Milk fat — Determination of peroxide value

Matière grasse laitière — Détermination de l'indice de peroxyde
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Foreword

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ISO 3976|IDF 74 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 edition of ISO 3976|IDF 74 cancels and replaces ISO 3976:1977, which has been technically revised. A comparison of the results using the new reagent (methanol/1-decanol/n-hexane mixture) with those found using chloroform/methanol is given in Annex C.
Foreword

IDF (the International Dairy Federation) is a worldwide federation of the dairy sector with a National Committee in every member country. Every National Committee has 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 ISO-IDF Action Team on Fat, of the Standing Committee on Main components of milk, under the aegis of its project leader, Mr A. van Reusel (BE).

This edition of ISO 3976 | IDF 74 cancels and replaces IDF 74A:1991, which has been technically revised. A comparison of the results using the new reagent (methanol/1-decanol/n-hexane mixture) with those found using chloroform/methanol is given in Annex C.
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WARNING — The use of this International Standard may involve the use of hazardous materials, operations, and equipment. This International Standard does not purport to address all the safety risks associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of local regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the determination of the peroxide value of anhydrous milk fat.

The method is suitable for anhydrous milk fat having a peroxide value up to 1,3 mmol of oxygen per kilogram.

NOTE For milk fat samples with peroxide values between 0,5 mmol and 1,3 mmol of oxygen per kilogram, an extended procedure (see Annex A) is used. For milk fat samples with peroxide values of more than 1,3 mmol of oxygen per kilogram, an iodine/thiosulfate method can be used (e.g. AOAC 920.160).

2 Terms and definitions

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

2.1 peroxide value
amount of substance determined by the procedure specified in this International Standard

NOTE The peroxide value is expressed as millimoles of oxygen per kilogram.

3 Principle

A test portion is dissolved in a mixture of methanol/1-decanol/n-hexane, then iron(II) chloride and ammonium thiocyanate are added. The peroxides oxidize the iron(II) which forms a red iron(III) complex with the ammonium thiocyanate. The amount of substance is calculated from a photometric determination of the red iron(III) complex, after a fixed period of reaction.

4 Reagents

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

4.1 Methanol/1-decanol/n-hexane mixture, in ratio 3:2:1 (volume fraction).

Mix 2 volume parts of 1-decanol with 1 volume part of n-hexane. Add 3 volume parts of anhydrous methanol to that mixture and mix again.
The mixture is flammable and has an unpleasant odour. Therefore, it is recommended to work in a fume cupboard and to wear gloves.

Petroleum ether with a boiling range at between 60 °C and 80 °C may be used instead of n-hexane.

4.2 Iron(II) chloride (FeCl₂) solution, c(Fe²⁺) ~1 mg/ml.

Prepare the iron(II) chloride solution in indirect, dimmed light.

Dissolve approx. 0.4 g of barium chloride dihydrate (BaCl₂·2H₂O) in about 50 ml water. Then dissolve approx. 0.5 g of iron(II) sulfate heptahydrate (FeSO₄·7H₂O) in about 50 ml water. Slowly pour the barium chloride solution, with constant stirring, into the iron(II) sulfate solution. Add about 2 ml of hydrochloric acid solution I (4.5) and mix again.

Allow the precipitate of barium sulfate to settle or centrifuge the mixture until the upper liquid layer is clear. Decant the thus-obtained clear solution into a brown bottle. Do not store the solution for more than 1 week.

Alternatively, the iron(II) chloride solution may be prepared by dissolving approximately 0.35 g of iron(II) chloride tetrahydrate (FeCl₂·4H₂O) in about 100 ml water. Add 2 ml of hydrochloric acid solution I (4.5) and mix.

4.3 Ammonium thiocyanate solution.

Dissolve approx. 30 g of ammonium thiocyanate (NH₄SCN) in water. Dilute with water to 100 ml. If the solution is not colourless, wash the solution several times with small amounts (e.g. 5 ml portions) of iso-amyl alcohol (3-methylbutan-1-ol).

4.4 Iron(III) chloride (FeCl₃) standard solution, c(Fe) = 10 µg/ml.

Dissolve 0.500 g of iron powder in about 50 ml of hydrochloric acid solution I (4.5) in a 500 ml one-mark volumetric flask. Add 1 ml to 2 ml of hydrogen peroxide solution (4.7). Remove the excess of hydrogen peroxide by boiling for 5 min. Cool to room temperature. Dilute to the 500 ml mark with water and mix.

The iron(III) chloride solution containing 1 g/l of Fe may also be prepared from standardized chemicals available commercially.

Transfer, using a pipette, 1 ml of the obtained solution to a 100 ml one-mark volumetric flask. Dilute to the 100 ml mark with methanol/1-decanol/n-hexane mixture (4.1) and mix.

4.5 Hydrochloric acid solution I, approx. c(HCl) = 10 mol/l.

4.6 Hydrochloric acid solution II, approx. c(HCl) = 0.2 mol/l.

Dilute 2 ml of hydrochloric acid solution I (4.5) with water to 100 ml.

4.7 Hydrogen peroxide solution (H₂O₂), of mass fraction approx. 30 %.

4.8 Dilute nitric acid (HNO₃), of mass fraction approx. 10 %.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Glassware.

Clean all glassware by soaking in dilute nitric acid (4.8) for 24 h. Rinse the glassware four times with tap water and four times with distilled or equivalent water before drying it in the oven (5.10) set at 100 °C for 1 h.