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Milk — Determination of freezing point — Thermistor cryoscope method (Reference method)

Lait — Détermination du point de congélation — Méthode au cryoscope à thermistance (Méthode de référence)

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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.

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.

International Standard ISO 5764 | IDF 108 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.

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This third edition of ISO 5764 | IDF 108 cancels and replaces the second edition (ISO 5764 | IDF 108:2002), the scope of which has been technically revised.

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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.

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 the IDF National Committees casting a vote.

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All work was carried out by the Joint IDF-ISO Action Team on *Water*, of the Standing Committee on *Main components in milk*, under the aegis of its project leader, Mrs. S. Orlandini (IT).

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Milk — Determination of freezing point — Thermistor cryoscope method (Reference method)

1 Scope

This International Standard specifies a reference method for the determination of the freezing point of raw bovine milk, heat-treated whole, reduced fat and skimmed bovine milk, as well as raw ovine and caprine milk, by using a thermistor cryoscope.

The freezing point can be used to estimate the proportion of extraneous water in milk. Calculation of the amount of extraneous water is subject to daily and seasonal variations, and is not within the scope of this International Standard.

Results obtained from samples with a titratable acidity exceeding 20 ml of 0,1 mol/l sodium hydroxide solution per 10 g of non-fat solids are not representative of the original milk.

NOTE 1 Sterilization and vacuum pasteurization can affect the freezing point of milk (see Reference [5]).

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NOTE 2 The method uses plateau-timed instruments. For routine measurements, other thermistor cryoscope methods, i.e. fixed time procedures, can be used. Guidelines for the application of other procedures are given in Annex C.

NOTE 3 The limit value mentioned for the titratable acidity in Clause 1 and 9.2 applies to bovine milk. It is possible that the limit values for ovine and caprine milk are higher.

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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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 6091, *Dried milk — Determination of titratable acidity (Reference method)* [1)]

3 Terms and definitions

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

3.1
freezing point of milk
temperature value obtained using the method specified in this International Standard

NOTE The freezing point is expressed in millidegrees Celsius.

1) Equivalent to IDF 86.

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4 Principle

A test sample of milk is supercooled to an appropriate temperature. Crystallization is induced by means sufficient to cause an instantaneous release of heat with an accompanying warming of the sample to a temperature plateau. The plateau is reached when the temperature rise has not exceeded 0,5 m°C over the previous 20 s. The temperature thus attained corresponds to the freezing point of the test sample.

The instrument is calibrated by adjusting it to give the correct readings for two sodium chloride standard solutions, using the same procedure as for test portions of milk.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade.

5.1 Water, in accordance with ISO 3696 grade 2 or water of equivalent quality distilled from borosilicate glass apparatus.

Boil the water and cool it to 20 °C ± 2 °C shortly before use.

5.2 Sodium chloride (NaCl), finely ground, dried in the electric furnace (6.7) at 300 °C ± 25 °C for 5 h or, alternatively, dried in the drying oven (6.8) at 130 °C ± 2 °C for at least 24 h, then cooled to room temperature in a desiccator (6.9).

5.3 Sodium chloride standard solutions.

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Preferably, make up the sodium chloride standard solution on a gram per kilogram basis (see Table 1, leftmost column) by weighing the required amount of prepared dry sodium chloride (5.2) to the nearest 0,1 mg and dissolving it in 1 000 g ± 0,1 g of water (5.1). Store standard solutions at about 5 °C in well-stoppered polyethylene bottles (6.10) of capacity not greater than 250 ml.

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Alternatively, weigh, to the nearest 0,1 mg, the appropriate amount (see Table 1, middle column) of prepared dry sodium chloride (5.2) in a weighing bottle (6.5). Dissolve in water (5.1) and transfer quantitatively to a 1 000 ml one-mark volumetric flask (6.6). Dilute to the 1 000 ml mark with water (5.1) at 20 °C ± 2 °C and mix.

