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Milk products and milk-based foods — Determination of fat content by the Weibull-Berntrop gravimetric method (Reference method) —

Part 3: Special cases

Produits laitiers et produits à base de lait — Détermination de la teneur en matière grasse par la méthode gravimétrique Weibull-Berntrop (Méthode de référence) —

Partie 3: Cas particuliers

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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 Electro technical Commission (IEC) on all matters of electro technical 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 8262-3│IDF 124-3 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

This edition of ISO 8262-3│IDF 124-3 cancels and replaces ISO 8262-3:1987, of which it constitutes a minor revision.

ISO 8262│IDF 124 consists of the following parts, under the general title *Milk products and milk-based foods — Determination of fat content by the Weibull-Berntrop gravimetric method (Reference method)*:

— *Part 1: Infant foods*

— *Part 2: Edible ices and ice-mixes*

— *Part 3: Special cases*

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 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 the IDF National Committees casting a vote.

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All work was carried out by the Joint ISO-IDF Group of Experts on *Fat determination* (E 31), under the aegis of its chairman, Mr J. Eisses (NL).

This edition of ISO 8262-3 | IDF 124-3 cancels and replaces IDF 126A:1988, of which it constitutes a minor revision.

ISO 8262 | IDF 124 consists of the following parts, under the general title *Milk products and milk-based foods — Determination of fat content by the Weibull-Berntrop gravimetric method (Reference method)*:

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— *Part 3: Special cases*

Introduction

ISO 8262 | IDF 124 has been prepared within the framework of producing a series of reference methods, which are harmonized to the greatest possible extent, for the gravimetric determination of the fat content of milk, milk products and milk-based foods. These methods are based on the Röse-Gottlied (RG) method, or the Weibull-Berntrop (WB) method, or the Schmid-Bondzynski-Ratzlaff (SBR) principle.

For this part of ISO 8262 | IDF 124, dealing with milk-based and with liquid, concentrated or dried milk products in poor condition and/or containing insoluble non-milk ingredients, a method based on the WB principle has been chosen for the following reasons:

a) the RG procedure is not suitable when a distinct quantity of free fatty acids is present, or when the product contains lumps and/or non-milk ingredients insoluble in ammonia, since the extraction of fat is incomplete;

b) the SBR procedure is not suitable owing to a considerable lactose content, which gives rise to some ether-extractable compounds in the digestion with acid and thus gives too high values for the fat content;

c) the WB procedure, although it also includes acid digestion, is not adversely affected by the ether-extractable compounds, since the acid digest is filtered and washed, and the dried residue on the filter does not contain compounds that are extractable by light petroleum;

d) the method described is already used for this purpose in many countries.

The original Weibull method was designed for bread; a considerably modified method, as specified in this International Standard, was developed by Berntrop. This version has found wide application for the determination of fat in many types of food product.

Milk products and milk-based foods — Determination of fat content by the Weibull-Berntrop gravimetric method (Reference method) —

Part 3:
Special cases

1 Scope

This part of ISO 8262 | IDF 124 specifies the reference method for the determination of the fat content of milk-based and of liquid, concentrated or dried milk products to which the Röse-Gottllieb method is not applicable; i.e. those containing distinct quantities of free fatty acids or those which are not completely soluble in ammonia owing to the presence of lumps or non-milk ingredients, such as custards, porridges or certain milk-based products for bakery purposes.

NOTE 1 Reference Röse-Gottlieb methods for the determination of the fat content of milk, of cream, of evaporated and sweetened condensed milk, and of dried milk products are specified in ISO 1211, ISO 2450, ISO 1737 and ISO 1736 respectively.

The method is also applicable to fresh cheese types, such as cottage cheese and quarg, as well as to fresh cheeses with added fruit, syrup, “muesli”, etc. for which the SBR method is not suitable owing to the higher carbohydrate contents and/or extreme inhomogeneity.

NOTE 2 A reference Schmid-Bondzynski-Ratzlaff method for the determination of the fat content of cheese and processed cheese products having lactose contents below 5 % (mass fraction) of the non-fat solids is specified in ISO 1735.

2 Terms and definitions

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

2.1
fat content
all the substances determined by the method specified in this part of ISO 8262 | IDF 124

NOTE It is expressed as a mass fraction in percent.

3 Principle

A test portion is digested by boiling with dilute hydrochloric acid. The hot digest is filtered through a wetted filter paper to retain fatty substances, then the fat is extracted from the dried filter paper using *n*-hexane or light petroleum. The solvent is removed by distillation or evaporation and the substances are extracted and weighed. (This is usually known as the Weibull-Berntrop principle.)

