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Animal and vegetable fats and oils — Determination of solid fat content by pulsed NMR —

Part 2:
Indirect method

Corps gras d'origines animale et végétale — Détermination de la teneur en corps gras solides par RMN pulsée —

Partie 2: Méthode indirecte

Reference number
ISO 8292-2:2008(E)

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 8292-2 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

This part of ISO 8292, together with ISO 8292-1, cancel and replace ISO 8292:1991.

ISO 8292 consists of the following parts, under the general title *Animal and vegetable fats and oils — Determination of solid fat content by pulsed NMR*:

- *Part 1: Direct method*
- *Part 2: Indirect method*

Animal and vegetable fats and oils — Determination of solid fat content by pulsed NMR —

Part 2:
Indirect method

1 Scope

This part of ISO 8292 specifies an indirect method for the determination of the solid fat content in animal and vegetable fats and oils (hereafter designated “fats”) using low-resolution pulsed nuclear magnetic resonance (NMR) spectrometry.

Two alternative thermal pre-treatments are specified: one for general purpose fats not exhibiting pronounced polymorphism and which stabilize mainly in the β′-polymorph; and one for fats similar to cocoa butter which exhibit pronounced polymorphism and stabilize in the β-polymorph. Additional thermal pre-treatments, which may be more suitable for specific purposes, are given in an informative annex.

The indirect method is less easy to carry out and less reproducible than the direct method, but is more accurate and more universally applicable to all fats.

NOTE A direct method is specified in ISO 8292-1.

2 Normative references

The following referenced documents are indispensable for the application 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*

ISO 3960, *Animal and vegetable fats and oils — Determination of peroxide value — Iodometric (visual) endpoint determination*

ISO 8292-1, *Animal and vegetable fats and oils — Determination of solid fat content by pulsed NMR — Part 1: Direct method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8292-1 apply.

4 Symbols and abbreviated terms

f	conversion (extrapolation) factor to correct the NMR signal observed at 11 μs to that at time zero
n_p	number of pulses
S_1	magnetization decay signal measured at about 11 μs
S_2	magnetization decay signal measured at about 70 μs
SFC	solid fat content
S_L	magnetization decay signal corresponding to the liquid phase
S_S	magnetization decay signal corresponding to the solid phase
$S_\mathrm{S+L}$	magnetization decay signals corresponding to both solid and liquid phases
t_rep	repetition time
$w_\mathrm{SFC,i}$	"true" SFC (measured in accordance with this part of ISO 8292)
$w_{\mathrm{SFC},T}$	SFC at measurement temperature, T

5 Principle

The sample is tempered to a stable state at a specific temperature and then heated to, and stabilized at, the measurement temperature. Unless otherwise specified, measurement temperatures can be any or all of: 0 °C; 5 °C; 10 °C; 15 °C; 20 °C; 25 °C; 27,5 °C; 30 °C; 32,5 °C; 35 °C; 37,5 °C; 40 °C; 45 °C; 50 °C; 55 °C; 60 °C.

After electromagnetic equilibration in the static magnetic field of the NMR spectrometer and application of a 90° radio frequency pulse, the magnetization decay signal from the protons in the liquid phase only is measured and the solid fat calculated by reference to a standard sample consisting entirely of liquid fat. Provided that the liquid fat measurements have been made at the start of the SFC determination, results can be displayed immediately as in the direct method.

6 Triolein standard sample

The liquid fat standard sample shall contain a minimum 99 % mass fraction of triolein. The peroxide value determined according to ISO 3960 shall be less than 5. Store triolein stocks at a temperature below 0 °C. Store reference tubes containing triolein in a refrigerator at 0 °C to 8 °C when not in use, and replace them every 2 weeks.

7 Apparatus

7.1 Pulsed nuclear magnetic resonance (NMR) spectrometer, low resolution

The NMR spectrometer shall have:

a) 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) an automatic measuring device which operates as soon as the measurement tubes (7.2) are inserted;

c) an adjustable measurement repetition time;

d) a 10 mm measurement cell/probe for test portion tubes which is temperature-controlled at 40 °C.

For preference, the instrument should be equipped with a computer which automatically takes the required measurements, performs the required calculations and presents the results directly on the computer screen or other display.

7.2 Measurement tubes

Made of glass with plastic caps, with outer diameter (10 ± 0,25) mm, wall thickness (0,9 ± 0,25) mm, and length at least 150 mm, or as specified by the NMR spectrometer manufacturer.

