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BSI Standards Publication

Instant dried milk — Determination of the dispersibility and wettability

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National foreword

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Instant dried milk — Determination of the dispersibility and wettability

Poudre de lait instantanée — Détermination de la dispersibilité et de la mouillabilité

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Forewords

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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The committee responsible for this document is ISO/TC 34, *Food and food products*, Subcommittee SC 5, *Milk and milk products* and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

IDF (the International Dairy Federation) is a non-profit private sector organization representing the interests of various stakeholders in dairying at the global level. IDF members are organized in National Committees, which are national associations composed of representatives of dairy-related national interest groups including dairy farmers, dairy processing industry, dairy suppliers, academics and governments/food control authorities.

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This document was prepared by the IDF Standing Committee on *Analytical Methods for Composition* and ISO Technical Committee ISO/TC 34, *Food and food products*, Subcommittee SC 5, *Milk and milk products*.

This IDF Reviewed method is equal to an ISO Publicly Available Specification (ISO/PAS) or an ISO Technical Specification (ISO/TS) and is therefore published jointly under ISO conditions.

The work was carried out by the IDF/ISO Project Group C28 of the Standing Committee on *Analytical Methods for Composition* under the aegis of its project leader, Dr. Steve Holroyd (NZ).

This ISO Technical Specification/IDF Reviewed Method replaces the standard IDF 87:1979, *Instant dried milk — Determination of the dispersibility and wettability* which has been withdrawn.

Introduction

The degree to which a dried milk is "instant" depends on various properties which can be classified as wettability, sinkability, dispersibility, and solubility. The first three affects the last to some extent and, hence, dispersibility, as defined and determined in this Technical Specification, is probably the best single criterion for assessing the overall instant characteristics of a dried milk. Provisional recommendations for acceptable dispersibility values for instant dried skimmed milk and instant dried whole milk are given in Annex A. Although wettability is a property not easily distinguishable from sinkability and is difficult to measure accurately, in milk powders, an approximate determination of wettability, which can be made rapidly, provides a useful indication of the degree to which a dried milk is likely to possess instant characteristics. In Annex B, a rapid routine method for the determination of the wettability (wetting time) in water of instant dried milk is described.

TECHNICAL SPECIFICATION

ISO/TS 17758:2014(E)
IDF/RM 87:2014(E)

Instant dried milk — Determination of the dispersibility and wettability

1 Scope

This Technical Specification specifies a method for the determination of the dispersibility in water of instant dried milk.

The method is applicable to instant dried skimmed milk manufactured by either the "straight-through" or the "re-wet" process and also to instant dried whole milk.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5537|IDF 26, *Dried milk — Determination of moisture content (Reference method)*

ISO 6731|IDF 21, *Milk, cream and evaporated milk — Determination of total solids content (Reference method)*

3 Principle

A test portion of the sample of known moisture content is evenly spread on the surface of water at a temperature of 25 °C. The mixture is stirred manually for a short time; then, part of the mixture is filtered through a sieve, and the total solids content of the collected liquid determined. Dispersibility is calculated from the mass of the test portion and the values for moisture content and total solids content.

4 Reagents

Use only distilled or demineralized water or water of equivalent purity.

5 Apparatus

5.1 Container, capacity about twice the volume of the laboratory sample (Clause 6), with airtight lid.

5.2 Apparatus, as specified in ISO 5537|IDF 26.

5.3 Balance, top-pan, capable of being read to the nearest 0,1 g.

5.4 Scoop, suitable for transporting the test portion (8.3.2) for weighing.

5.5 Thermometer, suitable for measuring a temperature of (25 ± 1) °C.

5.6 Glass beaker, with spout, capacity 600 ml, external diameter (90 ± 2) mm, overall height (126 ± 3) mm, graduated at 150 ml and 250 ml, with the rim lying in a horizontal plane parallel to the base [see Figure 1 a)].

5.7 Glass plate, 120 mm × 120 mm, thickness 2,5 mm, with ground edges [see Figure 1 a)].

5.8 Glass tubing, length 65 mm, outside diameter (80 ± 1,8) mm, wall thickness (2,5 ± 0,3) mm, with the ends ground, parallel, and at right angles to the longitudinal axis [see Figure 1 a)].

5.9 Stand and clamp, for holding the glass tubing (5.8).

5.10 Brush, suitable for removing the test portion from the scoop (5.4).

5.11 Spatula, stainless steel, thickness 1 mm, overall length 250 mm, length of blade 135 mm, width of blade 25 mm [see Figure 1 b)].

These dimensions should be adhered to as closely as possible.

5.12 Stopwatch, 60 s, numbered at intervals of 5 s, indicating intervals of 1 s and 0,5 s (or less).

It is helpful to have the dial marked at 5 s, 25 s, and 55 s.

