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Wheat, rye and their flours, durum wheat and durum wheat semolina — Determination of the falling number according to Hagberg-Perten

Blés tendres, seigles et leurs farines, blés durs et leurs semoules — Détermination de l'indice de chute selon Hagberg-Perten

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Contents

Page

Foreword

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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.

ISO draws attention to the fact that it is claimed that compliance with this document may involve the use of a patent concerning the falling number apparatus specified in 6.1.

ISO takes no position concerning the evidence, validity and scope of this patent right.

The holder of this patent right has assured the ISO that he is willing to negotiate licences under reasonable and non-discriminatory terms and conditions with applicants throughout the world. In this respect, the statement of the holder of this patent right is registered with the ISO. Information may be obtained from:

Perten Instruments AB
P.O. Box 5101
S-141 05 HUDDINGE
Sweden

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights other than those identified above. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 3093 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*.

This fourth edition cancels and replaces the third edition (ISO 3093:2004), of which it constitutes a minor revision. It also incorporates the Technical Corrigendum, ISO 3093:2004/Cor.1:2008.

Wheat, rye and their flours, durum wheat and durum wheat semolina — Determination of the falling number according to Hagberg-Perten

1 Scope

This International Standard specifies the determination of the α-amylase activity of cereals by the falling number (FN) method according to Hagberg-Perten.

This method is applicable to cereal grains, in particular to wheat and rye and their flours, durum wheat and its semolina.

This method is not applicable to the determination of low levels of α-amylase activity.

By converting the FN into a liquefaction number (LN), it is possible to use this method to estimate the composition of mixtures of grain, flour or semolina with known FNs necessary to produce a sample of a required FN.

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 712, *Cereals and cereal products — Determination of moisture content — Reference method*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

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

3.1
falling number
FN
t
total time required to activate a viscometer stirrer and allow it to fall a predetermined distance through an aqueous gel prepared from heating a mixture of flour or semolina, and water in a viscometer tube, and which is undergoing liquefaction due to attack by the enzyme α-amylase

NOTE 1 Time is counted from immersion in the water bath.

NOTE 2 The falling number is expressed in seconds.

3.2
liquefaction number
LN
n_L
result of a simple calculation to convert the **falling number** (3.1) into a value used to estimate the composition of mixtures of grain, flour or semolina necessary to produce a sample of the required falling number

NOTE LNs, unlike FNs, are additive.

4 Principle

The α-amylase activity is estimated using the starch present in the sample as a substrate. The determination is based on the ability of an aqueous suspension of flour, semolina or wholemeal cereal product to gelatinize rapidly in a boiling water bath, and on the measurement of starch liquefaction by the α-amylase present in the sample.

Liquefaction affects the thickness of the starch gel and, hence, the resistance to the viscometer stirrer and the time taken for it to fall a defined distance.

5 Reagents

5.1 Water, produced by distillation or demineralization, complying with ISO 3696, grade 3.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Apparatus for FN determination[1], comprising the following components.

6.1.1 Water bath, with integral heating unit, cooling system and water level indicator.

6.1.2 Electronic timer.

6.1.3 Viscometer stirrer, metallic, able to move freely within the ebonite stopper.

Its rods shall be straight and the wheels free from distortion and wear.

6.1.4 Precision viscometer tubes, manufactured from special glass, with the following dimensions:

— inner diameter: 21,00 mm ± 0,02 mm;

— outer diameter: 23,80 mm ± 0,25 mm;

— inner height: 220,0 mm ± 0,3 mm.

6.1.5 Rubber stoppers, to fit the viscometer tubes.

6.2 Automatic dispenser or **pipette**, ISO 8655-2[4], allowing a volume of 25,0 ml ± 0,2 ml to be delivered.

1) The “Falling Number” apparatus having a specifically designed viscometer stirrer produced by Perten Instruments is an example of a suitable device available commercially. This device forms the subject of a patent. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

6.3 Analytical balance, capable of being read to the nearest 0,01 g.

6.4 Laboratory mill[2], hammer type, and fitted with a 0,8 mm screen allowing the production of a wholemeal product meeting the particle size specification shown in 8.1.3.

Check the performance of the mill periodically using a well-mixed sample of ground grain (as produced in 8.1.2).

The mill may be equipped with an automatic feeding device, particularly for the grinding of high moisture content grain.

6.5 Laboratory sieve, of nominal size of openings 800 µm, ISO 565 [1] and ISO 3310 (all parts)[2].

7 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 13690[5].

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

Storage time and storage conditions of the sample in the laboratory may have a significant effect on FNs.

8 Preparation of test sample

8.1 Whole grain

8.1.1 Elimination of impurities

If necessary, clean the sample to eliminate impurities (e.g. stones, dust, husks and other cereal grains). Take a representative 300 g test sample from the laboratory sample.

A smaller test sample of about 200 g, although it provides less reproducible results, may be used for routine inspections. If the sample is less than 200 g, the results risk being marred by errors.

8.1.2 Grinding of grain samples

Feed the laboratory mill (6.4) carefully with grain to avoid heating and overloading. The feed to the mill may be controlled automatically with an automatic feeding device. Grinding should be continued for 30 s to 40 s after the last of the sample has entered the mill. Discard the bran particles remaining inside the mill, provided these do not represent more than 1 % of the quantity of grain sampled for grinding. Thoroughly mix all of the milled product before use.