4 Reagents and materials

Use only reagents of recognized analytical grade that leave no appreciable residue when the determination is carried out by the method specified. Use distilled or deionized water, or water of at least equivalent purity.

4.1 Dilute hydrochloric acid, containing approximately 20 % (mass fraction) of HCl, ρ_{20} approximately 1,10 g/ml.

Dilute 100 ml of concentrated hydrochloric acid (ρ_{20} = 1,18 g/ml) with 100 ml of water and mix.

4.2 Extraction solvent, free from water: *n*-hexane or light petroleum having any boiling range between 30 °C and 60 °C.

To test the quality of the extraction solvent, distil 100 ml of it from an extraction flask (5.4) prepared as specified in 7.4. Use an empty extraction flask, prepared in the same way, to check the mass (see 10.1). The solvent shall leave no residue greater than 1,0 mg.

Replace or distil the solvent if it does not meet this requirement.

4.3 Filter papers, of diameter 150 mm, pleated, medium grade, preferably defatted.

To test the quality of the filter paper, carry out a blank test as specified in 7.3, using a solvent satisfying the requirement of 4.2. Use an empty extraction flask (5.4), prepared as specified in 7.4, to check the mass (see 10.1). The paper shall leave no residue greater than 2,5 mg.

Replace unsatisfactory filter papers.

4.4 Blue litmus paper.

4.5 Diatomaceous earth (optional; see 7.5.3).

4.6 Pure lactose (optional; see 7.5.3).

4.7 Cotton wool, defatted by extraction with the solvent (4.2) for 1,5 h and dried.

5 Apparatus

WARNING — Since the determination involves the use of volatile flammable solvents, electrical apparatus employed may be required to comply with legislation relating to the hazards in using such solvents.

Usual laboratory equipment and, in particular, the following.

5.1 Analytical balance.

5.2 Blender, for homogenizing the laboratory sample, if necessary. For example, use a food chopper or a high-speed blender with a blender jar, of capacity 1 litre, fitted with a lid.

5.3 Extraction apparatus, continuous or semi-continuous. For example, use a Soxhlet type, consisting of an extraction flask (flat-bottomed, short-necked) of capacity 150 ml, an extractor with a siphoning volume of 40 ml to 60 ml, and an efficient reflux condenser fitted with a drying tube or plug of cotton wool.

5.4 Extraction flasks, of capacity 150 ml, flat-bottomed and short-necked.

5.5 Extraction thimbles, made of defatted filter paper, glass, alumina or polytetrafluoroethylene (PTFE), contributing no appreciable residue in the blank test, or made of cellulose, single thickness, of internal diameter 22 mm and external length 80 mm, for use with the extraction apparatus (5.3).

5.6 Water baths, capable of being maintained at the following temperatures:

— 40 °C to 60 °C (see 7.1.1);

— 30 °C to 40 °C (see 7.1.2).

5.7 Heating apparatus, for the extraction apparatus. For example, use a water bath, sand bath or a thermostatically controlled hotplate.

5.8 Boiling aids, fat-free, such as glass beads or pieces of non-friable, non-porous porcelain or silicon carbide.

5.9 Conical flask, of capacity 250 ml, fitted with a reflux condenser, preferably of the Liebig type.

5.10 Heating apparatus, for heating a conical flask fitted with a condenser. For example, use a wire gauze and gas burner, an electric hotplate or a sand bath.

5.11 Filter funnel, suitable for use with the pleated filter paper (4.3).

5.12 Beakers with spouts, of capacities 100 ml and 250 ml.

5.13 Distillation apparatus, to enable the solvent to be gently distilled from the flasks at a temperature not exceeding 100 °C.

5.14 Drying oven, electrically heated, with ventilation port(s) fully open, capable of being maintained at a temperature of 102 °C ± 2 °C throughout the working space.

The oven shall be fitted with a suitable thermometer.

5.15 Measuring cylinders, of capacities 50 ml, 100 ml and 250 ml.

5.16 Tongs, made of metal, suitable for holding flasks or beakers.

5.17 Tweezers, flat-tipped, for holding filter papers and thimbles.

6 Sampling

A representative sample should 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 8262 | IDF 124. A recommended sampling method is given in ISO 707 | IDF 50.

All liquid, viscous or pasty laboratory samples shall be kept at a temperature of 2 °C to 4 °C from the time of sampling to the time of commencing the procedure. In the case of a sealed can or bottle, store it unopened at a temperature below 20 °C.

7 Procedure

7.1 Preparation of test sample

7.1.1 Liquid products

Shake and invert the container. Open the container, pour the product slowly into a second container (provided with an airtight lid) and mix by repeated transfer, taking care to incorporate in the sample any fat or other