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## Dried milk and dried milk products — Determination of insolubility index

*Lait sec et produits laitiers en poudre — Détermination de l'indice  
d'insolubilité*

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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 8156|IDF 129 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 8156|IDF 129 cancels and replaces ISO 8156:1987, of which it constitutes a minor revision.

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## 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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ISO 8156|IDF 129 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 Joint ISO/IDF/AOAC Group of Experts on *Physical properties of dried milk products*, under the aegis of its chairman, Mr J. de Vilder (BE).

This edition of ISO 8156|IDF 129 cancels and replaces IDF 129A:1988, of which it constitutes a minor revision.

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## Introduction

**0.1** There are several rather elaborate gravimetric methods for determining the solubility of dried milk (for example, References [1] and [2]), but for routine purposes, including grading, the most widely used procedure is the so-called solubility index method of the American Dry Milk Institute (see Reference [3]), in which a test portion is mixed with water and the reconstituted product is centrifuged. The volume, in millilitres, of the sediment finally obtained (i.e. insoluble residue) is the solubility index. Since the solubility index is thus inversely related to solubility, it seems more direct and more rational to use the term “insolubility index” to describe what is determined by a “solubility” method such as that of the ADMI. Accordingly, “insolubility index” was adopted to designate what is determined in the sediment-volume solubility method described in this International Standard. Use of this new expression also serves to differentiate the method described in this International Standard from the solubility index method of the ADMI.

Although the ADMI solubility index method has been in use in many countries for a considerable period, it became evident some time ago that its precision (repeatability, reproducibility), which is not stated by ADMI, is unsatisfactory with some types of spray-dried whole milk and with roller-dried milk and milk products. This led to the conclusion that the apparatus and technique of the ADMI method are inadequately defined, and are unsuitable for some dried milks, and consequently either the ADMI method should be more closely specified, and possibly modified in some respects, or an alternative method developed. The latter approach was at first favoured because of difficulty in obtaining the special mixer (and spare parts) manufactured in the USA for the ADMI method. However, when improved models of this mixer came to be manufactured in several countries and hence were readily available, it was decided to concentrate on improving the precision of the ADMI method while retaining its principal features so that most of the existing ADMI solubility index specifications for grading would still be applicable.

**0.2** In any sediment-volume solubility method applied to a dried milk or a dried milk product, the temperature at which the test portion is reconstituted is the main factor influencing what the result will be. In the ADMI solubility index method, a reconstituting temperature of 75 °F (23,9 °C) is used with spray-dried or roller-dried whole milk, skimmed milk and buttermilk, instant or non-instant as appropriate. But for the insolubility index method, it was decided to adopt the principle that the reconstituting temperature should be either 24 °C or 50 °C depending on whether the product, in normal usage from its quality specification, is expected to be reconstitutable in “cold” water or “warm” water respectively. **This means that the reconstituting temperature to be used in the insolubility index method will, in general, be 24 °C for spray-dried products and 50 °C for roller-dried products.** Exceptions to this general rule are spray-dried milk-based baby food and, in some instances, spray-dried whole milk or partly skimmed milk, intended to be reconstituted in warm water. However, it is important to note that if the insolubility index of spray-dried fat-containing milks is determined at 50 °C, the values obtained will all tend to be very small because the method will no longer detect products that have been subjected to excessive dry heat through faulty manufacture or storage. This is because milk protein denatured by dry heat is insoluble at 24 °C and, along with entrapped or combined fat, is precipitated as sediment when centrifuging is performed. At 50 °C, the dry-heat-denatured protein is soluble and this, with the release of the associated fat, can cause a marked reduction in the volume of sediment (see References [4] to [6]).

**0.3** The insolubility index method described in this International Standard is thus basically the same as the ADMI solubility index method, but with all the apparatus and experimental conditions as closely defined as practicable and the reconstituting temperature either 24 °C or 50 °C, as appropriate (see 0.2). The latter innovation means that an insolubility index value will require to be accompanied by the reconstituting temperature used, for example 0,25 ml (24 °C), 0,10 ml (50 °C). The precision of the insolubility index method has been determined in an interlaboratory collaborative study and is considered to be satisfactory.

# Dried milk and dried milk products — Determination of insolubility index

## 1 Scope

This International Standard specifies a method of determining the insolubility index, as a means of assessing the solubility, of dried whole milk, dried partly skimmed milk and dried skimmed milk, whether non-instant or instant.

NOTE These milks are defined in Reference [7] as “whole milk powder”, “partly skimmed milk powder” and “skimmed milk powder”, respectively.