It is recommended (especially in the case of successive grindings) to allow the meal to cool for 1 h before proceeding with the test.

8.1.3 Ground sample specification

WARNING — FNs can be affected by the particle size of the ground grain.

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2) LM 3100 and LM 120 mills are examples of suitable devices available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

The milled product shall comply with the specification given in Table 1.

Table 1 — Ground sample specification

Nominal size of openings of sieve µm	Amount of ground sample passing through sieve %
710	100
500	95 to 100
200	80 or less

Check the particle size distribution of the ground material regularly using a well-mixed sample of ground grain (8.1.2).

To do this, select the appropriate sieves, as specified in Table 1, and arrange in order of decreasing nominal size of openings with a suitable sieving aid in each sieve and a receiver pan on the bottom. Weigh out a representative sample of 50,0 g and place on the top sieve. Sieve in a horizontal plane, manually for at least 5 min until nothing passes through the 710 µm sieve, or mechanically for a period of 10 min. Weigh the material retained on each sieve and that contained in the receiver pan. Calculate the percentage of ground grain which passes through each sieve.

8.2 Flour and semolina samples

Flour samples shall not contain lumps. If necessary, sift the flour using the laboratory sieve (6.5) in order to remove lumps or foreign bodies.

For coarse commercial wholemeal flours or semolina, grind a sample using the laboratory mill (6.4) in order to produce a test sample which conforms to the particle size specification shown in Table 1. Mix the ground sample thoroughly before use.

9 Procedure

9.1 Determination of the moisture content

Determine the FN on flour or ground material with a moisture content of 15 % mass fraction.

Determine the moisture content of the prepared test material (8.1 and 8.2) using the method specified in ISO 712.

Alternatively, a rapid instrumental procedure may be used (e.g. near infrared reflectance) provided this has been calibrated using ISO 712.

9.2 Test portion

Carry out the determination on two test portions simultaneously or rapidly one after the other.

Refer to Table 2, column (2) which shows the required mass of sample to be taken, at different moisture contents, in order to ensure that a constant ratio of dry matter is used for the FN determination.

If greater differentiation of the FNs is required for samples with very high α-amylase activity, as is normally the case for rye, refer to column (3).

Weigh the test portion to the nearest 0,05 g.

Table 2 — Test portion as a function of moisture content of the sample

Moisture content %	Test portion, g for a nominal mass of 7 g at 15 % moisture content	Test portion, g for a nominal mass of 9 g at 15 % moisture content	Moisture content %	Test portion, g for a nominal mass of 7 g at 15 % moisture content	Test portion, g for a nominal mass of 9 g at 15 % moisture content
(1)	(2)	(3)	(1)	(2)	(3)
9,0	6,40	8,20	13,6	6,85	8,80
9,2	6,45	8,25	13,8	6,90	8,85
9,4	6,45	8,25	14,0	6,90	8,85
9,6	6,45	8,30	14,2	6,90	8,90
9,8	6,50	8,30	14,4	6,95	8,90
10,0	6,50	8,35	14,6	6,95	8,95
10,2	6,55	8,35	14,8	7,00	8,95
10,4	6,55	8,40	15,0	7,00	9,00
10,6	6,55	8,40	15,2	7,00	9,05
10,8	6,60	8,45	15,4	7,05	9,05
11,0	6,60	8,45	15,6	7,05	9,10
11,2	6,60	8,50	15,8	7,10	9,10
11,4	6,65	8,50	16,0	7,10	9,15
11,6	6,65	8,55	16,2	7,15	9,20
11,8	6,70	8,55	16,4	7,15	9,20
12,0	6,70	8,60	16,6	7,15	9,25
12,2	6,70	8,60	16,8	7,20	9,25
12,4	6,75	8,65	17,0	7,20	9,30
12,6	6,75	8,65	17,2	7,25	9,35
12,8	6,80	8,70	17,4	7,25	9,35
13,0	6,80	8,70	17,6	7,30	9,40
13,2	6,80	8,75	17,8	7,30	9,40
13,4	6,85	8,80	18,0	7,30	9,45

9.3 Determination of falling number

9.3.1 Fill the water bath (6.1.1) with water to the level dictated by the overflow. Turn on the cooling system and ensure that cold water is flowing through the cooling lid. Switch on the FN apparatus and bring the water to the boil. The water bath shall be boiling vigorously before any determinations are carried out and during the entire test period.

9.3.2 Transfer the weighed test portion (9.2) to a clean, dry viscometer tube (6.1.4). Add 25 ml ± 0,2 ml of water (5.1) at 22 °C ± 2 °C using the automatic dispenser or pipette (6.2).

9.3.3 Immediately stopper (6.1.5) the viscometer tube (6.1.4) and shake[3)] it vigorously up and down 20 to 30 times in order to obtain a uniform suspension. Ensure that dry flour or ground material is not trapped at the top of the tube against the stopper. If necessary, free trapped material by easing the stoppers slightly upwards and reshaking as necessary.

3) The “Shake-Matic” apparatus is an example of a suitable device available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.