INTERNATIONAL STANDARD

ISO 6091

IDF 86

Second edition
2010-06-01

Dried milk — Determination of titratable acidity (Reference method)

Lait sec — Détermination de l'acidité titrable (Méthode de référence)

Reference numbers
ISO 6091:2010(E)
IDF 86:2010(E)

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Published in Switzerland

Foreword

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ISO 6091 | IDF 86 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 second edition of ISO 6091 | IDF 86 cancels and replaces the first edition (ISO 6091:1980), of which it constitutes a minor revision.

Foreword

IDF (the International Dairy Federation) is a non-profit organization representing the dairy sector worldwide. IDF membership comprises National Committees in every member country as well as regional dairy associations having signed a formal agreement on cooperation with IDF. All members of IDF have 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.

The main task of Standing Committees is to prepare International Standards. Draft International Standards adopted by the Standing Committees are circulated to the National Committees for endorsement prior to publication as an International Standard. Publication as an International Standard requires approval by at least 50% of IDF National Committees casting a vote.

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ISO 6091 | IDF 86 was prepared by the International Dairy Federation (IDF) and Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by IDF and ISO.

All work was carried out by the former Joint ISO-IDF Action Team on *Carbohydrate and lactate determination*, now part of the Standing Committee on *Analytical methods for composition*.

This edition of ISO 6091 | IDF 86 cancels and replaces IDF 86:1981, of which it constitutes a minor revision.

Dried milk — Determination of titratable acidity (Reference method)

1 Scope

This International Standard specifies a reference method for the determination of the titratable acidity of all types of dried milk.

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 1736|IDF 9, *Dried milk and dried milk products — Determination of fat content — Gravimetric method (Reference method)*

ISO 5537|IDF 26, *Dried milk — Determination of moisture content (Reference method)*

3 Terms and definitions

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

3.1
titratable acidity of dried milk
volume of 0,1 mol/l sodium hydroxide solution required to titrate a quantity of the reconstituted milk corresponding to 10 g of solids-not-fat to pH 8,40

NOTE Titratable acidity is expressed in millilitres.

4 Principle

Milk is reconstituted by addition of water to a test portion of dried milk corresponding accurately to 5 g of solids-not-fat. The reconstituted milk is titrated with 0,1 mol/l sodium hydroxide solution to pH 8,40. The number of millilitres used in the titration is multiplied by 2, in order to obtain the number of millilitres in terms of 10 g of solids-not-fat.

The amount of sodium hydroxide solution required is a function of the amount of natural buffering substances present in the product, and of developed or added acid or alkaline substances.

5 Reagents and materials

Unless otherwise stated, use only reagents of recognized analytical grade and distilled or demineralized water or water of equivalent purity, freed from carbon dioxide by boiling for 10 min before use.

5.1 Sodium hydroxide, standard volumetric solution, c(NaOH) = 0,1 mol/l ± 0,000 2 mol/l, carbonate free.

Protect this solution against absorption of carbon dioxide.

5.2 Nitrogen.

6 Apparatus

Usual laboratory equipment and in particular the following.

6.1 Analytical balance.

6.2 pH-meter, with slope control, accurate to 0,01 pH unit, with a glass measuring electrode and a suitable reference electrode, calibrated using two buffer solutions of approximately pH 7 and pH 9, respectively, known to within ± 0,01 pH unit.

6.3 Magnetic stirrer.

6.4 Burette, graduated in 0,1 ml and with an accuracy of 0,05 ml, ISO 385[1] class A.

6.5 Measuring cylinder, capacity 50 ml, ISO 4788[3] class A.

6.6 Conical flask, capacity 100 ml or 150 ml, with a ground neck and ground-glass stopper. The neck shall be sufficiently wide to accommodate the two electrodes, the burette tip and the nitrogen line.

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707|IDF 50[2].

It is important that the laboratory receive a truly representative sample which has not been damaged or changed during transport or storage.

8 Preparation of test sample

Transfer the sample to a clean, dry container (provided with an airtight lid) that has a capacity of about twice the volume of the sample.

Close the container immediately and thoroughly mix the contents by repeatedly shaking and inverting the container. During these operations, exposure of the sample to the atmosphere should be avoided as far as possible to minimize absorption of water.

9 Procedure

9.1 Test portion

Weigh (500/w) ± 0,01 g of the test sample (see Clause 8) into the conical flask (6.6), w being the solids-not-fat content of the sample, expressed as a percentage mass fraction.

The solids-not-fat content of the sample may be calculated by subtracting the fat content, determined in accordance with ISO 1736|IDF 9, and the moisture content, determined in accordance with ISO 5537|IDF 26, from 100.

9.2 Determination

9.2.1 Reconstitute milk by adding 50 ml of water at about 20 °C to the test portion (see 9.1) and agitating vigorously. Allow to stand for about 20 min.

9.2.2 Titrate the contents of the conical flask by adding the sodium hydroxide solution (5.1) from the burette (6.4) until the pH-meter (6.2) measurement persists at pH 8,40 for about 5 s.

During the titration, the solution should be agitated using the magnetic stirrer (6.3), and absorption of carbon dioxide from the air should be avoided by flushing the conical flask with nitrogen (5.2). The titration should be completed within 1 min.

Record the volume, in millilitres, of sodium hydroxide solution used, to the nearest 0,05 ml.

10 Calculation

The titratable acidity, V_{ta}, is given by the equation

$$V_{ta} = 2V$$

where V is the volume, in millilitres, of the sodium hydroxide solution (5.1) used for the titration (see 9.2.2).

Express the result to one decimal place.

11 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst will not exceed 0,4 ml of 0,1 mol/l sodium hydroxide solution per 10 g of solids-not-fat.

12 Test report

The test report shall contain at least the following information:

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

b) the sampling method used, if known;

c) the test method used, including a reference to this International Standard (ISO 6091|IDF 86:2010);

d) any operating conditions not specified in this International Standard, or regarded as optional, as well as details of any incidents that may have influenced the result(s);

e) the test result(s) obtained;

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

Bibliography

[1] ISO 385, *Laboratory glassware — Burettes*

[2] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*

[3] ISO 4788, *Laboratory glassware — Graduated measuring cylinders*

ICS 67.100.10

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