

INTERNATIONAL STANDARD

ISO
8156

First edition
1987-03-15



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

Dried milk and dried milk products — Determination of insolubility index

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

ITeH STANDARD PREVIEW
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ISO 8156:1987

<https://standards.iteh.ai/catalog/standards/sist/0a44b367-761f-40db-a008-1d12855a6da5/iso-8156-1987>

Reference number
ISO 8156: 1987 (E)

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.

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 Kreveland & 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-5¹⁾ 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 ($\leq 0,01$ 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 $\pm 0,2$ °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 $24,0 \pm 0,2$ °C and/or $50,0 \pm 0,2$ °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 $\times$ 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 $100 \pm 0,5$ 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 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.

d) When the mixing jar, containing 100 ml of water at 24 °C with or without the addition of an appropriate test portion (8.2), 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 ADML 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 other 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.

6.9 Interval timer, indicating 0 to 60 s and 0 to 60 min.

6.10 Spoon spatula(s), of length approximately 210 mm.

6.11 Centrifuge tubes, glass, conical, with the shape, dimensions, graduation scheme, inscriptions and patch of matt surface shown in figure 2, and provided with rubber stoppers. The graduation lines, graduation numbers and the inscription "ml (20 °C)" shall be marked in a permanent manner and the graduation lines shall be fine and clean. The maximum errors ($\pm$) in capacity at 20 °C shall be as follows :

- at 0,1 ml : $\pm 0,05 \text{ ml}$;
- from 0,1 to 1 ml inclusive : $\pm 0,1 \text{ ml}$;
- from 1 to 2 ml inclusive : $\pm 0,2 \text{ ml}$;
- from 2 to 5 ml inclusive : $\pm 0,3 \text{ ml}$;
- from 5 to 10 ml : $\pm 0,5 \text{ ml}$;
- at 10 ml : $\pm 1 \text{ ml}$

NOTE — For routine production control purposes, tubes of different dimensions may be used provided that they comply with limits for maximum error in capacity, as listed above. In all cases of dispute or where a definitive result is required, the tubes specified in 6.11 should be used.

6.12 Centrifuge, electric, with speed indicator (min^{-1} or r/min), with vertical-loading swing-out cups for accommodating the centrifuge tubes (6.11), and capable of producing an acceleration of $160 g_n$ at the internal bottom of the tubes and maintaining a temperature of 20 to 25 °C within the closed centrifuge.

NOTE — The acceleration, in terms of g_n , produced in a centrifuge is equal to

$$1,12 r n^2 \times 10^{-6}$$

where

- r is the effective horizontal radius of spinning, in millimetres;
- n is the rotational frequency, per minute.

6.13 Siphon fitting or suction tube attached to water pump, for removing supernatant liquid from a centrifuge tube (6.11), made of glass tubing and with upturned tip. A suitable siphon fitting is shown in figure 2.

6.14 Stirring rod, made of glass, of length 250 mm and diameter 3,5 mm.

6.15 Magnifying lens, suitable for aiding the reading of the volume of sediment (see 8.13).

7 Sampling

Take the laboratory sample in accordance with ISO 707 and, if necessary, store it in a clean, dry, securely closed, airtight and preferably opaque container, which may be the intact unopened retail container. If the laboratory sample is in a transparent container, keep the container in the dark.

Note and report any deviations from these requirements.

8 Procedure (see also clause 10, and 10.3 in particular)

8.1 Preparation of the test sample

Before proceeding with the determination, ensure that the laboratory sample (clause 7) has been maintained at laboratory temperature (20 to 25 °C) for at least 48 h so that any influence on insolubility index attributable to the physical state of the fat is constant from sample to sample. Then thoroughly mix the laboratory sample by repeatedly rotating and inverting the container. If the container is too full to allow thorough mixing, transfer all the laboratory sample to a clean, dry, airtight container of adequate capacity and mix as described.

In the case of instant dried milk, mixing shall be performed very gently to avoid reducing the particle size of the sample.

