



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 37517-2019

**Animal and vegetable fats and oils - Determination of solid fat
content by pulsed NMR - Indirect method**

Issued on: May 10, 2019

Implemented on: December 1, 2019

Issued by: SAMR; SAC

Table of Contents

Foreword...	3
1 Scope...	4
2 Normative references...	4
3 Terms and definitions...	4
4 Symbols and abbreviated terms...	4
5 Principle...	5
6 Triolein standard sample...	5
7 Apparatus...	6
8 Sampling...	8
9 Procedure...	8
10 Expression of results...	12
11 Precision...	13
12 Test report...	13
Annex A (Informative) Results of interlaboratory tests...	15
Annex B (Informative) Theory of indirect method...	19
Annex C (Informative) Additional measurement protocols...	22
Bibliography...	24

Foreword

This Standard was drafted in accordance with the rules given in GB/T 1.1-2009.

This Standard is equivalent to ISO 8292-2:2008 Animal and vegetable fats and oils - Determination of solid fat content by pulsed NMR - Indirect method by adopting the translation method.

The documents of our country that have a consistent correspondence with the international documents that are referenced in this Standard are as follows:

- GB/T 15687-2008, Animal and vegetable fats and oils - Preparation of test sample (ISO 661:2003, IDT);
- GB/T 31743-2015, Animal and vegetable fats and oils - Determination of solid fat content by pulsed NMR - Direct method (ISO 8292-1:2008, IDT).

This Standard makes the following editorial changes:

- Change the standard name to "Animal and vegetable fats and oils - Determination of solid fat content by pulsed NMR-Indirect method", to be consistent with the existing standard series;
- Change "The peroxide value determined according to ISO 3960 shall be less than

5 meq/kg." to "The peroxide value determined according to ISO 3960 shall be

less than 2.5 mmol/kg", and perform the unit conversion.

This Standard was proposed by National Food and Strategic Reserves Administration.

This Standard shall be under the jurisdiction of National Technical Committee 270 on Grain and Oil of Standardization Administration of China (SAC/TC 270).

Drafting organizations of this Standard: Academy of State Administration of Grain.

Main drafters of this Standard: Zhang Rui, Xue Yalin, Hao Xicheng.

Animal and vegetable fats and oils - Determination of solid fat content by pulsed NMR - Indirect method

1 Scope

This Standard 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 in this Standard: one for general purpose fats not exhibiting pronounced polymorphism and which stabilize mainly in the beta'-polymorph; and one for fats similar to cocoa butter which exhibit pronounced polymorphism and stabilize in the beta-polymorph. Additional thermal pre-treatments, which may be more suitable for specific purposes, are given in Annex C.

The indirect method is less easy to carry out and less repeatable 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

The following symbols and abbreviated terms apply to this document.

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 mm +/- 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 ~ 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 mm +/- 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 °C +/- 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.

.....