 0,1 °C and 10 °C ± 0,1 °C.

6.5 Water-baths, accurate to ± 0,1 °C, set for each of the measurement temperatures and each containing one of the metal blocks (6.2), supported so that the upper surface of the block is 5 mm above the surface of the liquid.

6.6 Non-freezing liquid bath, capable of being maintained at 0 °C ± 0,1 °C.

6.7 Pulsed nuclear magnetic resonance instrument, low resolution, having the following characteristics:

a) magnet with a sufficiently uniform field to ensure that the half-life of the magnetization of a reference sample of liquid fat is longer than 1 000 µs;

b) measurement dead time plus impulse width of less than 10 µs;

c) automatic measuring device which operates as soon as the measuring tubes are inserted;

d) adjustable measurement repetition time;

e) adaptor in the magnetization zone to take the measuring tubes (6.1);

f) digital voltmeter for direct measurements.

NOTES

3 For preference, the instrument should be equipped with a computer which automatically takes three successive measurements each at 10 µs and 70 µs, with 2 s intervals, and which also calculates the mean solid content which is read directly from the digital voltmeter screen.

4 An instrument of the Bruker Minispec[1] type is suitable.

5 It is recommended that the work area be maintained at a temperature lower than that at which the magnet is stabilized.

6 For determinations on cocoa butter and similar fats, the computer should be adjusted so that there is a 6 s interval between two successive measurements. Thus the displayed result is from one determination only.

7 Sampling

Sampling should 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 Thermal pretreatment

Fill one measuring tube (6.1) for each measurement temperature T with a portion of the test sample (clause 8). Place the tube successively in the water-baths (6.4) at the temperatures and for the times indicated in 9.1.1 to 9.1.4, wiping the tube before placing it in the block in the water-bath (6.5) at T °C.

Where a "longer" time is used, this shall be the same for all samples.

9.1.1 Fats not in 9.1.2, 9.1.3 or 9.1.4

Melt the test portion at 80 °C.

Keep it at

60 °C for 5 min (or longer);

0 °C for 60 min ± 2 min;

T °C for 30 min (or longer).

9.1.2 Tallow and tallow fractions (tallow stearine, oleo oil)

Melt the test portion at 80 °C.

Keep it at

60 °C for 15 min (or longer);

0 °C for 24 h ± 0,5 h;

T °C for 30 min (or longer).

9.1.3 Palm oil

Melt the test portion at 80 °C.

Keep it at

60 °C for 5 min (or longer);

10 °C for 16 h ± 0,5 h;

1) Bruker Minispec is an example of a suitable apparatus available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this apparatus.

T °C for 30 min (or longer).

9.1.4 Cocoa butter and other fats showing pronounced polymorphism

Melt the test portion at 80 °C.

Keep it at

60 °C for 5 min (or longer);

0 °C for 90 min ± 5 min;

26 °C for 40 h ± 0,5 h;

0 °C for 90 min ± 5 min;

T °C for 60 min (or longer).

9.2 Determination

9.2.1 Prepare the nuclear magnetic resonance instrument (6.7) in accordance with the manufacturer's instructions, using the calibration material (clause 5) as required at each measurement temperature.

9.2.2 Take the tubes one at a time from the blocks after the appropriate time (see 9.1.1 to 9.1.4), wipe off any condensation and place them immediately in the instrument. Read the solid fat content from the digital display.

9.3 Number of determinations

Carry out two determinations at each chosen temperature on test portions taken from the same test sample.

10 Expression of results

Express the result as the arithmetic mean of the two determinations provided that the requirement for repeatability (see 11.2) is satisfied.

11 Precision

11.1 Statistical summary

International collaborative studies carried out on four types of fat (which do not exhibit pronounced polymorphism) in 36 laboratories and organized by Unilever Research Laboratory, Vlaardingen, gave the results (evaluated in accordance with ISO 5725) shown in table 1.

11.2 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, shall not exceed the value given in table 1 for the appropriate solid fat content.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different materials, is not expected to exceed the value given in table 1 for the appropriate solid fat content on more than one occasion in twenty at the 90 % confidence level.

12 Test report

The test report shall specify the method and instrument used, the measurement temperatures used and the results 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 results.

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

Table 1 — Statistical summary of collaborative results

Solid fat content	Repeatability		Reproducibility	
	absolute	relative	absolute	relative
% (m/m)	%	%	%	%
10	1,0	10	1,8	18
30	1,3	4,3	2,8	9,3
50	1,6	3,2	3,8	7,6
60	1,8	2,7	4,8	6,9

NOTE — The fats used in the collaborative study were fish oil, palm oil, lard and a margarine. None of these fats exhibited polymorphism.

Annex A
(informative)

Bibliography

[1] ISO 5555:1991, *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.*

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