The method is also applicable to dried whey, dried buttermilk and dried milk-based baby food, as well as to any of the dried products listed in which milk fat has been replaced by another fat, or which has been roller-dried instead of spray-dried.

## 2 Terms and definitions

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

### 2.1

#### insolubility index

volume, in millilitres, of sediment (insoluble residue) obtained when a dried milk or dried milk product is reconstituted and the reconstituted milk or milk product is centrifuged, under the conditions specified in this International Standard

## 3 Principle

Water at 24 °C (or at 50 °C if appropriate, see 0.2) is added to a test portion, which is reconstituted using a special mixer. After a specified standing period, a specified volume of the reconstituted milk or milk product is centrifuged in a graduated tube. The supernatant liquid is removed and the sediment is redispersed after the addition of water at the same temperature as used for the reconstitution. The mixture is centrifuged and the volume of sediment (insoluble residue) obtained is recorded.

## 4 Reagents

Use only distilled water or demineralized water, or water of at least equivalent purity.

**4.1 Silicone antifoaming agent**, for example an aqueous emulsion containing 30 % (mass fraction) of silicone.

Test the suitability of the silicone antifoaming agent by carrying out the procedure described in Clause 7 without a test portion. No more than a trace of silicone fluid ( $\leq 0,01$  ml) should be visible at the bottom of the tube at the end of the procedure.

## 5 Apparatus

Usual laboratory apparatus and, in particular, the following.

**5.1 Thermometer(s)**, capable of measuring a temperature of 24 °C and/or 50 °C with an error not exceeding  $\pm 0,2$  °C.

Since the temperature of reconstitution is the most important experimental factor governing values obtained for the insolubility index, it is essential that a thermometer of the specified accuracy is used for the procedures specified in 7.1 and 7.3 (and also 7.4.8).

**5.2 Water bath(s)**, capable of being maintained at  $24,0\text{ °C} \pm 0,2\text{ °C}$  and/or  $50,0\text{ °C} \pm 0,2\text{ °C}$ , in which one or more mixing jars (5.3) can be placed (see 9.3).

**5.3 Mixing jar**, made of glass, of capacity 500 ml, as supplied for use with the mixer (5.8).

The mixing jar (clover-leaf pattern) is illustrated in Figure 1; the dimensions are approximate.

**5.4 Scoop**, with a smooth surface, or **sampling paper**, black, glazed (of dimensions 140 mm  $\times$  140 mm), for weighing the test portion (7.3).

**5.5 Balance**, accurate to 0,01 g.

**5.6 Measuring cylinder**, made of plastic material, of capacity 100 ml  $\pm$  0,5 ml (at 20 °C).

NOTE The lower heat capacity of a plastic measuring cylinder, as compared to a glass one, minimizes possible changes in the temperature of the water placed in the cylinder (see 7.4.1).