5.13 Test sieve, diameter 200 mm, woven metal wire cloth, nominal size of apertures 150 µm, with receiver, complying with ISO 565[1].

5.14 Conical flask, capacity 250 ml, with stopper.

5.15 Glass funnel, suitable for transferring the contents of the sieve receiver to the conical flask (see 8.3.9).

5.16 Apparatus, as specified in ISO 6731|IDF 21.

6 Sampling

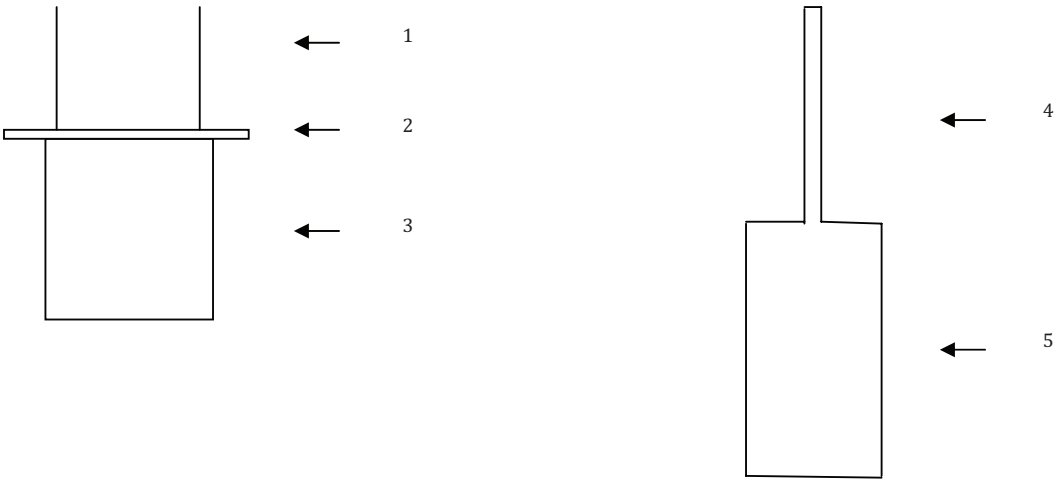
Sampling is not part of the method specified in this Technical Specification. A recommended sampling method is given in ISO 707|IDF 50[2].

It is important that the laboratory receives a representative sample which has not been damaged or changed during transport or storage.

The laboratory sample shall be placed in a clean, dry, securely closed, airtight container. With instant dried milk, it is essential that care is taken during sampling to avoid reducing the particle size of the dried milk and to prevent this happening during transport of the laboratory sample, the sample container should be completely filled. Note and report any deviation from these requirements.

7 Preparation of test sample

Transfer all the laboratory sample, which should be at ambient temperature, to the container (5.1) and immediately close the container. Thoroughly mix the sample by very gently inverting and rotating the container in such a way as to avoid reducing the particle size of the sample.



Key

1 glass tubing (5.8)
2 glass plate (5.7)
3 glass beaker (5.6)
4 spatula (5.11) handle
5 spatula (5.11) blade

Figure 1 — Apparatus for the determination of dispersibility and wetting time (diagrammatic)

8 Procedure

8.1 Pretreatment of test sample

Before proceeding with the operations specified in 8.2 and 8.3, ensure that the test sample (Clause 7) has been at laboratory temperature (20 °C to 25 °C) for at least 48 h. This pretreatment is necessary with all test samples so that any influence on dispersibility attributable to the physical state of the fat is constant from sample to sample.

8.2 Moisture content of pretreated test sample

Carry out, in duplicate, the procedure specified in ISO 5537|IDF 26 to obtain two single values (to the nearest 0,01 % mass fraction) for the moisture content of the pretreated test sample (8.1). Record the mean of these values, to the nearest 0,1 % mass fraction, as the moisture content.

8.3 Test method

8.3.1 Carry out the test method in duplicate to obtain duplicate single values for dispersibility.

8.3.2 Mix the pretreated test sample (8.1) by very gently inverting and rotating the container a few times. Using the balance (5.3), weigh out in the scoop (5.4) (26 ± 0,1) g of instant dried skimmed milk or (34 ± 0,1) g of instant dried whole milk.

8.3.3 Weigh out (250 ± 0,1) g of water, adjusted to (25 ± 1) °C, in a dry glass beaker (5.6), taking care that the inside of the beaker above the final water level remains dry.

NOTE Recommended water temperature is 25 °C. In laboratories, for skimmed milk powder, water temperature used is 25 °C and for whole milk powder, water temperature is 40 °C.