7.3 Temperature-maintenance equipment

7.3.1 General

In principle, temperature-controlled blocks have advantages over water baths because the tubes can never come into contact with water. In practice, as with aluminium blocks in water baths, the tubes may take a significant time to come to the set temperature. Heat transfer can be improved if the tube wells are purged with a dry gas. Blocks are also more difficult to control precisely than water baths, although modern electronic controls may provide the required precision.

7.3.2 Water baths

Baths are required at temperatures of (0 ± 0,1) °C, (60 ± 0,1) °C, and, to within ± 0,1 °C, the measuring and tempering temperatures required according to the measurement protocol chosen. For the 60 °C, measurement temperature, and tempering temperature baths, temperature-controlled blocks (7.3.3) may be substituted. Each water bath shall be equipped with either one aluminium block (7.3.2.1) or one metal rack (7.3.2.2) to accommodate measurement tubes (7.2) immersible in the water to a depth of 60 mm.

Metal racks are preferred to aluminium blocks, especially when a large number of test samples with high SFC are being measured or when the rapid or ultra-rapid measurement protocols are being used. When using aluminium blocks, there may be a significant time lag after the tube is inserted before the fat in the tube reaches the set temperature of the water bath. The perceived advantage of blocks is that the tubes can remain dry and do not need to be wiped dry with a paper tissue before insertion into the spectrometer. In practice, however, it is usually found that due to splashing or condensation, the tubes do become wet so that drying is always recommended, see Clause 9.

7.3.2.1 Aluminium blocks, with holes of diameter (10,35 ± 0,1) mm, and depth 70 mm. 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 at least 17 mm (centre to centre).

7.3.2.2 Metal racks, open-sided, with holes of diameter 11 mm to 15 mm; the distance between the axes of two adjacent holes shall be at least 20 mm (centre to centre).

7.3.3 Temperature-controlled blocks, with holes

The blocks, with electronic control, shall be capable of being maintained to within ± 0,1 °C. These blocks may be used instead of water baths (except the 0 °C bath, because of the large amount of cooling required). The diameter of the holes shall be (10,35 ± 0,1) mm.

Blocks are particularly useful at temperatures of 35 °C or more when no cooling is required (assuming the ambient room temperature is below 22 °C) and where temperature control is less critical because of the usually lower absolute solid fat levels.

7.4 Oven, with fan

The oven shall be capable of being maintained at (80 ± 2) °C.

Since the purpose of the 80 °C temperature is to melt the test portion and destroy its previous thermal history, it shall be at least 20 °C above the melting temperature of the fat. If this is not the case, then the oven temperature shall be raised accordingly and the fact recorded in the test report (Clause 12). This is rarely necessary, as the fats concerned contain large amounts of long-chain saturated fatty acids, e.g. fully hydrogenated liquid vegetable oils.

Although a water bath (7.3.2) or temperature-controlled block (7.3.3) may be used for the 80 °C temperature, it is preferable to use an oven. In a block or bath it is almost inevitable that fat will contact the sides, at a temperature above that of immersion, when filling the tubes. An oven ensures that all the fat in the tube is completely melted and there are no seed crystals remaining with an unknown thermal history which could seed the molten fat when it is eventually moved to the 0 °C crystallization temperature. Thus, an oven is likely to give more reliable and reproducible results.

7.5 Stop-clock

An analogue clock with a large sweep second hand is preferred, although a digital clock may be used.

8 Sampling

A representative sample shall have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this part of ISO 8292. A recommended sampling method is given in ISO 5555.

9 Procedure

9.1 Measurement protocol and test sample

Choose the required protocol from Table 1 according to the sample type and other requirements. For some types or applications of fats, the protocols given in Table 1 will not be appropriate. The measurement protocols given in Annex C may be more suitable.

Prepare the test sample in accordance with ISO 661.

9.2 Oven, water baths and temperature-controlled blocks

Set this equipment up for the required temperatures as specified in the protocol.

9.3 NMR spectrometer

Set the conditions for the spectrometer according to the measurement protocol chosen in 9.1.

9.4 Filling the measurement tubes

From each test sample, fill two measurement tubes. Additionally, fill two tubes with triolein reference sample (Clause 6). The indirect method has a lower repeatability than the direct method and two tubes are recommended to obtain similar repeatability for the two methods.

Fill the tubes to a depth of about 10 mm, or as specified by the instrument manufacturer.

