

# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

## ISO RECOMMENDATION

### R 1736

DRIED MILK

DETERMINATION OF FAT CONTENT

(REFERENCE METHOD)

1st EDITION

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## BRIEF HISTORY

The ISO Recommendation R 1736, *Dried milk – Determination of fat content (Reference method)*, was drawn up by Technical Committee ISO/TC 34, *Agricultural food products*, the Secretariat of which is held by the Magyar Szabványügyi Hivatal (MSZH).

Work on this question led to the adoption of Draft ISO Recommendation No. 1736, which was circulated to all the ISO Member Bodies for enquiry in April 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Romania
Austria	India	South Africa, Rep. of
Belgium	Iran	Sweden
Brazil	Israel	Switzerland
Chile	Netherlands	Turkey
Czechoslovakia	New Zealand	U.A.R.
France	Peru	United Kingdom
Germany	Poland	U.S.S.R.
Greece	Portugal	

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

NOTE. – This ISO Recommendation has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists : U.S.A.) on the basis of an IDF Standard, for the purpose of being included in the FAO/WHO Code of Principles concerning Milk and Milk Products and Associated Standards.

The text as approved by the above organizations was also published by FAO/WHO (Code of Principles, Standard No. B-2), by the IDF (IDF Standard 9A) and by the AOAC (Official Methods of Analysis, 11th Edition, 16.156/16.157).

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## DRIED MILK

## DETERMINATION OF FAT CONTENT

## (REFERENCE METHOD)

## 1. SCOPE AND FIELD OF APPLICATION

## 1.1 Scope

This ISO Recommendation describes a reference method for the determination of the fat content of dried milk.

## 1.2 Field of application

The method is suitable for dried whole milk, dried partially skimmed milk and dried skimmed milk.

## 2. DEFINITION

By the *fat of dried milk* is meant the substances extracted by the procedure described.

The fat content is expressed as a percentage by mass.

## 3. PRINCIPLE

Extraction of the fat from an ammoniacal ethanolic solution of dried milk with diethyl ether and light petroleum, evaporation of the solvents and weighing of the residue (commonly known as the Röse-Gottlieb method).

## 4. REAGENTS

All reagents should be of analytical reagent quality and should leave no residue greater than that permitted for the blank test (see clause 7.2). If necessary, solvents may be redistilled in the presence of about 1 g of butterfat per 100 ml of solvent. Water used should be distilled water or water of at least equivalent purity.

4.1 *Ammonia solution*, approximately 25 % (m/m)  $\text{NH}_3$  ( $\rho_{20}$  approximately 0.91 g/ml), or a stronger solution of known concentration.

4.2 *Ethanol*, 94 to 97 % (V/V) or, if not available, ethanol denatured with methanol, ethyl methyl ketone, benzene or light petroleum.

4.3 *Diethyl ether*, peroxide-free.

## NOTES

1. To test for peroxides, add to 10 ml of the ether in a small glass stoppered cylinder, previously rinsed with the ether, 1 ml of freshly prepared 10 % potassium iodide solution. Shake and let stand for 1 minute. No yellow colour should be observed in either layer.

2. Diethyl ether may be freed and maintained free from peroxides by adding wet zinc foil that has previously been immersed completely in dilute acidified copper sulphate solution for 1 minute and then washed in water. Approximately 80 cm<sup>2</sup> of zinc foil should be used per litre and it should be cut in strips long enough to reach at least half way up the container.

4.4 *Light petroleum* (petroleum ether) with any boiling range between 30 and 60 °C.

4.5 *Mixed solvent*, prepared shortly before use by mixing equal volumes of diethyl ether (4.3) and light petroleum (4.4).

NOTE. - Where mixed solvent is specified, the diethyl ether or the light petroleum may be used alone instead.

## 5. APPARATUS

- 5.1 *Analytical balance.*
- 5.2 *Suitable extraction apparatus*, provided with ground glass stoppers, bark corks of good quality or other closures unaffected by the solvents used.  
Treat bark corks by extracting successively with diethyl ether and light petroleum. Then keep these corks for at least 20 minutes in water at 60 °C or above, and cool in the water so that they are saturated when used.
- 5.3 *Thin-walled, flat-bottomed flasks* of 150 to 250 ml capacity.
- 5.4 *Drying oven*, well ventilated, thermostatically controlled and adjusted to operate at  $102 \pm 2$  °C, or  
*vacuum drying oven*, temperature 70 to 75 °C, pressure less than 66 mbar (50 mmHg).
- 5.5 *Material to facilitate boiling*, fat-free, non-porous, non-friable in use, for example glass beads or pieces of silicon carbide.  
NOTE. – The use of this material is optional (see clause 7.3).
- 5.6 *Water bath* at 60 to 70 °C.
- 5.7 *Centrifuge* in which the extraction apparatus (5.2) can be spun at 500 to 600 rev/min.

### NOTES

1. The use of a centrifuge is optional (see clause 7.5.2).
2. When using a centrifuge not provided with a three-phase motor, sparks may occur and care is therefore necessary to avoid explosion or fire due to the occurrence of solvent vapour following breakage of apparatus.

## 6. SAMPLING

Carry out the sampling by the appropriate method described in ISO Recommendation R 707, *Milk and milk products – Sampling*.

## 7. PROCEDURE

### 7.1 Preparation of the sample

Transfer the dried milk to a clean, dry container (provided with an airtight lid) of a capacity about twice the volume of the sample. Close the container immediately and thoroughly mix the dried milk by repeatedly shaking and inverting the container. During the preparation of the sample, exposure of the dried milk to the atmosphere should be avoided as far as possible to minimize absorption of moisture.

### 7.2 Blank test

At the same time as the determination of the fat content of the sample, perform a blank determination on 10 ml of distilled water using the same type of extraction apparatus, the same reagents in the same amounts and the same procedure as described in clauses 7.3 and 7.5. If the result of the blank determination exceeds 0.0005 g, the reagents should be checked and the impure reagent or reagents purified or replaced.

### 7.3 Preparation of flask

Dry a flask (5.3) (if desired, with some material (5.5) to promote gentle boiling during the subsequent removal of the solvents) in the oven (5.4) for 30 to 60 minutes. Allow the flask to cool to the temperature of the balance room and then weigh it to the nearest 0.0001 g.

### 7.4 Test portion

Weight to the nearest 0.001 g directly in, or by difference into, the extraction apparatus (5.2) about 1 g of dried whole milk or about 1.5 g of dried partially skimmed milk or dried skimmed milk. Add 10 ml of water and shake until the test portion is completely dispersed.