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Butter, edible oil emulsions and spreadable fats — Determination of fat content (Reference method)

Beurre, émulsions d'huile alimentaire et matières grasses tartinables — Détermination de la teneur en matière grasse (Méthode de référence)

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Foreword

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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.

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ISO 17189 | IDF 194 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has the right to be represented on the IDF Standing Committees carrying out the technical work. IDF collaborates with ISO and AOAC International in the development of standard methods of analysis and sampling for milk and milk products.

Draft International Standards adopted by the Action Teams and Standing Committees are circulated to the National Committees for voting. Publication as an International Standard requires approval by at least 50% of IDF National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Action Team, *Fat*, of the Standing Committee on *Main components in milk*, under the aegis of its project leader, Mr J.M. Evers (NZ).

Butter, edible oil emulsions and spreadable fats — Determination of fat content (Reference method)

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this standard to establish safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the fat content of butter, edible oil emulsions (2.2) and spreadable fats (margarine, vegetable oil spreads, dairy spreads and blended spreads).

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
fat content
mass fraction of substances determined by the procedure specified in this International Standard

NOTE The fat content is expressed as a mass fraction in percent.

2.2
edible oil emulsion
high fat product (> 75 % fat) having the same constituents as butter but a composition that does not meet the Codex definition for butter

NOTE Reduced fat butters (e.g. 3/4 fat, 1/2 fat) are considered to belong to the class of spreadable fats.

3 Principle

Fat is extracted from the test portion using a specified solvent. The solvent/fat phase is separated from the aqueous phase and transferred quantitatively to a fat-collecting vessel. The solvent is removed by distillation or evaporation and the mass of substances extracted is determined.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

The reagents shall leave no appreciable residue upon evaporation when the determination is carried out by the method specified (see 8.1.2).

4.1 Extraction solvent, petroleum ether, with any boiling range between 30 °C and 60 °C, or, as equivalent, *n*-hexane [$CH_3(CH_2)_4CH_3$], with a boiling point of 69 °C, both complying with the requirements for the extraction solvent blank test (8.1.2).

4.2 Ethanol (C_2H_5OH), of concentration at least 94 % (volume fraction).

4.3 Congo-red solution

Dissolve 1 g of Congo-red in about 50 ml of water in a 100 ml one-mark volumetric flask. Make up to the mark with water.

WARNING — Take appropriate safety precautions when handling Congo-red solid as this chemical may be a carcinogen.

NOTE The use of this solution, which helps the analyst to better see the interface between the solvent layer and the aqueous layer, is optional (see 8.4.1). Other aqueous colour indicators may be used provided they do not affect the fat result.

5 Apparatus

WARNING — As the determination involves the use of volatile flammable solvents, all electrical apparatus used shall comply with legislation relating to the hazards in using such solvents.

Usual laboratory equipment and, in particular, the following.

5.1 Analytical balance, with a readability of 0,1 mg.

5.2 Drying oven, electrically heated, ventilated, thermostatically controlled, capable of maintaining a temperature of 102 °C ± 2 °C throughout its working space. The oven shall be fitted with a suitable thermometer.

5.3 Desiccator, containing a suitable drying agent, for example, freshly dried silica gel with hygrometric indicator.

If the method is used purely to obtain a routine result, that is, where high accuracy and precision are not required, then the fat-collecting vessels may be cooled to the temperature of the weighing room on the laboratory bench protected from dust.

5.4 Fat-collecting vessels, such as glass boiling flasks with ground necks, of capacity 125 ml, or metal dishes.

When using metal dishes, it is recommended to use dishes with relatively high walls (e.g. 6 cm). This will reduce the risk of fat loss through splashing of the solvent during solvent transfer from the centrifuge tube to the fat-collecting vessel, or through vigorous boiling during the evaporation of the solvent.

5.5 Boiling aids, fat free, of non-porous porcelain or silicon carbide (optional when metal dishes are used).

5.6 Tongs, made of metal, or **cotton gloves**, for holding the fat-collecting vessels (5.4).

5.7 Leak-proof centrifuge tubes, with screw cap, of capacity 50 ml, of plastic which is resistant to the solvent (4.1) for at least the duration of the test.

NOTE Tubes having a large opening (e.g. 25 mm to 35 mm) are preferred to facilitate the addition of the test portion.

5.8 Vortex mixer

5.9 Centrifuge, capable of holding the leak-proof centrifuge tubes (5.7) and capable of producing a radial acceleration of 50 g to 100 g at the outer end of the tubes.

NOTE The use of the centrifuge is optional but recommended (see 8.4.3).

5.10 Automatic pipettor, or other suitable liquid-transfer apparatus (e.g. of capacity 5 ml), for quantitative transfer of the solvent/fat phase.

5.11 Distillation or evaporation apparatus (e.g. steam bath), for distilling or evaporating the solvent from the fat-collecting vessels (see 8.4.8).

5.12 Solvent dispenser or **measuring cylinders**, of capacity 10 ml and 20 ml.

6 Sampling

It is important that the laboratory receive a sample that is truly representative and that 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 707.

The test sample shall be received in an airtight container. Its capacity shall be such that one-half to two-thirds is filled by the sample. Store the samples in the closed container at a temperature of between 5 °C and 14 °C until commencing the preparation of the test sample.

7 Preparation of test sample

7.1 Warm the test sample in the original unopened container to a temperature at which the sample will be soft enough to facilitate thorough mixing to a homogeneous state (either by a mechanical shaker or by hand) without any rupture of the emulsion. The temperature shall normally not exceed 35 °C for samples of butter and edible oil emulsions, or 30 °C in the case of spreadable fat samples.

7.2 Where applicable, cool the test sample to ambient temperature with mixing until cooling is complete. As soon as possible after cooling, open the sample container and stir briefly for no longer than 10 s with a suitable device, e.g. a spoon or spatula, before weighing.

8 Procedure

8.1 Blank tests

8.1.1 Blank test for method

Simultaneously with the determination of the test portion (see 8.4), carry out a blank test using the same procedure for preparation of the fat-collecting vessel (see 8.2), but without weighing of the test portion (see 8.3) and the addition of the Congo-red solution (see 8.4.1) (i.e. add solvents only).

8.1.2 Blank test for extraction solvent

To test the quality of the extraction solvent (4.1), evaporate 60 ml of the solvent from a prepared empty fat-collecting vessel (see 8.2). Additionally, use another prepared empty fat-collecting vessel (see 8.2) for mass control purposes. The extraction solvent shall leave no residue greater than 1,0 mg (see Annex A). Replace or redistil unsatisfactory extraction solvents.

8.2 Preparation of fat-collecting vessel

8.2.1 Dry the empty fat-collecting vessel (5.4) with a few boiling aids (5.5) in the drying oven (5.2) set at 102 °C for at least 30 min.

