
**Milk and milk products — Determination
of iron content — Spectrometric method
(Reference method)**

*Lait et produits laitiers — Détermination de la teneur en fer — Méthode
spectrométrique (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.

ISO 6732|IDF 103 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 6732|IDF 103 cancels and replaces the first edition (ISO 6732:1985), 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. IDF shall not be held responsible for identifying any or all such patent rights.

ISO 6732|IDF 103 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 *Minor compounds*, now part of the Standing Committee on *Analytical methods for composition*.

This edition of ISO 6732|IDF 103 cancels and replaces IDF 103A:1986, of which it constitutes a minor revision.

Milk and milk products — Determination of iron content — Spectrometric method (Reference method)

1 Scope

This International Standard specifies a spectrometric reference method for the determination of the iron content of milk and milk products.

This method is applicable to

- milk, skimmed milk, whey and buttermilk;
- plain yogurt and skimmed yogurt;
- evaporated milk and sweetened condensed milk;
- dried whole and skimmed milk, dried whey and dried buttermilk;
- cream and butter;
- anhydrous butterfat, butteroil, butterfat and ghee;
- ice-cream;
- cheese of various ages, and processed cheese;
- caseins, caseinates and coprecipitates.

2 Terms and definitions

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

2.1

iron content in milk and milk products

mass fraction of substances determined by the procedure specified in this International Standard

NOTE The iron content is conventionally expressed in milligrams per kilogram of sample.

3 Principle

Organic material is digested with a mixture of nitric and sulfuric acids, preceded, in the case of cream and anhydrous butterfat, butteroil, butterfat and ghee, by removal of the fat. In the case of butter, serum is separated and digested.

Iron(II) ions, obtained by reduction of iron(III) ions, are complexed with bathophenanthroline. The iron(II) compound is extracted with isoamyl alcohol. The absorbance of the red solution thus obtained is measured spectrometrically at a wavelength of 533 nm.

4 Reagents and materials

IMPORTANT — Maintain reagents, glassware and equipment, as well as the laboratory environment as clean as possible in order to avoid contamination by rust. Each laboratory should check and identify its own sources of contamination.

Use only reagents of very pure analytical grade and which, with the exception of the iron standard solutions (4.14 and 4.15), are free from iron.

4.1 Water, complying with grade 2 as defined in ISO 3696^[5].

4.2 Ethanol (C₂H₅OH), about 96 % volume fraction.

Distil, if necessary, in an iron-free distillation unit.

4.3 Diethyl ether (C₂H₅OC₂H₅).

Distil, if necessary, in an iron-free distillation unit.

4.4 Light petroleum, boiling range 40 °C to 60 °C.

Distil, if necessary, in an iron-free distillation unit.

4.5 Nitric acid (HNO₃), concentrated, $\rho_{20} = 1,42$ g/ml.

Distil in an iron-free distillation unit. Discard the first 50 ml of distillate. Do not store the nitric acid in a brown glass bottle.

4.6 Sulfuric acid¹⁾ (H₂SO₄), concentrated, $\rho_{20} = 1,84$ g/ml.

4.7 Potassium sulfate¹⁾, solution in sulfuric acid.

Dissolve 25 g of anhydrous potassium sulfate (K₂SO₄) in sulfuric acid (4.6) and make up to 100 ml with the same acid. Filter the solution, without suction, through an all-glass, iron-free, filter crucible, of porosity grade P 100 (pore diameter 40 µm to 100 µm).

If the potassium sulfate available is not iron-free, purify it as follows.

Dissolve 40 g of potassium sulfate in 500 ml of water (4.1) and add 3 ml of the hydroxylammonium chloride solution (4.10). Extract the solution with 10 ml of the bathophenanthroline solution (4.12). Remove the upper layer. Repeat these two operations until the upper layer remains colourless. Evaporate the water in a clean oven.

4.8 Hydrogen peroxide¹⁾ (H₂O₂), solution, $\rho_{20} = 1,099$ g/ml to 1,103 g/ml.

Store in a refrigerator.

1) Aristar, Suprapur and Ultrex reagents are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO or IDF of these products.