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Durum wheat semolinas — Determination of the undersize fraction

Semoules de blé dur — Détermination du taux d'affleurement

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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 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15793 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

Annex A of this International Standard is for information only.

Durum wheat semolinas — Determination of the undersize fraction

1 Scope

This International Standard specifies a method for the determination of the undersize fraction of durum wheat semolinas, which is an important characteristic.

It is applicable to samples of at least 1 kg of durum wheat semolina obtained solely by comminution, without subsequent combination, having a moisture content higher than 10 % and packaged in fully filled airtight packaging.

It is not applicable to determinations carried out using sieves of aperture size below 160 μm.

NOTE Determination of the undersize fraction is sometimes termed "granulation of semolinas".

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 712, *Cereals and cereal products — Determination of moisture content — Routine reference method.*

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
undersize fraction of semolina
by convention, the percentage of semolina which passes through the sieve specified for the semolina in question, under the operating conditions described in this International Standard

4 Principle

The semolina under test is sieved using a chosen sieve of specified aperture size, in accordance with the procedure described in this International Standard.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Laboratory sieving machine[1)], capable of being set at 200 r/min ± 5 r/min as well as at sieving amplitudes of 30 mm ± 2 mm and 60 mm ± 2 mm, and equipped with a sieve blocking system.

5.2 Circular sieve, with sieving medium, cover and receiver, in stainless steel or similar material, having the following characteristics:

a) frame diameter: 200 mm;

b) useful diameter of sieving medium: 185 mm ± 5 mm, corresponding to a sieving surface area of approximately 270 cm^2;

c) height: 20 mm minimum;

d) aperture size (or void): the following list gives examples of aperture sizes[2)] of sieving media currently used in European and international transactions:

— 160 μm ± 6,9 μm,

— 180 μm ± 7,6 μm,

— 200 μm ± 8,3 μm,

— 250 μm ± 9,9 μm,

— 300 μm ± 11 μm,

— 315 μm ± 12 μm,

— 425 μm ± 15 μm,

— 500 μm ± 18 μm.

5.3 Rubber balls (eight), having a diameter of 18 mm ± 1 mm and a mass of 5 g ± 0,5 g.

5.4 Spatula.

5.5 Soft-haired brush.

5.6 Flat container, having a capacity of approximately 2 litres, made of a non-electrostatic material.

5.7 Dishes (two), of suitable dimensions, made of a non-electrostatic material.

5.8 Analytical balance, capable of being read to the nearest 0,001 g.

5.9 Chronometer, capable of being read to the nearest 1 s.

6 Sampling

Sampling is not a part of the method specified in this International Standard. A recommended sampling method is given in ISO 13690 [2].

1) ROTACHOC is an example of a suitable apparatus and is supplied by Tripette et Renaud (France). This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this apparatus. Equivalent apparatus may be used if it can be shown to give the same results.

2) The tolerances are as given in ISO 3310-1 [1].

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

7 Preparation of test sample

Pour the entire contents of a package of approximately 1 kg of semolina into the container (5.6). Mix it carefully in order to make it as homogeneous as possible. The homogenization may be carried out by a mixing device if its container is made of non-electrostatic material.

8 Determination of moisture content of the sample

Determine separately the moisture content of the prepared test sample (clause 7) in accordance with ISO 712.

The moisture content shall be between 10 % and 15,50 %.

If this is not the case, the procedure described in clause 9 is not applicable (in accordance with clause 1).

9 Procedure

9.1 General

The operations described in 9.2 to 9.3.8 inclusive shall be carried out in less than 15 min.

9.2 Test portion

From the test sample prepared according to clause 7, immediately take, using a spatula (5.4), a test portion of 50 g $\pm$ 0,5 g, weighed to the nearest 0,01 g (m_0).

9.3 Determination

9.3.1 Place the sieve, appropriate for the type of semolina to be tested, on the receiver (5.2) and put eight rubber balls (5.3) in the sieve.

9.3.2 Pour the test portion (9.2) into the sieve and put on the cover. Place the assembly on the sieving machine (5.1) which is set at an amplitude of 60 mm. Block using the tightening system and disengage the sieve cleaning system if the appliance is equipped with one.

9.3.3 Start the chronometer (5.9) and the sieving machine (5.1) simultaneously. Sieve the test portion for 5 min $\pm$ 5 s.

9.3.4 As soon as the sieving machine stops, release the tightening system and remove the assembly.

9.3.5 Remove the cover. Brush off (5.5) the particles of semolina adhering to the underside of the sieve into the receiver to avoid loss.

9.3.6 Discard the semolina remaining on the sieve.

9.3.7 Transfer quantitatively the contents of the receiver (the undersize fraction) into a dish (5.7), then weigh to the nearest 0,01 g (m).