NOTE The use of only 10 mm depth with the indirect method, as recommended by instrument manufacturers, will also require a different spacer to raise the tube so that all the test portion lies within the receiver coil of the NMR spectrometer. In practice, provided all the tubes are filled to the same height, it will be found that results are little different if the tubes are filled in the same way as for the direct method, i.e. to a depth of 30 mm to 50 mm, and used with the same spacer.

Cap the tubes and place in racks that keep the tubes vertical. If metal racks (7.3.2.2) are used, it is very convenient and time saving to put the filled tubes directly into the racks. The test portions can then be moved conveniently to the oven and to the water baths without further transfers and handling.

Table 1 — Measurement protocols

Measurement protocol		Applicable to	Instrumental conditions	First time at 0 °C	Tempering		Second time at 0 °C	Measurement conditions	
No.	Name			min	Time h	Temp. °C	min	Type	Time min
1I	Non-stabilizing indirect	Fats and blends (comprising mainly vegetable fats, hydrogenated and/or interesterified) crystallizing in the β'-polymorph and as used for margarines, spreads, shortenings and other general food applications	repetition time [a], t_{rep} = 2 s; No. pulses [b], n_p = 3	—	—		(60 ± 2)	Series	(30 ± 1)
2I	β-Stabilizing indirect	Cocoa butter, cocoa butter equivalents and similar fats containing large amounts of 2-oleo-di-saturated triacylglycerols and crystallizing in the β-polymorph	repetition time, t_{rep} = 6 s; No. pulses [c], n_p = 1	(90 ± 2)	(40 ± 0,5)	26	(90 ± 2)	Series	(60 ± 2)

[a] Needs to be 6 s for fats in the β-polymorph.

[b] Pulse data are averaged by the instrument. Ideally, three pulses are used, but some older instruments can be set to only either one or four (1^2 or 2^2) pulses, in which case use four pulses.

[c] Use of three pulses would result in sufficient time in the measurement cell to cause the test portion to partially melt and the SFC to reduce during the measurement.

9.5 Removing the thermal history

When all the required tubes have been filled, transfer them to the oven (7.4). Hold at the oven temperature for a minimum of 15 min.

9.6 Equilibrating at the initial temperature and measuring the 100 % liquid signal

Transfer all the tubes to the 60 °C water bath (7.3.2) or block (7.3.3). Hold for a minimum of 15 min. The time may be longer than this, but shall not be shorter as otherwise complete equilibration may not be achieved.

For all test portions and triolein reference sample (Clause 6), record the liquid magnetization decay signal measured at about 70 µs, S_2. If any of the test portions are not completely liquid, then the measurement procedure shall be repeated using a temperature above 60 °C, at which the test portion is known to melt completely.

9.7 Crystallization and tempering

From this stage onwards all the times shall be maintained within the tolerances specified here or in the measurement protocol.

If required by the chosen measurement protocol, transfer the tubes into the 0 °C bath. Leave in the 0 °C bath for the time specified as “first time at 0 °C” column in the measurement protocol.

If required by the chosen measurement protocol, transfer the tubes into the tempering bath set to the specified temperature. Leave in the tempering bath for the specified time.

Starting with the reference triolein, at (1,0 ± 0,5) min intervals, transfer the tubes into the 0 °C bath. Leave in the 0 °C bath for the time specified as “second time at 0 °C” column in the measurement protocol.

9.8 Measuring the SFC

For the indirect method, measurements are made in series.

At (1,0 ± 0,5) min intervals, in exactly the same sequence as they were placed in the 0 °C bath, transfer the two tubes containing test portions to the first (lowest) of the measurement temperature baths or blocks.

NOTE Experience shows that it is easily possible to transfer a tube from bath or block to the spectrometer and make the measurement within 15 s. Therefore two tubes may comfortably be processed within 1 min.

After the time specified in the measurement protocol, continuing in exactly the same sequence, transfer the tubes to the spectrometer. Wipe each tube briefly with a soft paper tissue to remove all water, before placing it in the measurement cell. Record the SFC (or if automatic calculation is not possible, the liquid magnetization decay signal measured at about 70 µs, S_2). If the NMR spectrometer is equipped with a computer then, provided that the triolein reference sample tubes are measured first, the SFC values can be displayed immediately in a similar way to that usual for the direct method.

Transfer the tubes containing each test portion to the second (next lowest) of the measurement temperature baths or blocks at (1,0 ± 0,5) min intervals.

