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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

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8156 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

NOTE — The method specified in this International Standard has been developed jointly with the International Dairy Federation (IDF) and the Association of Official Analytical Chemists (AOAC) and will also be published by these organizations.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Dried milk and dried milk products — Determination of insolubility index

0 Introduction

0.1 There are several rather elaborate gravimetric methods for determining the solubility of dried milk (for example Van Kreveld & Verhoog[1], 1963; British Standard 1743 : Part 2 : 1980[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 (ADMI[3], 1971) 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 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 ADMI. Accordingly, "insolubility index" was adopted to designate what is determined in the sediment-volume solubility method described in this International Standard; the use of this new expression also serves to differentiate the method described in this International Standard from the solubility index method of 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 or 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 may be 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 which 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 (Wright[4], 1932; Howat & Wright[5], 1933; Waite & White[6], 1949).

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 inter-laboratory collaborative study and is considered to be satisfactory.

1 Scope and field of application

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 (defined in FAO/WHO Standard A-5 [1] as "whole milk powder", "partly skimmed milk powder" and "skimmed milk powder", respectively) whether non-instant or instant.

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 Reference

ISO 707, *Milk and milk products — Methods of sampling.*

3 Definition

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.

4 Principle

Addition of water at 24 °C (or at 50 °C if appropriate, see 0.2) to a test portion and reconstitution using a special mixer. After a specified standing period, centrifuging a certain volume of the reconstituted milk or milk product in a graduated tube, removal of the supernatant liquid and redispersal of the sediment after adding water at the same temperature as used for reconstitution. Centrifuging the mixture and recording the volume of sediment (insoluble residue) obtained.

5 Reagent

During the analysis, use only distilled water or water of at least equivalent purity.

5.1 Silicone antifoaming agent, for example an aqueous emulsion containing 30 % (m/m) of silicone.

Test the suitability of the silicone antifoaming agent by carrying out the procedure described in clause 8 without a test portion. No more than a trace of silicone fluid ($\leqslant$ 0,01 ml) should be visible at the bottom of the tube at the end of the procedure.

6 Apparatus

6.1 Thermometer(s), capable of measuring a temperature of 24 °C and/or 50 °C with an error not exceeding ± 0,2 °C.

NOTE — Since the temperature of reconstitution is the most important experimental factor governing values obtained for the insolubility index, it is essential that a thermometer(s) of the specified accuracy is used for the procedures specified in 8.1 and 8.3 (and also 8.4.8).

6.2 Water bath(s), capable of being maintained at 24,0 ± 0,2 °C and/or 50,0 ± 0,2 °C, in which one or more mixing jars (6.3) can be placed (see 10.3).

6.3 Mixing jar, made of glass, of capacity 500 ml, as supplied for use with the mixer (6.8). The mixing jar (clover-leaf pattern) is illustrated in figure 1; the dimensions are approximate.

6.4 Scoop, with a smooth surface, or **sampling paper**, black, glazed (of dimensions 140 mm × 140 mm), for weighing the test portion (8.2).

6.5 Balance, accurate to 0,01 g.

6.6 Measuring cylinder, made of plastic material, of capacity 100 ± 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 8.3).

6.7 Brush, suitable for removing any residual test portion from the scoop or sampling paper (6.4).

6.8 Electric mixer, equivalent to that manufactured for the solubility index method of the American Dry Milk Institute[3], with the following characteristics.

a) The sixteen-bladed impeller (stainless steel) shall have the shape and diameter shown 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 (11/32 in), 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 (6.3) 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 ± 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 ± 2 mm, and to the plane of the lowest part of the impeller blades is 115 ± 2 mm. The impeller shall also be located centrally in the jar.

1) FAO/WHO Standard A-5 for whole milk powder, partly skimmed milk powder and skimmed milk powder, elaborated under the *Code of principles concerning milk and milk products*, 8th edition (1984), Rome : Food and Agriculture Organization of the United Nations/World Health Organization.