8.3.4 Place the beaker strictly horizontally on the base of the stand (5.9), place the glass plate (5.7) centrally on the top of the beaker, and place the glass tubing (5.8) on the glass plate, clamping the tubing so that it is centrally located above the beaker and the glass plate is free enough to be withdrawn [see Figure 1 a)].

8.3.5 Transfer all of the test portion (8.3.2) into the glass tubing, using the brush (5.10) if necessary, and spread the test portion evenly over the glass plate with the spatula (5.11).

8.3.6 Start the stopwatch (5.12) and, when after 1 min its main hand again indicates 0/60 s, withdraw the glass plate with one hand (holding the beaker with the other hand) so that the test portion progressively falls on to the surface of the water. The withdrawal of the plate shall be performed with a gentle continuous movement and shall be accomplished in approximately 2,5 s.

8.3.7 Immediately remove the beaker from below the glass tubing. When the main hand of the stopwatch indicates 5 s, lower the spatula down the side of the beaker until it touches the bottom. For the next 5 s, stir the contents of the beaker with the spatula, making one complete stirring movement per second, i.e. a smooth continuous movement of the spatula across the beaker from one side to the opposite side and back and taking 1 s, with the end of the spatula blade in continuous contact with the bottom of the beaker and slightly tilting the spatula away from the side of the beaker at the end of each half stirring movement so as to minimize accumulation of unwetted dried milk on the sides of the beaker. Without interruption, continue the stirring for 15 s in the same manner except that the spatula is maintained in a vertical position throughout. While making the 20 complete stirring movements in 20 s, continuously rotate the beaker on its base so that approximately one complete turn (360°) is achieved during the stirring.

8.3.8 After the completion of the stirring, allow the contents of the beaker to stand for 30 s, i.e. until the main hand of the stopwatch indicates 55 s, and then, without disturbing any sediment, quickly pour off the liquid down to approximately the 150 ml graduation mark distributing the decanted liquid as evenly as possible over the test sieve (5.13), below which is fitted the receiver. Do not tilt or move the sieve during the sieving. To facilitate the passage of the liquid through the sieve, the sieve is wetted before any use by rinsing it with water. All excess water is removed from the sieve by wiping it with a towel, the top and bottom surfaces of the wire cloth are only superficially wiped. The receiver shall be clean and dry.

8.3.9 After 30 s of the beginning of the sieving operation, i.e. when the main hand of the stopwatch has returned to the 25 s position, transfer as completely as possible the contents of the receiver to the conical flask (5.14) by means of the glass funnel (5.15), and stopper the flask.

8.3.10 Thoroughly mix the liquid in the flask by repeatedly inverting the flask. Carry out, in duplicate, the procedure specified in ISO 6731|IDF 21 to obtain two single values (to the nearest 0,01 % mass fraction) for the total solids content of the liquid. Record the mean of these values, to the nearest 0,1 % mass fraction, as the total solids content.

9 Expression of results

9.1 Method of calculation

Calculate each duplicate single value for dispersibility, *D*, expressed as a percentage, using Formula (1) for instant dried skimmed milk:

$$w_D = \frac{w_1 \times 962}{100 - \left(w_{H_2O} + w_1\right)} \quad (1)$$

and Formula (2) for instant dried whole milk:

$$D = \frac{w_1 \times 735}{100 - \left(w_{H_2O} + w_1\right)} \quad (2)$$

where

w_1 is the total solids content, expressed as a percentage mass fraction, of the liquid (8.3.10);

w_{H_2O} is the moisture content, expressed as a percentage mass fraction, of the pretreated test sample (8.2).

Provided these values comply with the requirements of 9.2, report the mean value expressed to the nearest 1 % as the dispersibility of the laboratory sample.

NOTE The formulae are derived as follows. If a mass of dry matter and moisture, m_{2+H_2O}, in grams, in a test portion of mass m_3, in grams, is dispersed in 250 g of water, then

$$w_1 = \frac{m_{2+H_2O} \times \left[\left(100 - w_{H_2O}\right)/100\right] \times 100}{250 + m_{2+H_2O}} \quad (3)$$

and therefore

$$m_{2+H_2O} = \frac{250 \times w_1}{100 - \left(w_{H_2O} + w_1\right)} \quad (4)$$

since

$$D = \frac{m_{2+H_2O} \times \left[\left(100 - w_{H_2O}\right)/100\right] \times 100}{m_3 \times \left[\left(100 - w_{H_2O}\right)/100\right]} = \frac{m_{2+H_2O} \times 100}{m_3} \quad (5)$$

then

$$D = \frac{250 \times w_1}{100 - \left(w_{H_2O} + w_1\right)} \times \frac{100}{m_3} \quad (6)$$

For instant dried skimmed milk, substituting m_3 = 26 into Formula (6) gives Formula (1). For instant dried whole milk, substituting m_3 = 34 into Formula (6) gives Formula (2).