Repeat the procedures from the second paragraph until all the tubes have been measured.

If necessary, calculate the SFC using Equation (1) (see Clause 10).

IMPORTANT — For reliable and reproducible results, adhere to the times and tolerances specified. This is easily achieved using a laboratory stop-clock (7.5), preferably an analogue clock with a large sweep second-hand, moving the tubes as the clock moves round to the appropriate time. Alternatively, if a digital clock is used, it is convenient to set it to 0:00 or 12:00 at the start.

9.9 Number of determinations

Carry out two determinations (9.4) on separate test portions in separate tubes taken from the same test sample.

9.10 Cleaning the measurement tubes

Measurement tubes shall be clean, dry, and free from all fat from previous measurements before filling with the test portion. Because of the narrow diameter of the tubes, cleaning often proves to be a problem. Solvents or narrow brushes are often used. Tubes may be cleaned in a laboratory automatic washer or a standard domestic dishwasher. However, for cleaning to be effective, it is necessary to ensure that the tubes are free of most of the fat and are maintained more or less vertically in the washer. This may be achieved as follows.

Either use a laboratory washer, equipped with special support "fingers" which can just fit into the tube and inject hot detergent solution inside.

Or use a washer without special "fingers" by supporting the tubes in a wire-mesh rack with slots of the correct size to take the tubes. The rack should be equipped with a wire-mesh lid to retain the tubes when the rack is inverted. An advantage of such a rack is that, as they are finished with at the end of the measurement sequence, the tubes can be placed directly upside down in the rack and then the filled rack placed in the 80 °C oven for some time to allow the fat to melt and most of it to drain away. Still inverted, the rack can then be transferred to the washer. After washing and drying, the tubes rack can be used as a convenient holder or the tubes removed and stored ready for re-use.

10 Expression of results

If the NMR spectrometer is not equipped with a computer or other automatic calculation device to read off the results, use the manually recorded signals to calculate the SFC at a given temperature, $w_{\mathrm{SFC},T}$, as a percentage mass fraction, using Equation (1):

$$w_{\mathrm{SFC},T} = \left(1 - \frac{S_{2,\mathrm{ref},60} \times S_{2,\mathrm{SFC},T}}{S_{2,\mathrm{SFC},60} \times S_{2,\mathrm{ref},T}}\right) \times 100 \qquad (1)$$

where

$S_{2,\mathrm{ref},60}$ is the magnetization decay signal measured at about 70 µs of the triolein reference using a 60 °C water bath or block;

$S_{2,\mathrm{ref},T}$ is the magnetization decay signal measured at about 70 µs of the triolein reference at the measurement temperature;

$S_{2,\mathrm{SFC},60}$ is the magnetization decay signal measured at about 70 µs of the test portion using a 60 °C water bath or block;

$S_{2,\mathrm{SFC},T}$ is the magnetization decay signal measured at about 70 µs of the test portion at the measurement temperature.

See Annex B for more details of the theory.

Express the result as the arithmetic mean of the two determinations (9.9), provided that the requirement for repeatability (11.2) of each $w_{\mathrm{SFC},T}$ value is satisfied. Report the result to one decimal place.

11 Precision

11.1 Interlaboratory test

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

There were insufficient data to carry out a meaningful statistical evaluation according to ISO 5725-1 and ISO 5725-2. Therefore only the actual results are given in Annex A.

11.2 Repeatability

There were insufficient data to carry out a meaningful statistical evaluation according to ISO 5725-1 and ISO 5725-2.

11.3 Reproducibility

There were insufficient data to carry out a meaningful statistical evaluation according to ISO 5725-1 and ISO 5725-2.

12 Test report

The test report shall specify at least the following information:

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

b) details of the NMR spectrometer used;

c) the method used, with reference to this part of ISO 8292;

d) the measurement protocol used;

e) the measurement temperatures used;

f) whether a water bath with aluminium blocks, water bath with metal racks or temperature-controlled blocks were used for temperature control;

g) the results obtained;

h) all operating details not specified in this part of ISO 8292, or regarded as optional, together with details of any incidents which may have influenced the results.