8.2 Preparation of the mixing jar

According to whether the insolubility index is to be measured at 24 °C or at 50 °C (see 0.2), adjust the temperature of the mixing jar (6.3) to $24,0 \pm 0,2 \text{ °C}$ or $50,0 \pm 0,2 \text{ °C}$, respectively, by standing the jar in the water bath (6.2), with the water level near the top of the jar, for a sufficient period of time.

NOTE — In the remainder of the text, the phrase "at $24,0 \pm 0,2 \text{ °C}$ or $50,0 \pm 0,2 \text{ °C}$ as appropriate" means whichever of these temperatures was adopted in this subclause.

8.3 Test portion

Weigh, to the nearest $\pm 0,01 \text{ g}$, in the scoop (6.4) or on the sampling paper (6.4), a test portion of

- a) 13,00 g in the case of dried whole milk, dried partly skimmed milk and infant food based on either;

b) 10,00 g in the case of dried skimmed milk and dried buttermilk;

c) 7,00 g in the case of dried whey.

(See 10.4.)

8.4 Determination

8.4.1 Remove the mixing jar (see 8.2), from the water bath, quickly wipe dry the outside of the jar, and, using the measuring cylinder (6.6), add to the jar $100 \pm 0,5$ ml of water at $24,0 \pm 0,2$ °C or $50,0 \pm 0,2$ °C as appropriate (see the note to 8.2).

8.4.2 Add 3 drops of the silicone antifoaming agent (5.1) to the water in the mixing jar and then transfer the test portion (8.3) to the jar, using the brush (6.7) if necessary so that all of the test portion falls onto the surface of the water.

8.4.3 Fit the mixing jar to the mixer (6.8), switch on the mixer, and after the mixer has operated for exactly 90 s, switch it off. If the mixer is of the type with a non-synchronous motor, speed control and speed indicator, bring the rotational frequency of the impeller to $3\,600 \pm 100 \text{ min}^{-1}$ within the first 5 s of the 90 s mixing period.

8.4.4 Remove the mixing jar from the mixer (allow a few seconds for liquid on the impeller to drain into the jar), and leave the jar to stand, at laboratory temperature (see 10.2), for not less than 5 min and not more than 15 min (see 10.3).

8.4.5 Add 3 drops of the silicone antifoaming agent to the mixture in the mixing jar (see 10.5), thoroughly mix the contents of the jar by stirring (not too vigorously) for 10 s with a spoon spatula (6.10) and immediately pour sufficient of the mixture into a centrifuge tube (6.11) to fill the tube up to the 50 ml mark, i.e. until the top level is coincident with the 50 ml mark.

8.4.6 Place the centrifuge tube (counterbalanced) in the centrifuge (6.12), bring the centrifuge as quickly as possible to the rotational frequency producing an acceleration of $160 g_n$ at the internal bottom of the tube and then spin the tube at this rotational frequency for 5 min, at 20 to 25 °C.

8.4.7 Remove the centrifuge tube from the centrifuge and, using a spoon spatula (6.10), remove and discard any top layer of fatty material in the tube.

Hold the centrifuge tube in a vertical position and remove the supernatant liquid with a siphon fitting (6.13) or a suction tube (6.13) until the top level is coincident with the 15 ml mark if a roller-dried product is being tested or coincident with the 10 ml mark if a spray-dried product is being tested, taking care not to disturb the sediment. However, if it is evident that the volume of sediment exceeds 15 ml or 10 ml, respectively, discontinue the procedure at this stage and record the insolubility index as "15 ml" or "greater than 10 ml", indicating the reconstituting temperature as shown in 9.1. Otherwise, proceed as described in 8.4.8.

8.4.8 Add water at 24 °C or 50 °C as appropriate (see the note to 8.2) to the centrifuge tube until the top level is coincident with the 30 ml mark, completely disperse the sediment with the stirring rod (6.14), tap the bottom of the rod against the inside of the tube to collect adhering liquid, and add more water at the same temperature until the top level is coincident with the 50 ml mark.

