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**Cheese — Determination of propionic  
acid level by chromatography —**

**Part 1:  
Method by gas chromatography**

*Fromages — Détermination de la teneur en acide propionique par  
chromatographie —*

**iTeh STANDARD PREVIEW**  
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## Forewords

**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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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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 19046-1 | IDF/RM 233-1 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 1: Method by gas 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 gas 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

Preparation of the test sample by addition of the internal standard and homogenization in the presence of sulfuric acid. Continuous extraction of the mixture in a liquid-liquid extractor by a mixture of ethers. Separation of the volatile fatty acids from the fatty phase in the form of their sodium salts (soaps) after neutralization in the presence of phenolphthalein (or other equivalent indicator) and drying of the soaps recovered in the aqueous phase. Separation of the propionic acid using gas chromatography and quantification by reference to an internal standard.

### 5 Reagents

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

- 5.1 **Valeric acid (internal standard)**  $[\text{CH}_3(\text{CH}_2)_3\text{COOH}]$  of purity  $\geq 99\%$  mass fraction.
- 5.2 **Sulfuric acid solution**  $[\text{H}_2\text{SO}_4]$  containing a mass/volume fraction of 10 %.
- 5.3 **Petroleum ether**, with any boiling range between 45°C and 65°C.
- 5.4 **Diethyl ether**  $[\text{C}_2\text{H}_5\text{O C}_2\text{H}_5]$ .
- 5.5 **Mixed solvent**, prepared shortly before use by mixing equal volumes of diethyl ether (5.4) and petroleum ether (5.3).
- 5.6 **Ethanol**  $[\text{C}_2\text{H}_5\text{OH}]$  containing a volume fraction of ethanol of approximately 77 %.
- Into a 50 ml volumetric flask, add 40 ml  $\pm$  2 ml of ethanol 96 % not denaturated and make up to the mark with water.
- 5.7 **Sodium hydroxide**,  $c(\text{NaOH})$  around 1 mol/l.
- 5.8 **Indicator solution**. Phenolphthalein solution  $[\text{C}_{20}\text{H}_{14}\text{O}_4]$  containing a mass fraction of 1 % (in ethanol 95 % to 96 %) or other indicator with equivalent turning zone.
- 5.9 **Trifluoroacetic acid**  $[\text{CF}_3\text{COOH}]$  dissolved in ethanol 95 % to 96 % (6,67 % in volume fraction).
- 5.10 **Propionic acid**  $[\text{CH}_3 \text{CH}_2\text{COOH}]$  of purity  $\geq 99,5\%$  mass fraction.

5.11 **Calibration solution.**

Weigh to the nearest 0,1 mg about 400 mg of propionic acid (5.10) and about 100 mg of valeric acid (5.1). Dissolve up to 100 g with ethanol (5.6).

## 6 Apparatus

**WARNING — The determination involves the use of volatile flammable solvents. All electrical apparatus employed shall be safe relating to the hazards in using such solvents.**

Usual laboratory equipment and, in particular, the following.

- 6.1 **Grinding or grating device.**
- 6.2 **Homogenizer/mixer** (e.g. Ultraturrax type 251<sup>1)</sup>), equipped with a rotor.
- 6.3 **Mixer type vortex.**
- 6.4 **Soxhlet-type extractor apparatus** (for light solvents), equipped with flask heater.
- See [Annex A](#).
- 6.5 **Separating funnel**, of capacity 500 ml.
- 6.6 **Rotary evaporator and controlled vacuum device.**

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1) Ultraturrax type T251 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.



- 6.7 Drying oven**, capable of maintaining at a temperature around 60 °C.
- 6.8 Analytical balance**, capable of weighing to the nearest 1 mg, with a readability of 0,1 mg.
- 6.9 Measuring cylinders**, of capacity 50 ml and 100 ml.
- 6.10 Volumetric flasks**, of capacity 50 ml.
- 6.11 Glass flasks**, of capacity around 125 ml.
- 6.12 Glass flasks**, of capacity around 250 ml.
- 6.13 Dispensers**, of capacity 2,5 ml and 10 ml.
- 6.14 Gas chromatograph**, with the following equipment.
- 6.14.1 Carrier gas**, hydrogen, helium or nitrogen, purity  $\geq 99,999\ 7\ \%$ .
- 6.14.2 Semi-capillary column**, 30 m length, 0,53 mm internal diameter, 2  $\mu\text{m}$  film thickness, with a modified polyethylene glycols polar stationary phase [phase free fatty acid phase (FFAP), Carbowax<sup>TM2</sup>] or equivalent.
- 6.14.3 Gas chromatographic conditions.**
- The following conditions have been found suitable to obtain a correct separation of propionic acid:
- carrier gas flow: 7 ml/min; [ISO/TS 19046-1:2017](https://standards.iteh.ai/catalog/standards/sist/7a13037c-320a-4e0f-8540-100000000000/iso-ts-19046-1-2017)
  - oven temperature program: initial temperature of 80 °C, maintained for 0,5 min, raised at a rate of 20 °C min<sup>-1</sup> up to 170 °C, maintained at this temperature for 6,5 min.
- An example of a GC profile obtained with these conditions is shown in [Annex B](#).
- 6.14.4 Flame ionization detector**, set at a temperature of 250 °C.
- 6.14.5 On column injector**, set at a temperature of 250 °C.
- 6.14.6 Injection syringe**, of capacity 10  $\mu\text{l}$ .
- 6.14.7 Integration system**, preferably being computerized.
- 6.15 Glass tubes** (with stoppers).

## 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.

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2) Carbowax<sup>TM</sup> 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.