Annex A
(informative)

Results of interlaboratory tests

Table A.1 — Results of all laboratories — Measurement protocol 1I

		Hydrogenated soybean oil (A)		Shortening blend, hydrogenated hardstock (B)		Shortening blend, interesterified hardstock (C)		Palm kernel stearin (D)		Coconut oil (E)		Palm oil/palm stearin blend (F)	
Slip melting point		~37 °C		~40 °C		~40 °C		—		—		~45 °C	
Iodine value		—		—		—		~7		—		—	
Lab No	**Sample**												
3	**10 °C**	76,6		65,4		75,8		95,7		78,9		55,0	
7	**10 °C**	72,8	72,6	66,0	66,3	72,1	72,5	97,2	97,3	75,8	75,4	56,7	56,5
13	**10 °C**	78,4	78,8	67,6	67,5	77,9	77,9	96,0	96,1	81,8	81,5	57,1	57,4
15	**10 °C**	89,5	89,9	83,9	84,4	85,5	87,0	100,0	100,0	92,5	93,1	79,4	79,6
17	**10 °C**	80,9	81,2	71,2	71,0	80,0	80,0	96,4	96,3	84,0	83,8	61,9	61,8
18	**10 °C**	78,0	78,3	65,9	66,1	77,7	77,8	96,8	96,8	81,4	81,5	55,3	55,1
21	**10 °C**	52,0	51,6										
23	**10 °C**												
3	**20 °C**	56,7		43,8		53,8		94,3		38,0		35,9	
7	**20 °C**	56,1	55,9	45,6	45,8	53,9	54,0	94,8	95,0	39,6	39,8	37,2	37,2
13	**20 °C**	55,9	56,2	44,9	44,8	54,2	54,3	94,9	94,8	36,1	36,6	37,1	37,1
15	**20 °C**	74,8	75,7	64,4	66,5	69,6	73,6	100,0	100,0	49,7	47,3	53,9	59,4
17	**20 °C**	59,3	59,6	48,5	48,3	57,2	56,8	95,2	95,1	40,0	40,0	39,8	40,1
18	**20 °C**	55,8	55,7	43,7	43,8	53,6	53,7	95,2	95,2	36,4	36,6	35,3	35,4
21	**20 °C**	27,1	25,9	78,3	78,6	77,7	75,0	91,2	91,3	70,5	69,9	80,6	80,8
23	**20 °C**	56,2	55,7	42,7	44,0	54,2	53,7	95,0	94,4	37,6	37,7	35,8	36,4
3	**25 °C**	43,1		30,0		40,5		85,0		2,3		24,3	
7	**25 °C**	40,1	40,3	31,8	31,8	39,2	39,6	87,6	87,3	5,6	5,8	25,1	25,0
13	**25 °C**	40,7	41,0	32,1	31,9	40,7	40,7	83,5	83,8	0,4	0,6	25,8	26,0
15	**25 °C**												
17	**25 °C**	42,8	43,1	33,9	33,7	42,4	42,3	84,9	85,0	2,5	2,2	27,0	27,2
18	**25 °C**	41,5	41,5	32,1	32,0	40,4	40,5	84,1	84,6	1,7	1,7	25,4	25,5
21	**25 °C**	23,9	23,1	72,8	72,8	72,3	72,6	89,7	89,5	56,7	57,2	68,2	68,2
23	**25 °C**	43,0	42,4	32,7	33,8	41,3	41,1	84,8	84,9	−1,0	−0,8	26,9	27,7
3	**30 °C**	26,5		20,2		27,1		41,2		< 2,0		16,6	

Table A.1 (*continued*)

