INTERNATIONAL STANDARD

ISO 15322

IDF 203

First edition
2005-04-01

Dried milk and dried milk products — Determination of their behaviour in hot coffee (Coffee test)

Lait sec et produits laitiers secs — Détermination de leur comportement dans le café chaud (Essai du café)

Reference numbers
ISO 15322:2005(E)
IDF 203:2005(E)

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Published in Switzerland

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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 15322 | IDF 203 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF), in collaboration with AOAC International. It is being published jointly by ISO and IDF and separately by AOAC International.

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 and AOAC International 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 National Committees casting a vote.

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All work was carried out by the Joint ISO/IDF/AOAC Action Team *Physical properties and rheological tests*, of the Standing Committee on *Minor components and characterization of physical properties*, under the aegis of its project leader, Mr E. Refstrup (DK).

Introduction

Milk powder, particularly instant or agglomerated whole milk powder, is often used as coffee whitener by consumers and is thus reconstituted directly in hot coffee. Hence the behaviour of the milk powder in the hot coffee has become an increasingly important functional and aesthetic property. Some powders will leave floating particles or clusters of particles on the surface, while others will partially coagulate in the hot, acidic environments and result in sediment or sludge in the bottom of the cup.

The behaviour in hot coffee can be influenced by certain compositional and technological parameters, such as protein content, addition of stabilizing salts and preheat treatment of the milk. The temperature and pH of the coffee and the hardness of the water used to prepare the coffee are also of significance.

Simple methods for the determination of floaters have been described [for example in *Analytical Methods for Dry Milk Products*, 1978, using the A/S Niro Atomizer, Søborg, Denmark[1)]].

NOTE The method in this International Standard is based on a method developed at the New Zealand Dairy Research Institute, Palmerston North, New Zealand.

1) A/S Niro Atomizer, Søborg, Denmark, is the trade name of a supplier.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO or IDF of this product.

Dried milk and dried milk products — Determination of their behaviour in hot coffee (Coffee test)

1 Scope

This International Standard specifies a method for the determination of the behaviour in hot coffee of dried milk and dried milk products, either instant or non-instant.

2 Normative references

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

ISO 8156|IDF 129, *Dried milk and dried milk products — Determination of insolubility index*

3 Terms and definitions

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

3.1
coffee test
amount of sediment determined by the procedure specified in this International Standard

NOTE The results are expressed in millilitres.

4 Principle

A test portion is added to coffee preheated to the required temperature. The mixture is stirred manually with a special spatula in a specified way and for a certain time. After a specified standing period, the mixture is centrifuged in two graduated tubes. The sum of the volume of sediment in the two tubes is recorded.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified, and distilled or demineralized water or water of equivalent purity. The reagents shall leave no appreciable residue when the determination is carried out by the method specified.

5.1 Instant coffee powder

The chemical composition and, in particular, the acidity of the applied coffee may affect the result of the coffee test. For routine control purposes, therefore, always use the same brand of coffee.

Store the instant coffee powder in a clean, dry, securely closed, airtight and preferably opaque container, which may also be an unopened container at room temperature for not longer than 6 months.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 **Analytical balance**, capable of weighing to the nearest 0,01 g.

6.2 **Thermometer**, capable of measuring up to 100 °C, with accuracy ± 0,5 °C. Suitable thermometers are of the thermocouple or thermistor type.

6.3 **Scoop**, with a smooth surface, suitable for weighing out the test portion.

6.4 **Glass beaker**, with spout, of capacity 250 ml, graduated at 100 ml.

6.5 **Spatula**, of stainless steel, of overall length 250 mm, with blade of length 135 mm and width 25 mm (see ISO 8156).

6.6 **Centrifuge**, electric, with a speed indicator (min^{-1} or rev/min), with vertical-loading swing-out cups for accommodating the graduated centrifuge tubes (6.7), capable of producing a radial acceleration of 160g at the inside bottom of the tubes (see ISO 8156).

6.7 **Graduated centrifuge tubes**, made of glass, with conical shape, of capacity 50 ml (see ISO 8156).

6.8 **Stopwatch**, capable of indicating time intervals of 1 s or less.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 707.

If necessary, store the test sample in a clean, dry, securely closed, airtight and preferably opaque container, which may be an unopened container.

8 Preparation of test sample

Keep the test sample at room temperature (20 °C to 25 °C). Thoroughly mix the sample while avoiding breaking the particles, by repeatedly rotating and inverting the container.

The sample container shall not be more than two-thirds full. If it is too full, transfer all the test sample to a clean, airtight container of adequate capacity, then mix as described.