8.4.9 Close the centrifuge tube with a rubber stopper, invert the tube slowly five times so as to mix its contents thoroughly, remove the stopper (draw the bottom of the stopper across the rim of the tube to collect adhering liquid), and then spin the tube in the centrifuge for 5 min at the required rotational frequency and temperature, as described in 8.4.6.

NOTE — It is recommended that the centrifuge tube be placed in the centrifuge cup so that, when the tube is in the spinning position, the scale lines are not facing upwards or downwards but are midway between these two positions. Then, if the top of the sediment is sloping, the volume of sediment will be easier to estimate.

8.4.10 Remove the centrifuge tube from the centrifuge, hold the tube in a vertical position against a suitable background (see the note) with the top of the sediment at eye level, and, using the magnifying lens (6.15), read the volume of sediment to the nearest 0,05 ml if the volume is less than 0,5 ml and to 0,1 ml if the volume is more than 0,5 ml. Estimate the volume if the top of the sediment is sloping. If the top of the sediment is uneven, leave the tube in a vertical position for a few minutes; the top of the sediment will usually become less uneven and the volume of sediment can be read more easily. Record the temperature of the water used for the reconstitution.

NOTE — Viewing the tube against either a light or a dark background, as preferred, makes the top of the sediment more distinct.

9 Expression of results

9.1 Method of calculation

The insolubility index of the laboratory sample is equal to the volume, in millilitres, of sediment recorded in 8.4.10. Report the result with the temperature of the water used for reconstitution, for example as follows :

0,10 ml (24 °C)

4,1 ml (50 °C)

9.2 Precision

NOTE — The values of repeatability and reproducibility were determined in an inter-laboratory test, as described in ISO 5725, *Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests*, involving 10 laboratories and 10 samples.

9.2.1 Repeatability

The difference between two single values for insolubility index, obtained simultaneously or within a short time-interval by one analyst on the same test sample using the same apparatus, should not exceed $0,138 M$, where M is the mean of the two values.

9.2.2 Reproducibility

The difference between two single values for insolubility index, obtained by two analysts in different laboratories on test samples from the same laboratory sample, should not exceed $0,328 M$, where M is the mean of the two values.

10 Notes on procedure

10.1 It is essential that the procedure, once started, be completed without unnecessary delay at any stage and that all requirements relating to temperature and timing be strictly observed.

10.2 Since the determination of insolubility index may be affected by ambient temperature, it is recommended that the procedure be carried out in a laboratory in which the temperature is maintained in the range 20 to 25 °C.

10.3 Tests have shown that the permitted 5 to 15 min variation in "standing" time (8.4.4) has an insignificant effect on the insolubility index. This range of 10 min allows several samples, preferably requiring the same reconstitution temperature, to be tested as a batch, provided that the temperature of each mixing jar is adjusted simultaneously (see 8.2) and the test portions (8.3) are all weighed together. In these circumstances, it may be found advantageous to modify the procedure described in 8.2 and 8.4.1 by adding $100 \pm 0,5$ ml of water (at approximately the temperature required) to each of the mixing jars in the water bath and when

the temperature of the water has stabilized at the correct value, to remove one jar from the water bath and proceed as described in 8.4.1 to 8.4.4 inclusive, and so on with each of the other jars in turn. Thereafter, a batch procedure can be adopted, with simultaneous centrifuging of the tubes.

10.4 Each test portion (8.3) is such that when mixed with 100 ml of water the total solids content, expressed as a percentage by mass of the mixture, approximates to that of the original liquid product. The sizes of the test portions for dried whole milk, dried skimmed milk and dried buttermilk are the same as specified in the solubility index method^[3], which deals only with these three products.

10.5 The addition of 3 drops of the silicone antifoaming agent (5.1) in 8.4.5 may not appear necessary with samples whose propensity to foam during mixing is less than others. Nevertheless, the 3 drops should always be added to keep the procedure exactly the same for all samples.

11 Test report

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, together with any circumstances that may have influenced the result.

The test report shall include all details required for the complete identification of the sample.