		Hydrogenated soybean oil (A)		Shortening blend, hydrogenated hardstock (B)		Shortening blend, interesterified hardstock (C)		Palm kernel stearin (D)		Coconut oil (E)		Palm oil/palm stearin blend (F)	
Slip melting point		~37 °C		~40 °C		~40 °C		—		—		~45 °C	
Iodine value		—		—		—		~7		—		—	
7	**30 °C**	22,6	22,5	19,7	19,6	24,6	24,5	48,2	48,2	0,0	0,0	15,6	15,6
13	**30 °C**	23,4	23,3	21,2	20,9	26,6	26,3	36,0	36,6	0,3	0,3	17,0	17,0
15	**30 °C**	37,1	38,1	33,8	37,3	40,9	44,6	47,6	47,3	2,5	0,6	30,1	34,3
17	**30 °C**	25,4	25,1	22,1	22,1	28,0	28,2	43,4	43,3	0,0	0,0	17,9	17,8
18	**30 °C**	24,7	24,7	21,3	21,4	26,5	26,7	36,6	37,1	0,2	0,1	16,8	16,8
21	**30 °C**	20,9	20,0	48,6	48,9	50,0	50,5	82,0	82,2	31,0	30,9	41,0	41,4
23	**30 °C**	26,7	26,3	22,4	23,6	28,1	27,8	40,6	40,0	–2,7	–2,3	19,1	19,5
3	**35 °C**	14,0		13,6		18,1		4,2		< 2,0		11,6	
7	**35 °C**	8,4	8,3	10,5	10,6	12,9	13,0	2,6	3,0	0,0	0,0	9,2	9,2
13	**35 °C**	9,4	9,2	12,2	12,3	15,0	14,9	2,9	3,0	0,0	0,2	10,9	10,9
15	**35 °C**	19,3	20,9	23,6	27,0	28,9	30,7	5,6	5,7	2,5	1,6	20,7	23,5
17	**35 °C**	10,6	10,7	13,2	12,9	16,6	26,3	4,3	4,0	0,0	0,0	11,3	11,4
18	**35 °C**	11,6	11,4	13,3	13,5	16,3	16,3	3,7	3,8	0,2	0,3	12,0	12,0
21	**35 °C**	16,3	15,0	1,2	1,2	1,5	2,2	32,7	33,8	1,6	2,0	4,4	4,6
23	**35 °C**	12,4	11,8	13,7	14,9	16,4	16,3	3,7	3,3	–2,1	–1,8	14,6	15,3
3	**40 °C**	3,7		4,8		7,2		< 2,0		< 2,0		9,3	
7	**40 °C**	1,6	1,4	3,9	4,0	3,0	3,1	0,0	0,0	0,0	0,0	5,1	5,3
13	**40 °C**	1,6	1,5	4,6	4,8	5,0	5,2	0,1	0,0	0,0	0,2	6,7	6,6
15	**40 °C**	5,6	6,7	9,9	12,7	13,9	17,6	0,4	1,6	0,9	2,6	11,9	21,9
17	**40 °C**	1,6	1,7	4,9	4,5	5,2	5,4	0,0	0,0	0,0	0,0	6,1	6,0
18	**40 °C**	2,1	2,1	5,6	5,6	6,5	6,4	0,4	0,5	0,1	0,0	8,8	9,0
21	**40 °C**	11,8	10,8	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
23	**40 °C**												
3	**45 °C**											5,9	
7	**45 °C**											2,3	2,4
13	**45 °C**											4,6	4,6
15	**45 °C**												
17	**45 °C**											3,3	3,5
18	**45 °C**											5,1	5,4
21	**45 °C**												
23	**45 °C**												

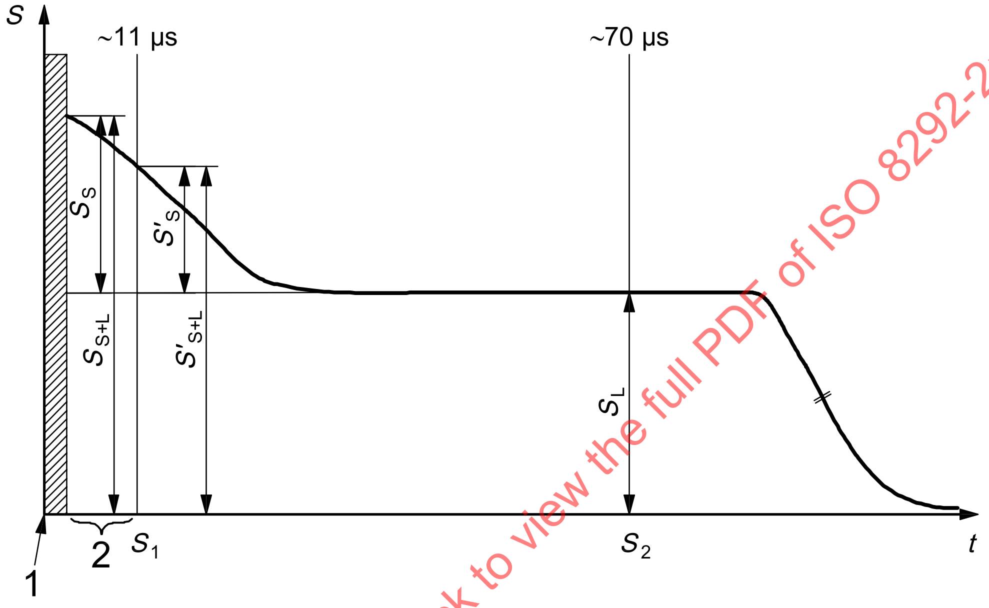
Table A.2 — Results of all laboratories — Measurement protocol 2I

