TECHNICAL SPECIFICATION

ISO/TS 19046-2

IDF/RM 233-2

First edition
2017-05

Cheese — Determination of propionic acid level by chromatography —

Part 2: Method by ion exchange chromatography

Fromages — Détermination de la teneur en acide propionique par chromatographie —
Partie 2: Méthode par chromatographie par échange d'ions

Reference numbers
ISO/TS 19046-2:2017(E)
IDF/RM 233-2:2017(E)

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Forewords

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This document 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.

A list of all parts in the ISO/TS 19046 | IDF/RM 233 series can be found on the ISO website.

IDF (the International Dairy Federation) is a non-profit private sector organization representing the interests of various stakeholders in dairying at the global level. IDF members are organized in National Committees, which are national associations composed of representatives of dairy-related national interest groups including dairy farmers, dairy processing industry, dairy suppliers, academics and governments/food control authorities.

ISO and IDF collaborate closely on all matters of standardization relating to methods of analysis and sampling for milk and milk products. Since 2001, ISO and IDF jointly publish their International Standards using the logos and reference numbers of both organizations.

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ISO/TS 19046-2 | IDF/RM 233-2 was prepared by the IDF Standing Committee on *Analytical Methods for Composition* and ISO Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products.*

The IDF Reviewed method is equal to an ISO Publicly Available Specification (ISO/PAS) or an ISO Technical Specification (ISO/TS) and is therefore published jointly under ISO conditions.

The work was carried out by the IDF/ISO Project Group on Propionic acid (C25) of the Standing Committee on *Analytical Methods for Composition* under the aegis of its project leader P. Trossat (FR).

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Cheese — Determination of propionic acid level by chromatography —

Part 2:
Method by ion exchange chromatography

WARNING — This document can involve the use of products and implementation of procedures and equipment of a hazardous nature. This document does not aim to address all the risks related to its use. It is the responsibility of the user of this document to establish appropriate hygiene and safety practices before using it, and to determine the applicability of any other restrictions.

1 Scope

This document specifies a method for the determination of propionic acid level in cheese, using ion exchange chromatography.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
level of propionic acid
mass fraction of propionic acid determined following the procedure described in this document

Note 1 to entry: The level of propionic acid is expressed in mg/100 g of cheese.

4 Principle

Aqueous extraction of propionic acid by dilution of samples in the mobile phase, centrifugation of the solution obtained, and filtration and assay of the propionic acid in the filtrate by ion exchange chromatography.

5 Reagents

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

5.1 Propionic acid [$CH_3\ CH_2COOH$] of purity ≥ 99,5 % mass fraction.

5.2 Sulfuric acid [H_2SO_4] containing a mass/volume fraction of 98 %.

5.3 Water, high performance liquid chromatography (HPLC) grade.

5.4 Mobile phase

Weigh exactly 10,1 g of sulfuric acid (5.2) and transfer into a 5 000 ml flask (6.3) already containing 2 l of water (5.3), make up to the mark with water (5.3) and filter using a vacuum filtration system (6.7).

5.5 Standard solutions

5.5.1 Standard stock solution of propionic acid

Weigh exactly 200 mg of propionic acid (5.1) in a 100 ml one mark volumetric flask (6.3). Dilute to the mark with the mobile phase (5.4) and filter standard stock solution using a vacuum filtration system (6.7).

5.5.2 Standard solutions of propionic acid

Prepare two additional standard solutions by diluting standard stock solution (5.5.1) in mobile phase (5.4).

— 25 ml of standard stock solution in a 50 ml one mark volumetric flask (6.3). Dilute to the mark with the mobile phase (5.4).

— 25 ml of standard stock solution in a 100 ml one mark volumetric flask (6.3). Dilute to the mark with the mobile phase (5.4).

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Grinding or **grating device**.

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

6.3 Volumetric flasks, of capacity 50 ml, 100 ml and 5 000 ml.

6.4 Single-use syringes, 1 ml capacity equipped with a 0,2 µm polyvinylidene fluoride (PVDF) syringe filter.

6.5 Hot plate stirrer.

6.6 Laboratory centrifuge, capable to produce a centrifugal acceleration of approximately 13 000*g*.

6.7 Vacuum filtration system, equipped with a 0,2 µm filtration membrane.

6.8 HPLC system, with the following equipment.

6.8.1 Column oven, set at 50 °C.

6.8.2 Ion exchange column, Aminex HPX 87 H BIO RAD[1)] (column size: 300 mm × 7,8 mm, particle size: 9 µm) equipped with a Carbo H BIO RAD precolumn or equivalent.

1) Aminex HPX 87 H 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.

6.8.3 Chromatographic conditions

— Mobile phase: H_2SO_4 0,02 mol/l (5.4) kept under helium at 20 ml/min;

— flow rate: 0,6 ml/min;

— quantity injected: 10 µl;

— rinse under H_2SO_4 0,02 mol/l, inverted column, for 4 h (needed after each analytical series).

An example of the HPLC chromatogram profile obtained with these conditions is shown in Annex A.

6.8.4 UV detector, set at 210 nm.

6.8.5 Injection syringe or **injection system**, capable to inject 10 µl.

6.8.6 Integration system, preferably being computerized.

7 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 707 | IDF 50.

It is important that the laboratory receives a sample that is representative and has not been damaged or changed during transport or storage.

8 Preparation of test sample

Prior to analysis, grind or grate the test sample by using an appropriate grinding or grating device (6.1), if necessary, after removing the rind, the smear or the mouldy surface layer of the cheese.

Weigh, to the nearest 0,1 mg, about 5 g to 10 g of cheese in a glass flask and record the mass m_1. Add around 35 ml of mobile phase (5.4) to the test sample.

— Mix with a magnetic stirrer (6.5) for 1 h at 70 °C.

— Cool down the solution and transfer into a 50 ml one mark volumetric flask. Dilute to the mark with the mobile phase (5.4).

Filter the solution using a 0,2 µm syringe filter (6.4).

NOTE A centrifugation at 13 000*g* for 10 min can be applied if difficulties are met with the direct filtration.

9 Quantitative determination by HPLC

9.1 Calibration

Inject 10 µl of the three standard solutions (5.5) into the HPLC system (6.8) applying chromatographic conditions described in 6.8.3 and calibrate according the manufacturer requirements.

9.2 Sample test

Inject 10 µl of the test sample prepared as specified in Clause 8 into the HPLC system (6.8) applying the chromatographic conditions described in 6.8.3.