9.2 Repeatability

The difference between duplicate single values for dispersibility obtained within a short time interval by the same analyst shall not exceed 4 %.

10 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample, including the dates of receipt and testing of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this Technical Specification, i.e. ISO/TS 17758|IDF/RM 87:2014;

d) all operating details not specified in this Technical Specification, or regarded as optional, together with details of any incidents which might have influenced the test result(s);

e) the test result(s) obtained, expressed as percentage(s);

f) if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Recommendation for interpretation of dispersibility values

On the basis of knowledge and experience at the time of publication of this Technical Specification , it is recommended that a dried milk purporting to be “instant” should be regarded as failing to possess sufficient instant characteristics if its dispersibility (mean of duplicate single values), when determined as specified in this Technical Specification, is less than 95 % for dried skimmed milk and less than 90 % for dried whole milk.

Annex B
(informative)

Determination of the wettability (wetting time) of instant dried milk

B.1 General

The wetting time of instant dried milk, as defined and determined in this annex, has been found to have an approximate inverse relationship to dispersibility (9.1), but the quantitative relationship is different for instant dried skimmed milks manufactured by different processes and for instant dried whole milk. These relationships are only approximate because the precision with which wetting time can be determined is not high and other properties, e.g. sinkability and self-dispersibility, can have an overriding effect on either wetting time or dispersibility. Accordingly, neither acceptable wetting times nor the relationships between wetting time and dispersibility are given in this annex. However, if comparative values for wetting time and dispersibility are accumulated, e.g. by a manufacturer, a determination of wetting time, which can be made rapidly, should indicate whether an instant dried milk is likely to possess an acceptable dispersibility.

This annex specifies a rapid routine method for the determination of the wetting time in water of instant dried milk. The method is applicable to instant dried skimmed milk manufactured by either the "straight-through" or the "re-wet" process and also to instant dried whole milk.

B.2 Principle

A test portion of the sample is evenly spread on the surface of water at a temperature of 25 °C using the apparatus and technique specified in Clause 5 and 8.3. The time required for all the particles of the sample to become wetted, i.e. to sink below the surface of the water and any remaining on the surface to assume a typical "wet" appearance, is obtained.

B.3 Reagents and apparatus

As specified in Clause 4 and in 5.1 and in 5.3 to 5.12 inclusive.

B.4 Sampling and samples

As specified in Clauses 6 and 7.

B.5 Procedure

B.5.1 Pretreatment of test sample

As specified in 8.1.

B.5.2 Test method

B.5.2.1 Carry out the test method in triplicate to obtain triplicate single values for wetting time.

B.5.2.2 Mix the pretreated test sample (B.5.1) by very gently inverting and rotating the container a few times. Using the balance (5.3), weigh out in the scoop (5.4) (10 ± 0,1) g of instant dried skimmed milk or instant dried whole milk.

B.5.2.3 Weigh out (250 ± 1) g of water, adjusted to (25 ± 1) °C, in a dry glass beaker (5.6), taking care that the inside of the beaker above the final water level remains dry.

B.5.2.4 Carry out the operations specified in 8.3.4 to 8.3.6.

B.5.2.5 Then immediately, but gently, remove the beaker from below the glass tubing and allow it to stand. As soon as all the particles of the test portion have become wetted (see B.2), stop the stopwatch and record the time in seconds (to the nearest second) elapsed since starting the watch.

B.6 Expression of results

B.6.1 Method of calculation

Calculate each triplicate single value for wetting time, t_1, in seconds, using Formula (9):

$$t_1 = t_2 - 60 \tag{9}$$

where t_2 is the time, in seconds, recorded (B.5.2.5).

Report the mean value expressed to the nearest second as the wetting time of the laboratory sample (see B.6.2).

B.6.2 Repeatability

Since the repeatability of the test method can vary widely from sample to sample, it would be inappropriate to give a value for this precision parameter; it is for the same reason that the test method is carried out in triplicate with each sample. As the extent of agreement among the triplicate values can indicate the uniformity with which instantizing has been accomplished, especially with instant dried whole milk, it is recommended that the three single values, as well as the mean, are reported.

B.7 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample, including the dates of receipt and testing of the sample;

b) the sampling method used, if known;

c) the test method used, with reference to this annex of this Technical Specification, i.e. ISO/TS 17758|IDF/RM 87:2014, Annex B;

d) all operating details not specified in this Technical Specification, or regarded as optional, together with details of any incidents which might have influenced the test result(s);

e) the test result(s) obtained, expressed in seconds (see B.6.2);

f) if the repeatability has been checked, the final quoted result obtained.

Bibliography

[1] ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

[2] ISO 707|IDF 50, *Milk and milk products — Guidance on sampling*

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