Table 1 — Freezing point of sodium chloride standard solutions

NaCl solution g/kg	NaCl solution at 20 °C g/l	Freezing point m°C
6,763	6,731	−400,0
6,901	6,868	−408,0
7,625	7,587	−450,0
8,489	8,444	−500,0
8,662	8,615	−510,0
8,697	8,650	−512,0
8,835	8,787	−520,0
9,008	8,959	−530,0
9,181	9,130	−540,0
9,354	9,302	−550,0
9,475	9,422	−557,0
10,220	10,161	−600,0

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Before using a standard solution, gently invert and rotate the bottle several times to mix its contents thoroughly.

Do not agitate the standard solution violently at any time, as this can lead to incorporation of air. Pour samples of standard solutions from the bottles; never use pipettes for this purpose. Do not use standard solutions from bottles less than one-quarter full, or more than 2 months old, or containing visible moulds.

Only use unpreserved sodium chloride standard solutions for this reference method. For routine methods, sodium chloride standard solutions with a fungicidal or fungistatic agent may be used. For guidance, see Annex C.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Cryoscope, consisting of a thermostatically controlled cooling device, a thermistor probe with associated circuit, a read-out device, a sample agitator and a crystallization device (see Figure 1).

6.1.1 Cooling device.

Several types of thermostatically controlled cooling devices can be used, e.g.:

a) immersion type: a cooling bath with a suitable buffer capacity;

b) circulation type: a continuous stream of cooling liquid around the sample tube;

c) cooling block type: a cooling block with a small amount of cooling liquid.

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After the initiation of freezing, keep the temperature of the cooling liquid around the sample tube constant at −7,0 °C ± 0,5 °C.

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NOTE A suitable cooling liquid is a 33 % (volume fraction) aqueous solution of propylene glycol.

6.1.2 Measuring device, associated circuitry and read-out device**.**

The thermistor shall be of the glass probe type with diameter of 1,60 mm ± 0,4 mm and an electrical resistance of between 3 Ω and 30 kΩ at 0 °C.

The type and dimensions of the shank material (including a possible filler) shall not allow a heat transfer into the sample greater than $2{,}5 \times 10^{-3}$ J/s, under operating conditions.

When the probe is in measurement position, the thermistor bead shall lie on the axis of the sample tube and at equal distances from the inner walls and the inner bottom of the tube (see Figure 1).

The thermistor and the associated circuitry shall show a discrimination of 1 m°C or better over a range of −400 m°C to −600 m°C.

The linearity of the circuit shall be such that no error greater than 1 m°C is introduced at any point within the range of −400 m°C to −600 m°C when the instrument is correctly operated.

The read-out device shall provide a discrimination of 1 m°C or better over a range of at least 0 m°C to −1 000 m°C.

6.1.3 Stirring wire, inert to milk, used to stir the test portion during cooling.

The stirring wire shall be adjustable for amplitude and mounted vertically in accordance with the manufacturer's instructions. The wire shall vibrate laterally with an amplitude of 2 mm to 3 mm to ensure that the temperature within the test portion remains uniform during cooling. At no time during its normal stirring operation shall the wire strike the glass probe or the wall of the tube.

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6.1.4 Device for initiating freezing, that, when operated, instantaneously initiates freezing of the test portion when reaching −3,0 °C.

The stirring wire (6.1.3) may be used for this purpose. One method is to increase the amplitude of vibration for 1 s to 2 s such that the stirring wire strikes the wall of the sample tube (6.2).

6.2 Sample tubes, symmetrical, made of borosilicate glass, of length 50,5 mm ± 0,2 mm, of external diameter 16,0 mm ± 0,2 mm and of internal diameter 13,7 mm ± 0,3 mm (see Figure 1).

The wall thickness throughout the tube shall not vary by more than 0,1 mm.

The tubes shall be equally shaped so that equal freezing points are obtained for equal volumes of the same solution. Check on equality before using the tubes.