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Dimensions in millimetres

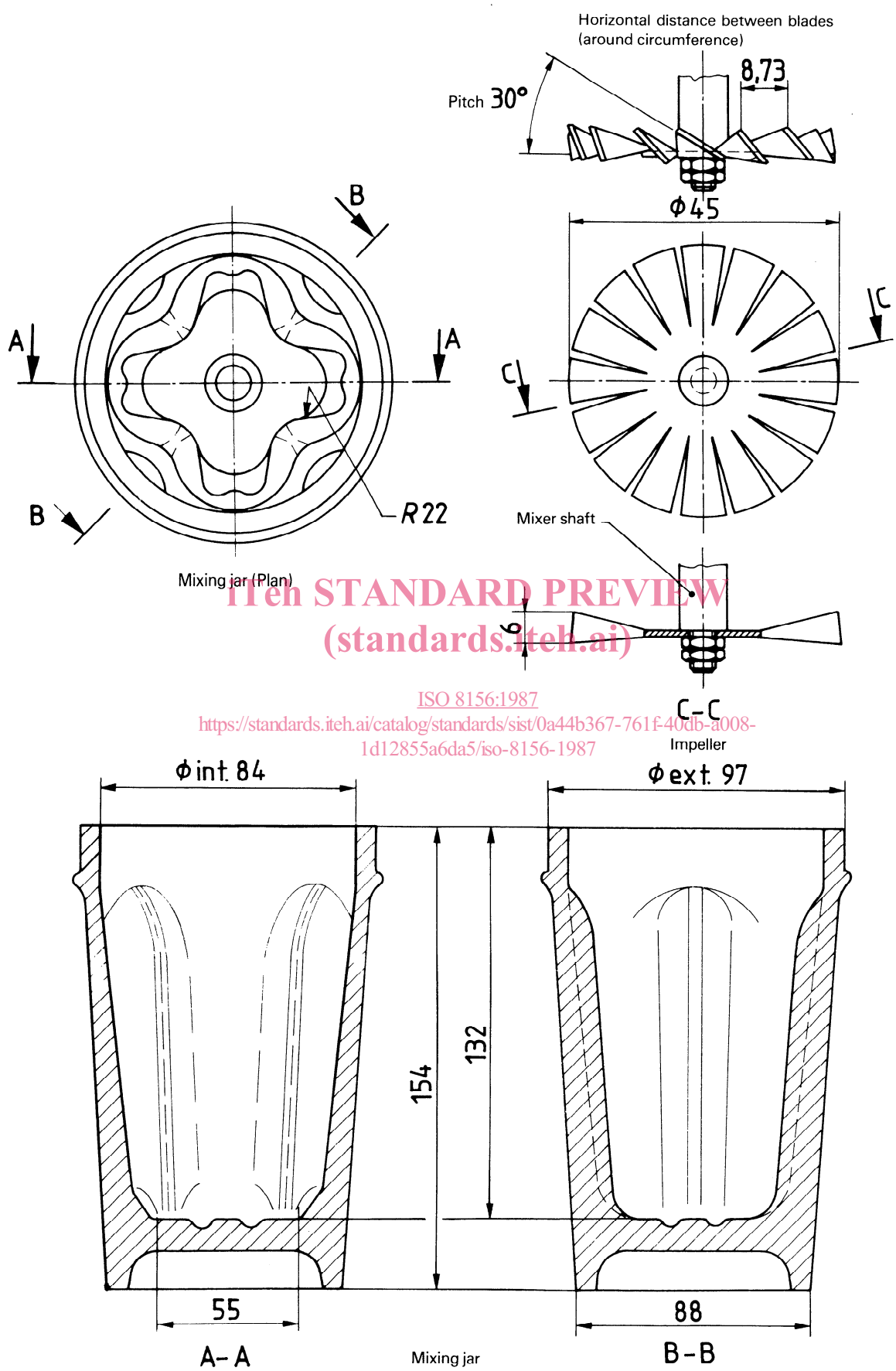