		Palm oil/palm stearin blend (F)		**Cocoa butter, soft Brazilian type (G)**		**Cocoa butter, standard West African type (H)**		**Illipe butter (Borneo tallow, Tengkawang fat) (I)**		**Cocoa butter equivalent, standard type,** $w_{\mathrm{SFC},30}$ ≈ 35 % to 40 % **(J)**		**Palm mid-fraction, hard/CBE grade (K)**	
Slip melting point		~45 °C		—		—		—		—		—	
Iodine value		—		—		—		—		—		~34	
Lab No	**Sample**	**F**	**F**	**G**	**G**	**H**	**H**	**I**	**I**	**J**	**J**	**K**	**K**
3	**10 °C**	—	—										
15	**10 °C**	64,4	64,3										
18	**10 °C**	52,3	51,9										
21	**10 °C**	52,0	51,6										
23	**10 °C**												
3	**20 °C**	25,9	26,9	80,1	80,2	80,0	79,9	94,2	94,9	71,3	71,1	83,3	83,5
15	**20 °C**	36,9	39,1	86,6	86,8	87,6	87,8	96,9	97,0	80,4	79,7	87,6	87,6
18	**20 °C**	25,6	25,4	80,0	79,8	80,3	80,0	91,9	91,8	70,8	71,0	81,3	81,5
21	**20 °C**	27,1	25,9	78,3	78,6	77,7	75,0	91,2	91,3	70,5	69,9	80,6	80,8
23	**20 °C**	25,9	25,9	77,1	76,7	77,1	76,6	91,4	91,6	70,0	70,0	80,6	80,5
3	**25 °C**	24,5	24,5	75,6	76,0	75,6	76,6	93,5	92,8	59,7	59,3	71,9	71,9
15	**25 °C**												
18	**25 °C**	22,4	22,4	73,2	73,0	73,6	73,6	90,0	90,0	56,6	56,8	69,8	69,7
21	**25 °C**	23,9	23,1	72,8	72,8	72,3	72,6	89,7	89,5	56,7	57,2	68,2	68,2
23	**25 °C**	23,1	23,1	72,5	72,0	72,6	72,0	89,9	90,0	57,6	58,0	68,9	68,7
3	**30 °C**	20,8	20,7	49,3	50,4	50,9	51,5	85,3	85,3	33,2	32,5	43,9	43,7
15	**30 °C**	28,7	31,8	52,7	51,3	59,9	60,4	89,8	90,0	30,0	29,9	46,5	45,5
18	**30 °C**	19,4	19,3	46,2	46,1	49,0	49,0	82,6	82,6	28,5	28,7	40,8	40,9
21	**30 °C**	20,9	20,0	48,6	48,9	50,0	50,5	82,0	82,2	31,0	30,9	41,0	41,4
23	**30 °C**	20,0	20,1	46,9	46,4	49,6	48,8	82,5	82,6	30,8	31,6	41,2	40,5
3	**35 °C**	16,0	16,0	< 2,0	< 2,0	< 2,0	< 2,0	36,5	36,5	2,2	2,0	4,7	5,5
15	**35 °C**	21,6	24,4	0,2	0,6	2,2	3,5	39,9	39,7	0,2	4,5	3,3	2,2
18	**35 °C**	14,4	14,2	0,4	0,1	1,5	1,7	37,7	37,1	1,0	1,3	4,4	3,8
21	**35 °C**	16,3	15,0	1,2	1,2	1,5	2,2	32,7	33,8	1,6	2,0	4,4	4,6
23	**35 °C**	14,9	15,0	−1,3	−1,4	−0,1	0,1	36,0	36,0	−0,3	−0,2	2,0	1,8
3	**40 °C**	11,5	11,3	< 2,0	< 2,0	< 2,0	< 2,0	< 2,0	< 2,0	< 2,0	< 2,0	< 2,0	< 2,0
15	**40 °C**	15,5	18,1	1,2	0,5	−0,9	7,6	1,2	2,5	−0,9	2,5	0,2	1,2
18	**40 °C**	10,1	10,1	0,2	0,0	0,0	0,0	0,0	0,0	0,0	0,1	0,0	0,0
21	**40 °C**	11,8	10,8	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
23	**40 °C**												
3	**45 °C**	—	—										
15	**45 °C**												
18	**45 °C**	5,5	5,5										
21	**45 °C**	7,5	6,3										
23	**45 °C**												

Annex B
(informative)

Theory of indirect method

A short radio-frequency pulse is applied which rotates the magnetic field through 90°, i.e. perpendicular to the prevailing magnetic field applied by the permanent magnet. Mainly due to spin-spin relaxation, the magnetization signal in the detector decays over several hundred milliseconds as shown in Figure B.1.