9 Procedure

9.1 Carry out the test method in duplicate to obtain two single values for the coffee test.

9.2 Weigh, to the nearest 0,01 g, 0,79 g to 0,81 g of instant coffee powder (5.1) in a dry 250 ml glass beaker (6.4).

9.3 Weigh, to the nearest 0,01 g, 1,19 g to 1,21 g of test sample (Clause 8) in the scoop (6.3).

9.4 Add boiling water to the coffee powder in the 250 ml glass beaker (9.2). Make up to the 100 ml mark with boiling water.

9.5 Check the temperature of the reconstituted coffee with the thermometer (6.2). When it has reached 80 °C ± 0,5 °C, tip in the weighed amount of test portion (9.3) and start the stopwatch (6.8).

Carry out the test with a coffee brand suitable for a temperature of 80 °C. The coffee brand and the temperature should be harmonized in order to make the test so strict that not all powders used show no or almost no flocculation, neither do all powders settle (largely or) completely.

9.6 After 5 s, stir the contents of the beaker gently with the spatula (6.5). Stir six times clockwise and six times counter-clockwise over 6 s. Use circular movements of the spatula, following the side of the beaker. The spatula shall also touch the bottom of the beaker at all times.

9.7 After completion of stirring, allow the contents of the beaker to stand for 10 min.

9.8 Give the contents of the beaker one stir then immediately pour the contents into two 50 ml centrifuge tubes (6.7). Allow the centrifuge tubes to stand for at least 5 min.

9.9 Place the centrifuge tubes in the centrifuge (6.6). Centrifuge with fast acceleration, at a rotational frequency producing a radial acceleration of 160g at the inside bottom of the tubes, for 5 min at between 20 °C and 25 °C and atmospheric pressure.

9.10 Remove the centrifuge tubes from the centrifuge. Hold each tube in a vertical position against a suitable background with the top of the sediment at eye level. If the volume is less than 0,5 ml, read the volume of sediment in each tube to the nearest 0,05 ml. If the volume is equal to or more than 0,5 ml, read the sediment volume in each tube to the nearest 0,1 ml.

10 Calculation and expression of results

10.1 Calculation

Calculate the volume of the sediment of the test sample, V_s, in millilitres, using the following equation:

$$V_s = V_1 + V_2$$

where

V_1 is the volume of the sediment in the first centrifuge tube (9.10), in millilitres;

V_2 is the volume of the sediment in the second centrifuge tube (9.10), in millilitres.

10.2 Expression of results

Express the test results to two decimal places if the volume of the sediment is less than 0,5 ml, and to one decimal place if the volume of the sediment is equal to or more than 0,5 ml.

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A. The values derived from this interlaboratory test may not be applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than 0,15 ml.

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than 0,65 ml.

12 Test report

The test report shall specify:

a) all information required for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, together with reference to this International Standard;

d) all operating details not specified in this International Standard, or regarded as optional, together with details of any incident that may have influenced the results (e.g. floating particles, colour and oiling off, and the pH of the reconstituted coffee);

e) the test result(s) obtained, and if the repeatability has been checked, the final quoted results obtained.

Annex A
(informative)

Results of interlaboratory test

An international collaborative test was organized involving seven laboratories on test samples of 10 types of dried milk and dried milk products. The test samples were divided again into 20 duplicated samples. The results obtained were subjected to statistical analysis in accordance with ISO 5725 [2)] to give the precision data shown in Table A.1.

NOTE 1 ISO 5725:1986 was not followed exactly as the test was carried out with only seven laboratories. Due to the amount of test samples, the test result was considered as being reliable.

NOTE 2 The values for repeatability and reproducibility limits also tend to demonstrate somewhat higher figures for the obtained largest mean values as shown in Table A.1.

Table A.1 — Results of interlaboratory test

	Samples										
	A	B	C	D	E	F	G	H	I	J	Mean
Number of participating laboratories	7	7	7	7	7	7	7	7	7	7	
Mean value (ml)	0,101	0,288	0,247	0,286	0,350	0,369	1,543	0,321	0,233	0,991	0,473
Repeatability standard deviation, s_r (ml)	0,006	0,014	0,019	0,009	0,033	0,000	0,089	0,076	0,031	0,091	0,050
Coefficient of variation of repeatability (%)	16,61	14,00	21,41	9,07	26,19	0,00	16,20	66,12	37,75	25,60	29,41
Repeatability limit, r (= 2,8 s_r) (ml)	0,017	0,040	0,053	0,026	0,092	0,000	0,250	0,212	0,088	0,254	0,139
Reproducibility standard deviation, s_R (ml)	0,047	0,121	0,115	0,122	0,084	0,147	0,445	0,095	0,128	0,486	0,231
Coefficient of variation of reproducibility (%)	131,5	117,6	130,3	119,4	66,90	111,6	80,69	82,81	154,3	137,2	136,7
Reproducibility limit, R (= 2,8 s_R) (ml)	0,133	0,339	0,322	0,341	0,234	0,411	1,245	0,256	0,359	1,361	0,646

2) ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory test* (now withdrawn), was used to obtain the precision data.