9.3.8 Conduct a new determination by sampling another test portion (9.2) and proceeding as described in 9.3.1 to 9.3.7.

9.4 Calculation

Calculate the undersize fraction (T) of each test portion of the semolina according to the following equation:

$$T = \frac{m}{m_0} \times 100\ \%$$

where

m is the mass, in grams, of the undersize fraction obtained;

m_0 is the mass, in grams, of the test portion.

Calculate the arithmetic mean of the two determinations and express the results to the nearest 0,01 %.

10 Expression of results

10.1 Examine the repeatability of the measurements by taking the following criteria into consideration.

a) If the mean value is higher than 15 %

Accept the result only if both values do not differ by more than ± 1,0 % (i.e. ± 0,50 g).

b) If the mean value is lower than 15 %

Accept the result only if both values do not differ by more than ± 0,5 % (i.e. ± 0,25 g).

If the result does not meet the criteria given in a) and b), repeat all of the operations with two other test portions.

10.2 If the repeatability conditions are fulfilled, examine whether the mean value obtained is higher or lower than 15 % (i.e. 7,5 g).

a) If the mean value is higher than 15 %

Accept this value as the final result of the test and express it to the nearest 0,1 %.

b) If the mean value is lower than 15 %

Repeat the operations with two other test portions following the same procedure as previously described (see 9.2 to 9.4), but modifying the operating conditions as follows:

— in 9.2: using a spatula (5.4), take a test portion of 100 g ± 0,50 g, weighed to the nearest 0,01 g;

— in 9.3.1: put four rubber balls (5.3) into the sieve;

— in 9.3.2: set the sieving machine (5.1) at an amplitude of 30 mm ± 2 mm.

Proceed as before on two test portions. Calculate the arithmetical mean of the two results and express it to the nearest 0,01 %.

Examine the repeatability of the measurements taking the following criteria into consideration.

1) **If the mean value is higher than 15 %:** accept the result only if both values do not differ by more than ± 1,0 % (i.e. ± 1,0 g).

2) **If the mean value is lower than 15 %:** accept the result only if both values do not differ by more than ± 0,5 % (i.e. ± 0,50 g).

10.3 If the result does not meet these criteria, repeat all of the operations with two other test portions proceeding as described in 10.2 b). Otherwise, take the arithmetical mean as the final result and express it to the nearest 0,01 %.

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 the values specified, as appropriate, in 10.1 or 10.2 b).

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 25 % of the arithmetic mean of the two results, with minimum and maximum tolerances of 2 g and 10 g respectively.

NOTE These values correspond to what can be currently obtained, account being taken of the "rules of the game". Indeed, if a certain scattering of results is observed, it is not solely due to the method, but also results from the tolerances authorized for the sieve aperture sizes.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used and the aperture size chosen, with reference to this International Standard;

— all operating details not specified in this International Standard, or regarded as optional, together with details or any incidents which may have influenced the test result(s);

— the test result(s) obtained or, if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of an interlaboratory test

An interlaboratory test was carried out involving nine participating laboratories. This analysis was conducted on five durum wheat semolinas having different proportions of undersize fractions. The determinations were carried out using 160 μm and 250 μm aperture size sieves.

Each laboratory received samples of five semolinas. Four determinations were carried out on each sample using either a 160 μm or a 250 μm aperture size sieve.

As the mean values show, in the case of the 160 μm sieve with semolinas G, J and A, and in the case of the 250 μm sieve with semolinas D and G, the tests had to be repeated with the conditions specified in 10.2 b). In the case of the other samples this was not necessary and the tests were carried out only as specified in 9.2 to 9.3.

The results were analysed statistically according to ISO 5725-1 [3] and ISO 5725-2 [4].

Table A.1 — Statistical results for durum wheat semolinas

	160 μm sieve				250 μm sieve			
	Sample				Sample			
	G	J	A	K	D	G	J	K
Number of participating laboratories	9	9	6	9	6	9	9	9
Number of laboratories selected	7	6	5	6	5	9	9	8
Mean value, %	2,60	12,62	13,35	58,84	0,38	11,81	32,75	92,30
Standard deviation of repeatability, s_r, %	0,09	0,19	0,14	0,11	0,07	0,24	0,43	0,36
Coefficient of variation of repeatability, %	3,36	1,50	1,04	0,18	17,89	2,05	1,33	0,39
Repeatability limit, r [2,83 s_r]	0,24	0,53	0,39	0,29	0,19	0,67	1,20	0,99
Standard deviation of reproducibility, s_R, %	0,24	0,64	0,44	0,77	0,17	2,87	2,68	3,07
Coefficient of variation of reproducibility, %	9,15	5,04	3,29	1,30	44,42	24,29	8,18	3,32
Reproducibility limit, R [2,83 s_R]	0,66	1,76	1,22	2,12	0,46	7,95	7,42	8,50

Bibliography

[1] ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth.*

[2] ISO 13690, *Cereals, pulses and milled products — Sampling of static batches.*

[3] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[4] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

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