Key

1	pulse
2	dead time
S	magnetization decay signal
S_1	magnetization decay signal measured at about 11 µs
S_2	magnetization decay signal measured at about 70 µs
S_L	magnetization decay signal corresponding to liquid phase after about 70 µs
S_S	magnetization decay signal corresponding to solid phase at time 0
S'_S	magnetization decay signal corresponding to solid phase after about 11 µs
S_{S+L}	magnetization decay signal corresponding to both solid and liquid phases at time 0
S'_{S+L}	magnetization decay signal corresponding to both solid and liquid phases after about 11 µs
t	time

Figure B.1 — Decay of magnetization signal from a fat sample after application of a single 90° radio frequency pulse

The decay of the signal from the protons in the solid state is rapid, occurring over tens of microseconds, whereas the decay of the signal from the protons in the liquid state is much slower, occurring over tens to hundreds of milliseconds. In practice, in a commercial bench-top instrument, the liquid signal will decay within a few milliseconds. By suitable electronics, it should then be possible to measure the solid plus liquid and the liquid signals separately and hence determine the SFC. However, as shown in Figure B.1, the instrument has a dead time after the pulse when no measurements can be made. Thus, the total signal S_2 cannot be measured, but only $S'_{\mathrm{S+L}}$ after about 11 µs. The NMR spectrometer records two signals, S_1 and S_2, at about 11 µs and 70 µs, corresponding to $S'_{\mathrm{S+L}}$ and S_{L}, respectively.

Where the fat is known not to be in the β′-polymorph, e.g. cocoa butter (β) or a salatrim[1] (α), or where its polymorphism is unknown, the direct method may result in significant errors in the measured SFC. To overcome this and to avoid the approximation involved in using the f factor, only the liquid signal, S_2, is measured in the indirect method. The SFC at the measurement temperature, $w_{\mathrm{SFC},T}$, as a percentage mass fraction, is given by Equation (B.1):

$$w_{\mathrm{SFC},T} = \left(1 - \frac{n_{\mathrm{prot,liq},T}}{n_{\mathrm{prot,liq,melt}}}\right) \times 100 \qquad \text{(B.1)}$$

where

$n_{\mathrm{prot,liq},T}$ is the number of protons in the liquid phase at measurement temperature, T;

$n_{\mathrm{prot,liq,melt}}$ is the number of protons in the liquid phase of the fully melted fat.

The measurement of the signal for the fully melted fat must then be corrected for the expansion of the fat from temperature, T, using a reference oil (triolein), which is liquid at all temperatures of measurement. Equation (1) is:

$$w_{\mathrm{SFC},T} = \left(1 - \frac{S_{2,\mathrm{ref},60} \times S_{2,\mathrm{SFC},T}}{S_{2,\mathrm{SFC},60} \times S_{2,\mathrm{ref},T}}\right) \times 100 \qquad (1)$$

where

$S_{2,\mathrm{ref},60}$ is the magnetization decay signal measured at about 70 µs of the triolein reference using a 60 °C water bath or block;

$S_{2,\mathrm{ref},T}$ is the magnetization decay signal measured at about 70 µs of the triolein reference at the measurement temperature, T;

$S_{2,\mathrm{SFC},60}$ is the magnetization decay signal measured at about 70 µs of the test portion using a 60 °C water bath or block;

$S_{2,\mathrm{SFC},T}$ is the magnetization decay signal measured at about 70 µs of the test portion at the measurement temperature, T.

A small error is introduced by assuming that the proton density of the reference and the sample are the same, i.e. that they have the same fatty acid and triacylglycerol compositions, but it is negligible in practice and the indirect method gives essentially an accurate determination of the SFC. Even when the direct method is preferred for its convenience and precision, it may still be necessary to use the indirect method to determine the correct f factor for β-polymorphic fats as explained in ISO 8292-1, 8.3.

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[1] Salatrim = short- and long-chain acyl triglyceride molecules (see Reference [6], p. 251-2).