Figure 1 — Mixing jar and impeller

Dimensions in millimetres

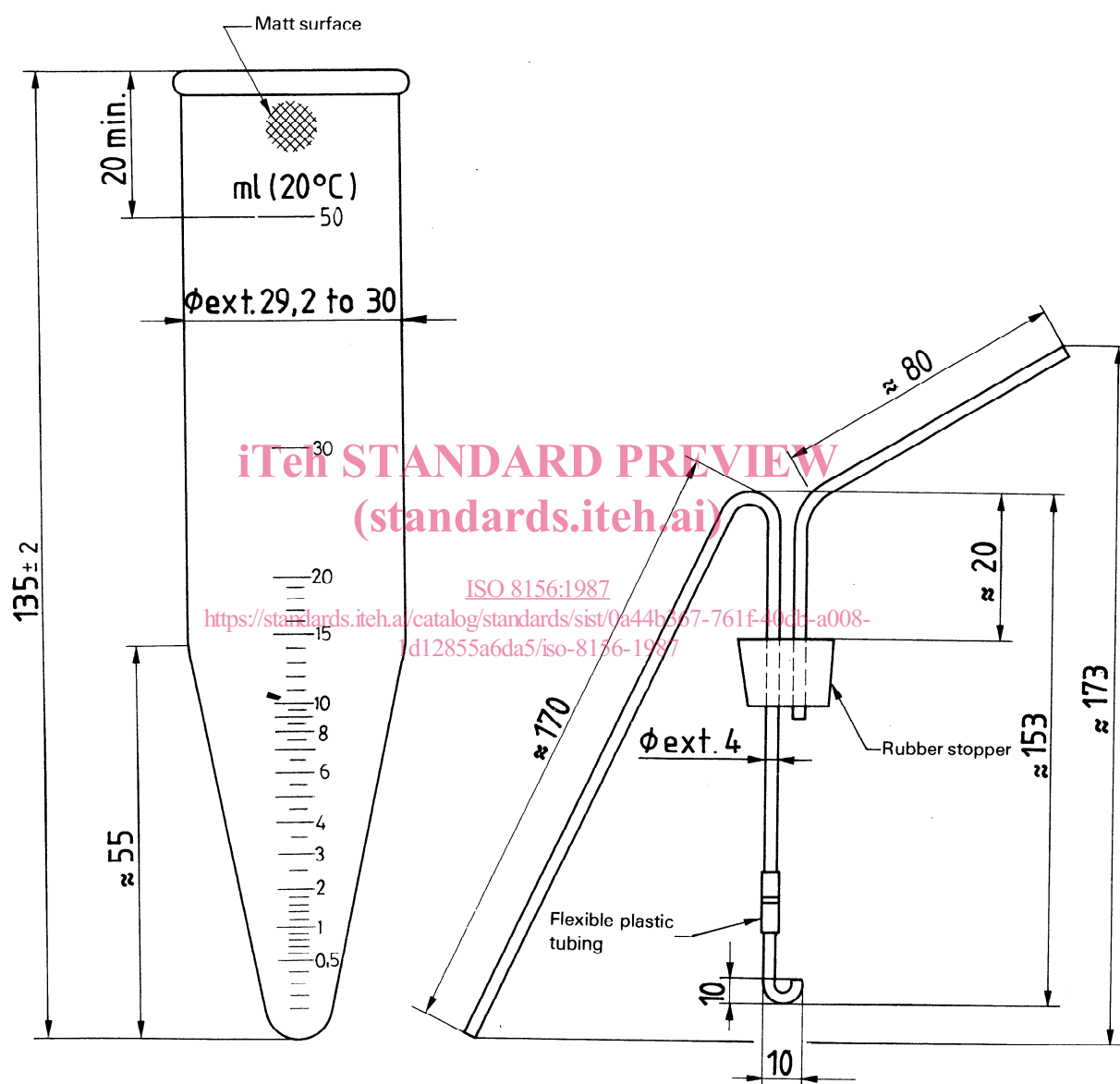


Figure 2 — Centrifuge tube and siphon fitting