TECHNICAL SPECIFICATION

# ISO/TS 11869

# IDF/RM 150

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# Fermented milks — Determination of titratable acidity — Potentiometric method

*Laits fermentés — Détermination de l'acidité titrable — Méthode potentiométrique*



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# Foreword

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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.

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An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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/TS 11869|IDF/RM 150 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 first edition cancels and replaces ISO 11869:1997, which has been technically revised.

# 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.

In other circumstances, particularly when there is an urgent market requirement for such documents, a Standing Committee may decide to publish an other type of normative document which is called by IDF: *Reviewed method*. Such a method represents an agreement between the members of a Standing Committee and is accepted for publication if it is approved by at least 50 % of each National Committee representative present casting a vote. A *Reviewed method* is equal to an ISO/PAS or ISO/TS and will, therefore, also be published jointly under ISO conditions.

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All work was carried out by the Joint ISO-IDF Project Group on *Titratable acidity* of the Standing Committee on *Analytical methods for composition* under the aegis of its project leader, Dr. J. Floor (ZA).

This edition of ISO/TS 11869|IDF/RM 150 cancels and replaces IDF 150:1991, of which it constitutes a technical revision.

# Fermented milks — Determination of titratable acidity — Potentiometric method

## 1 Scope

This Technical Specification specifies a potentiometric method for the determination of the titratable acidity of natural yoghurt, flavoured yoghurt, fruit yoghurt, drinking yoghurt, fresh cheese with or without fruit, buttermilk with or without fruit, and other fermented milk products.

## 2 Terms and definitions

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

**2.1**
**titratable acidity of fermented milks**
amount in millilitres of a 0,1 mol/l sodium hydroxide solution required to titrate 10 g of product to pH 8,30 ± 0,01



NOTE The titratable acidity is expressed in millimoles per 100 g.



## 3 Principle

A test portion is suspended in water. The suspension is titrated potentiometrically against sodium hydroxide solution [$c$(NaOH) = 0,1 mol/l] to pH 8,30 ± 0,01. The titratable acidity is calculated.

## 4 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and only distilled or deionized water, freed from carbon dioxide by boiling for 10 min before use.

**4.1 Sodium hydroxide**, standard volumetric solution, $c$(NaOH) = 0,1 mol/l ± 0,002 mol/l, carbonate free.

Protect this solution against absorption of carbon dioxide ($CO_2$) either by connecting a washing bottle with 10 % sodium hydroxide solution to the burette which itself contains the sodium hydroxide solution or by connecting a small tube with fresh sodium hydroxide or calcium oxide to the end of the burette to obtain a closed system.

NOTE $CO_2$ is either bound in the washing bottle or in the tube to protect the solution in the burette against absorption which would influence the concentration.

## 5 Apparatus

Usual laboratory equipment and, in particular, the following.

**5.1 Analytical balance**, capable of being read to the nearest 0,01 g.

**5.2 pH meter**, correctly calibrated in the range pH 7 to 10 following normal laboratory pH-calibration procedures.

**5.3 Spoon** or **spatula**.

**5.4 Homogenizer**, e.g. a macerator [Ultra-Turrax[1)] or equivalent].

**5.5 Burette**, of capacity 25 ml or 50 ml, graduated at least at every 0,05 ml, ISO 385[1], class A. Alternatively, an **automatic burette**, ISO 8655-3[4], fulfilling the same requirements may be used.

NOTE Instead of carrying out a manual titration, it is also possible to use an automatic titrator.

**5.6 Water bath**, capable of maintaining a temperature of 38 °C ± 1 °C.

## 6 Sampling

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



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



## 7 Preparation of test sample

### 7.1 Natural yoghurt, flavoured yoghurt, drinking yoghurt and other fermented milks

Bring the sample to a temperature of 22 °C ± 2 °C. Mix the sample carefully by means of a spoon or spatula (5.3) or homogenizer (5.4), using a rotary motion which passes from the lower layers to the surface layers of the sample so as to displace and mix them well.

### 7.2 Fruit yoghurt and other fermented milk products with added fruit

Bring the sample to a temperature of 22 °C ± 2 °C. Homogenize it using an appropriate device (5.4), in order to facilitate the grinding and dispersion of the fruit.

If fat separation is observed in the sample, raise the temperature of the sample in the water bath (5.6) to 38 °C ± 1 °C for a better homogenization. Thereafter, cool the sample again to a temperature of 22 °C ± 2 °C.

1) Ultra-Turrax is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO or IDF of this product.

# 8 Procedure

## 8.1 Test portion

Weigh, in a 50 ml beaker, approximately 10 g of prepared test sample (Clause 7), to the nearest 0,01 g. Add approximately 10 ml of water and mix.

## 8.2 Determination

**8.2.1** Insert the electrode of the pH meter (5.2) into the suspension (8.1).

**8.2.2** Use the sodium hydroxide solution (4.1), while stirring, to titrate the contents of the beaker until the pH remains stable at 8,30 ± 0,01 for 4 s to 5 s.

If an automatic titrator is used, it shall provide for such a stop delay time as well.

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

# 9 Calculation and expression of results

## 9.1 Calculation

Calculate the titratable acidity, $I$, in millimoles of sodium hydroxide per 100 g, using the following equation:



$$I = \frac{V \times 10}{m}$$



where

$V$ is the volume, in millilitres, of the sodium hydroxide solution (4.1) used for the titration (8.2.2);

$m$ is the mass, in grams, of the test portion (8.1).

NOTE For the expression of results in grams of lactic acid per 100 g of product, see Annex A.

## 9.2 Expression of test results

Express the test results to two decimal places.

# 10 Precision

## 10.1 Interlaboratory test

The method has been tested on various fermented milk products in six different laboratories. Each laboratory obtained the products locally. See Reference [5]. A summary of the results is given in Annex B. The value for repeatability derived from this interlaboratory study was calculated in accordance with ISO 5725-1[3].

## 10.2 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 0,20 mmol/100 g.

## 11 Test report

The test report shall contain at least the following information:

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

b) the sampling method used, if known;

c) the method used, with reference to this Technical Specification (ISO/TS 11869|IDF/RM 150:2012);

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

e) the test result(s) obtained;

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

# Annex A
(informative)

## Alternative expression of test results

In the previous version of this method, test results were expressed in grams of lactic acid per 100 g of product, $w$. This is slightly misleading as the method is not a procedure for the quantitative determination of lactic acid. However, if expression of the results in this way is preferred, the formula for the calculation of results is as follows:

$$w = \frac{V \times 0{,}9}{m}$$

where

$V$ is the volume, in millilitres, of the sodium hydroxide solution (4.1) used for the titration (8.2.2);

$m$ is the mass, in grams, of the test portion (8.1).