6.3 Main power supply, capable of operating within the manufacturer's specifications.

6.4 Analytical balance, capable of weighing to the nearest 0,1 mg.

6.5 Weighing bottle.

6.6 One-mark volumetric flasks, capacity 1 000 ml, ISO 1042 [2] class A.

6.7 Electric furnace, capable of being maintained at 300 °C ± 25 °C; or

6.8 Drying oven, capable of being maintained at 130 °C ± 2 °C.

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6.9 Desiccator, containing silica gel with hygrometer indicator.

6.10 Polyethylene bottles, of maximum capacity 250 ml, with a suitable stopper.

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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 [1].

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

Preferably, test samples immediately upon arrival at the laboratory. Provided it remains in a representative state, a test sample of raw milk may be stored at a temperature of between 0 °C and 6 °C for up to 48 h after sampling. For a test sample of processed milk, respect its shelf life.

8 Calibration of the thermistor cryoscope

Ensure that the cryoscope (6.1) is in working condition in accordance with the manufacturer's instructions. Check the position of the probe, the amplitude of vibration of the stirring wire, and the temperature of the cooling device (6.1.1).

Select two sodium chloride standard solutions (see Table 1) which closely bracket the expected value of the freezing point of the milk to be tested. The difference in the freezing points between the two selected sodium chloride standard solutions shall not be less than 100 m°C. Ensure that the temperatures of the selected sodium chloride standard solutions and that of the test sample are similar.

Pour 2,5 ml ± 0,1 ml of the sodium chloride standard solutions into clean, dry sample tubes (6.2) and calibrate the instrument as indicated by the manufacturer. Use sample tubes (6.2) of the same type as those being used during testing of the sample. Thereafter, the thermistor cryoscope is ready for use.

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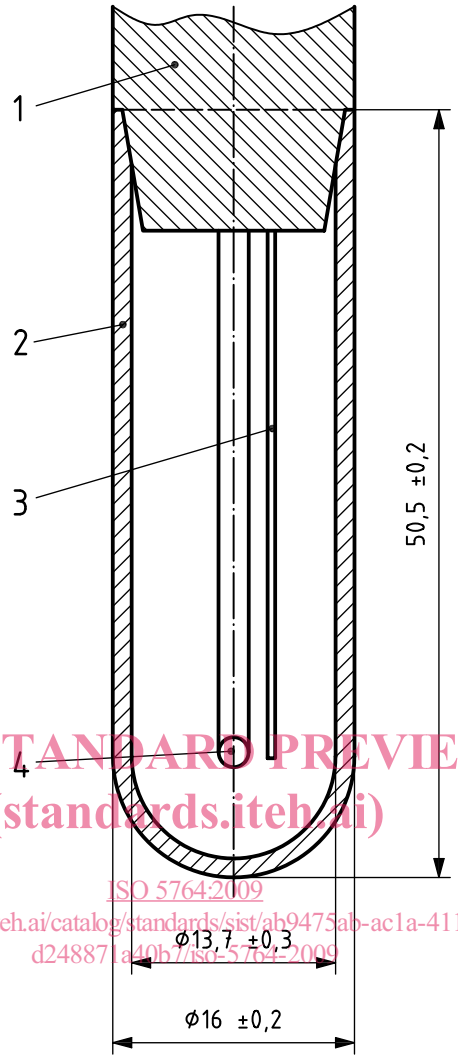
Dimensions in millimetres



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Key

1 mandrel
2 sample tube
3 stirring wire
4 thermistor bead

Figure 1 — Details of sample tube, thermistor probe, and stirring wire

9 Preparation of test sample

9.1 Preparation

If necessary, remove any visible foreign bodies or solid butterfat from the test sample by filtering into a clean, dry vessel. Mix the sample gently. Use a filter that is inert to milk and effective when used at laboratory temperature.

Test the samples at their storage temperature or after having reached the laboratory temperature before commencing the determination. The test samples and the sodium chloride standard solutions shall have similar temperatures when commencing the determination (see also Clause 8).

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