**5.7 Brush**, suitable for removing any residual test portion from the scoop or sampling paper (5.4).

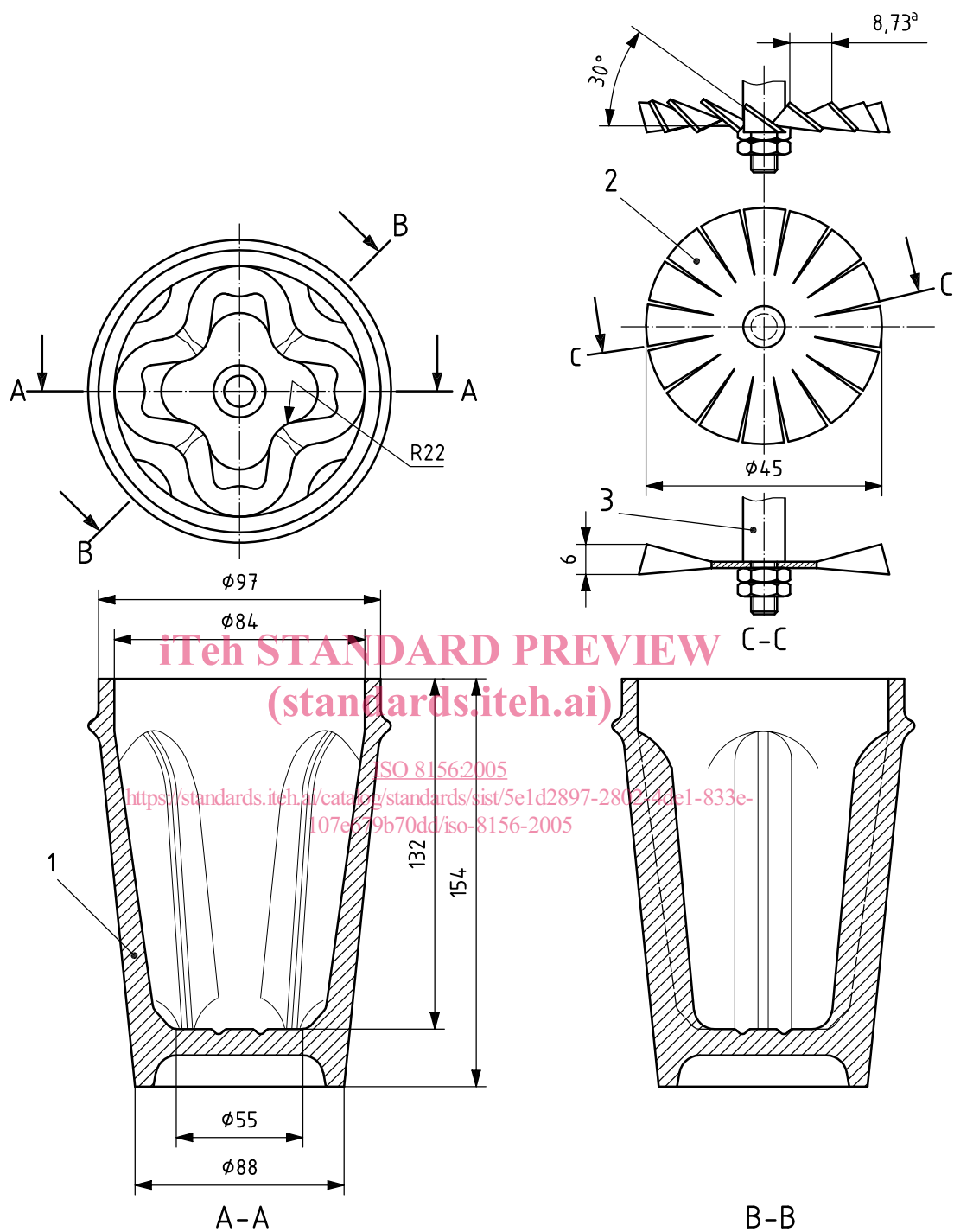
**5.8 Electric mixer**, equivalent to that manufactured for the solubility index method of the American Dry Milk Institute<sup>[3]</sup> with the following characteristics.

- a) The 16-bladed impeller (stainless steel) shall have the shape and diameter show in Figure 1, and shall be attached to the shaft of the mixer so that the "flat" side of the impeller is underneath, as also shown in Figure 1. The slope of the blades is upward from right to left; this is for clockwise rotation (see the Note).
- b) The pitch of the impeller blades shall be 30° and the horizontal distance between the blades (around the circumference of the impeller) shall be 8,73 mm, as shown in Figure 1. With usage of the impeller, these dimensions can change and hence periodic inspection and maintenance are essential.
- c) When the mixing jar (5.3) is fitted to the mixer, the length of the mixer shaft shall be such that the distance from the lowest part of the impeller to the bottom of the jar is 10 mm  $\pm$  2 mm; this means that for a jar of depth 132 mm the distance from the top of the jar to the lowest part of the impeller is 122 mm  $\pm$  2 mm, and to the plane of the lowest part of the impeller blades is 115 mm  $\pm$  2 mm. The impeller shall also be located centrally in the jar.
- d) When the mixing jar, containing 100 ml of water at 24 °C with or without the addition of an appropriate test portion (7.3), is fitted to the mixer and the mixer switched on, the impeller shall come to its operational fixed rotational frequency of  $(3\,600 \pm 100)\text{ min}^{-1}$  in less than 5 s. The direction of rotation of the impeller shall be clockwise (viewed from above). The rotational frequency of the impeller under load (as described above) shall be checked periodically with an electronic tachometer; this is especially necessary with older models of the mixer, with a non-synchronous motor, whose rotational frequency has to be adjusted to  $(3\,600 \pm 100)\text{ min}^{-1}$  by means of a speed control and a speed indicator (which may not remain accurate).

NOTE Some mixers for the ADMI method cause the impeller to rotate in an anticlockwise direction (viewed from above). These mixers require an impeller whose blades slope upward from left to right so that liquid in the mixing jar moves in the same way as with a clockwise rotating impeller. In all respects, including its manner of attachment to the shaft and its distance from the bottom of the mixing jar, an anticlockwise-rotating impeller is equivalent to a clockwise-rotating impeller.



Dimensions in millimetres



**Key**

1 mixer jar

2 impeller

3 mixer shaft

<sup>a</sup> Horizontal distance between blades (around circumference)

**Figure 1 — Mixing jar and impeller**