INTERNATIONAL STANDARD 3976

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Anhydrous milk fat — Determination of peroxide value (Reference method)

Matière grasse de lait déshydratée — Détermination de l'indice de peroxyde (Méthode de référence)

First edition — 1977-11-15

UDC 637.143 : 637.127.1 : 543.852.46 **Ref. No. ISO 3976-1977 (E)**

Descriptors : dairy products, milk, fats, chemical analysis, determination of content, oxygen, peroxide number.

Price based on 3 pages

FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3976 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in September 1975.

It has been approved by the member bodies of the following countries :

Australia	Germany	New Zealand
Austria	Ghana	Poland
Belgium	Hungary	Portugal
Bulgaria	India	Romania
Canada	Iran	South Africa, Rep. of
Czechoslovakia	Israel	Spain
Egypt, Arab Rep. of	Mexico	Turkey
France	Netherlands	Yugoslavia

The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

NOTE – The method specified in this International Standard has been developed by a Joint Group of Experts of the IDF (International Dairy Federation), the AOAC (Association of Official Analytical Chemists, U.S.A.) and ISO. The method will also be included in the FAO/WHO Code of Principles concerning Milk and Milk Products and Associated Standards.

Printed in Switzerland

Anhydrous milk fat — Determination of peroxide value (Reference method)

1 SCOPE AND FIELD OF APPLICATION

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

The method is applicable to anhydrous milk fat, anhydrous butter oil (anhydrous butterfat), butter oil (butterfat) or ghee having peroxide values not exceeding 1,0.

NOTE — These products are defined in IDF Standard 68 : 1971.

It is not applicable to products containing gallates as antioxidants.

2 REFERENCE

ISO/R 707, *Milk and milk products — Sampling.*

3 DEFINITION

peroxide value : The number of milliequivalents of oxygen per kilogram of anhydrous milk fat, determined by the procedure described.

4 PRINCIPLE

Dissolution of a test portion in a mixture of chloroform and methanol and addition of iron(II) chloride and ammonium thiocyanate. After a fixed reaction time, photometric determination of the red iron(III) complex.

5 REAGENTS

All reagents shall be of analytical reagent quality. The water used shall be distilled water or water of at least equivalent purity.

5.1 Chloroform/methanol mixture.

Mix 70 volumes of chloroform (trichloromethane) and 30 volumes of anhydrous methanol.

5.2 Iron(II) chloride solution.

This solution shall be prepared in indirect, dimmed light.

Dissolve approximately 0,4 g of barium chloride dihydrate ($BaCl_2.2H_2O$) in about 50 ml of water.

Dissolve approximately 0,5 g of iron(II) sulphate heptahydrate ($FeSO_4.7H_2O$) in about 50 ml of water.

Slowly pour the barium chloride solution, with constant stirring, into the iron(II) sulphate solution and add about 2 ml of approximately 10 N hydrochloric acid.

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

NOTE — The iron(II) chloride solution can also be prepared by dissolving approximately 0,35 g of iron(II) chloride tetrahydrate ($FeCl_2.4H_2O$) in about 100 ml of water and adding 2 ml of approximately 10 N hydrochloric acid.

5.3 Ammonium thiocyanate solution.

Dissolve approximately 30 g of ammonium thiocyanate (NH_4SCN) in water and dilute to 100 ml. If the solution is not colourless, remove the colour by extracting the solution several times with small amounts (for example 5 ml portions) of iso-amyl alcohol (3-methyl-butan-1-ol).

5.4 Iron(III) chloride, standard solution corresponding to 10 μg of Fe per millilitre.

Dissolve 0,500 g of iron powder or iron wire in about 50 ml of 10 N hydrochloric acid and 1 to 2 ml of about 30 % (*m/m*) hydrogen peroxide solution.

Remove the excess of hydrogen peroxide by boiling for 5 min. Cool to room temperature and dilute with water to 500 ml in a volumetric flask. Transfer, by means of a pipette, 1 ml of this solution to a 100 ml volumetric flask, dilute to the mark with the mixture of chloroform and methanol (5.1) and mix.

5.5 Hydrochloric acid, approximately 0,2 N solution.

Dilute 2 ml of approximately 10 N hydrochloric acid with water to 100 ml.

6 APPARATUS

6.1 Analytical balance.

6.2 Burettes, of 10 ml capacity, graduated in 0,02 ml, complying with class A of ISO/R 385.