INTERNATIONAL STANDARD

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IDF 23

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Milk fat products — Determination of water content — Karl Fischer method

Produits à base de matière grasse laitière — Détermination de la teneur en eau — Méthode de Karl Fischer

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 5536 | IDF 23 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 National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Action Team, *Water*, under the aegis of its project leader, Mr G.J. van Beutick (NL).

Introduction

This International Standard describes a manual titration although this technique will now outdated. In principle, three methods of endpoint detection are available: a visual endpoint detection as described in this International Standard; a photometric technique using a photometer for the endpoint detection using the same procedure as described in this International Standard; and an electrical endpoint detection using automated titration apparatus.

A preliminary study is already ongoing to introduce the more modern automated technique into this International Standard and also to include a replacement for the toxic reagents. When the preliminary study has reached its goals, this will lead to revision of this International Standard.

Milk fat products — Determination of water content — Karl Fischer 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 determines the applicability of regulatory limitations prior to use.

1 Scope

This International Standard describes a method for the determination of the water content of milk fat products by the Karl Fischer (KF) method.

The method is applicable to butteroil (anhydrous butteroil, anhydrous butterfat, anhydrous milk fat) with a water content not exceeding 1,0 % by mass.

2 Term and definition

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

2.1
water content of milk fat products
mass fraction of water determined by the procedure specified in this International Standard

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

3 Principle

A test sample is dissolved in a Karl Fischer reagent, then titrated. The sulfur dioxide, methanol and iodine react in the presence of a base to give equivalent amounts of methyl sulfate and iodide only in the presence of water. The water content of the test sample is calculated from the amount of reagent used.

4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity. Avoid absorption of moisture from the environment.

4.1 Karl Fischer reagent, a commercially available pyridine-free, one- or two-component reagent, with strength approximately (4 to 5) mg/ml (on label). Avoid using old stocks of reagent with lower strength.

4.2 Chloroform ($CHCl_3$), free from water.

4.3 Methanol (CH_3OH), with a volume fraction of water of 0,05 % only.

WARNING — Chloroform and methanol are toxic. Take all necessary precautions.

4.4 Chloroform/methanol mixture, in ratio 3:1.

Add to a measuring cylinder (5.4) 3 volumes of chloroform (4.2) and 1 volume of methanol (4.3) and mix.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

All apparatus shall be completely dry when used.

5.1 Analytical balance, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.

5.2 Burette, of capacity 10 ml or 25 ml, provided with a calcium chloride guard tube.

5.3 Conical flasks, of capacity 150 ml, with rubber stoppers or caps, perforated to allow the end of the burette to be fitted.

5.4 Bottles, of brown glass, of capacity 1 l, with ground glass stoppers.

5.5 Measuring cylinders, of suitable capacity for preparing the chloroform/methanol mixture (4.4), with ground glass stoppers, and graduated.

5.6 Pipette, of capacity 25 ml, provided with a suction device.

Alternatively, a 25 ml syringe may be used.

5.7 Titration system.

Alternatively, a commercially available Karl Fischer titration system may be used.

6 Sampling

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

7 Procedure

7.1 Preparation of test sample

Mix the test sample by blending thoroughly at about 50 °C to obtain complete dispersion of the water through the fat.

7.2 Blank test

Perform a blank test before every series of analysis, as the water content of the chloroform/methanol mixture (4.4) is not stable with time.

Use a pipette (5.6) to add 25 ml of the chloroform/methanol mixture (4.4) to a conical flask (5.3). Titrate the mixture with the Karl Fischer reagent (4.1) until a faint iodine colour (yellow-brown) persists for 3 s to 5 s.

NOTE In some automated systems, the presence of traces of water is automatically titrated so that there is no blank value.

7.3 Determination

7.3.1 Titre

Weigh a drop of water (approximately 50 mg), to the nearest 0,1 mg, into a tared 150 ml conical flask (5.3). Immediately stopper the flask. Use a pipette (5.6) to add 25 ml of the chloroform/methanol mixture (4.4) and swirl for 1 min.

Titrate the obtained solution with the Karl Fischer reagent (4.1) until a faint iodine colour (yellow-brown) persists for 3 s to 5 s.

7.3.2 Test portion

Weigh, to the nearest 1 mg, approximately 5 g of the prepared test sample (7.1) into a 150 ml conical flask (5.3).

Preferably, use a syringe to introduce the test sample (7.1) into the 150 ml conical flask, which has been preweighed to the nearest 1 mg. Determine the mass of the thus-introduced test portion by taking the difference in the weighings.

Use a pipette (5.6) to add 25 ml of the chloroform/methanol mixture (4.4) to the test portion. Dissolve the test portion by swirling for 1 min. Titrate with the Karl Fischer reagent (4.1) until a faint iodine colour (yellow-brown) persists for 3 s to 5 s.

Do not perform more than two titrations in one portion of chloroform/methanol mixture (4.4).

An electrometric procedure for the endpoint detection ("dead-stop") may be used.

NOTE For routine determinations using an automated system, the analysis rate can be improved by increasing the volume of solvent and so perform more titrations [e.g. using 40 ml of mixture (4.4) for four titrations].

8 Calculation and expression of results

8.1 Calculation

8.1.1 Calculate the titre, t, of the solution (7.3.1), in grams per millilitre, using the following equation:

$$t = \frac{m}{V_1 - V_b}$$

where

m is the mass, in grams, of the drop of water (7.3.1);

V_1 is the volume, in millilitres, of the Karl Fischer reagent (4.1) used for the titre determination (7.3.1);

V_b is the volume, in millilitres, of the Karl Fischer reagent (4.1) used in the blank test (7.2).

8.1.2 Calculate the water content of the sample, w, as a mass fraction in percent, using the following equation:

$$w = \frac{V_2 - V_b}{a} \times t \times 100\ \%$$

where

V_2 is the numerical value of the volume, in millilitres, of the Karl Fischer reagent (4.1) used for the test portion (7.3.2);

V_b is the numerical value of the volume, in millilitres, of the Karl Fischer reagent (4.1) used in the blank test (7.2);

a is the numerical value of the mass of the test portion (7.3.2), in grams.

8.2 Expression of results

Round the result to two decimal places.

9 Precision

9.1 Interlaboratory test

Details of the interlaboratory test on the precision of the method have been published (see reference [4]). The values for repeatability and reproducibility derived from this interlaboratory test were determined in accordance with ISO 5725-1 and ISO 5725-2.

The values obtained may not be applicable to concentration ranges and matrices other than those given.

NOTE IDF 135 provides specific guidance for interlaboratory tests on methods of analysis for milk and milk products. It is based on ISO 5725.

9.2 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, will in not more than 5 % of cases be greater than:

— for levels $< 0{,}1$ %: 0,015 %;

— for levels $\geqslant 0{,}1$ %: 0,05 %.

9.3 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 equipment, will in not more than 5 % of cases be greater than:

— for levels $< 0{,}1$ %: 0,03 %;

— for levels $\geqslant 0{,}1$ %: 0,10 %.

10 Test report

The test report shall specify:

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

b) the sampling method used, if known;

c) the test method used, with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

e) the test result(s) obtained; and

f) if the repeatability has been checked, the final quoted result obtained.