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Wheat — Determination of sedimentation index — Zeleny test

Blé tendre — Détermination de l'indice de sédimentation — Test de Zeleny

Reference number
ISO 5529:1992(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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

International Standard ISO 5529 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 4, *Cereals and pulses*.

This second edition cancels and replaces the first edition (ISO 5529:1978), of which it constitutes a technical revision.

This International Standard is based on Standard No. 116, *Sedimentation test (after Zeleny) to assess the milling value*, and Standard No. 118, *Experimental milling for the sedimentation test (Zeleny)*, of the International Association for Cereal Science and Technology (ICC).

Annex A forms an integral part of this International Standard.

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Wheat — Determination of sedimentation index — Zeleny test

1 Scope

This International Standard specifies a method, known as the "Zeleny sedimentation test", for assessing one of the factors determining the quality of wheat with regard to the baking strength of the flour which can be made from it.

The method is applicable only to *Triticum aestivum* wheat.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 712:1985, *Cereals and cereal products — Determination of moisture content (Routine reference method).*

ISO 2171:1980, *Cereals, pulses and derived products — Determination of ash.*

3 Definition

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

3.1 sedimentation index: The number indicating the volume, expressed in millilitres, of the sediment obtained under specified conditions from a suspension of test flour, prepared from the wheat, in a lactic acid solution.

4 Principle

Suspension of a test flour, prepared from the wheat under specified grinding and sieving conditions, in a lactic acid solution in the presence of bromophenol blue. After specified shaking and rest times, determination of the volume of the deposit resulting from the sedimentation of the flour particles.

5 Reagents

Use only reagents of recognized analytical grade, unless otherwise specified.

Use distilled water, or water of at least equivalent purity, containing less than 2 mg/kg of mineral matter.

5.1 Sedimentation test reagent

5.1.1 Lactic acid solution

Prepare a concentrated 85 % (V/V) lactic acid solution containing not more than 40 mg/kg of mineral matter.

Dilute 250 ml of this concentrated solution to 1 litre with water. Boil the dilute solution under reflux for 6 h (see note 1).

Titrate an aliquot portion of this solution with potassium hydroxide solution (for 5 ml of the lactic acid solution, about 28 ml of 0,5 mol/l potassium hydroxide solution is necessary). The concentration found shall be between 2,7 mol/l and 2,8 mol/l.

NOTE 1 Concentrated lactic acid contains associated molecules which, on dilution, dissociate slowly to a certain equilibrium. Boiling accelerates this dissolution process, which is essential in order to obtain reproducible sedimentation values.

5.1.2 Preparation of test reagent

Thoroughly mix 180 ml of the diluted lactic acid solution (5.1) with 200 ml of between 99 % (V/V) and 100 % (V/V) propan-2-ol containing not more than 40 mg/kg of mineral matter, and make up to 1 000 ml with water.

Keep in a stoppered flask and do not use the reagent until it has been left to stand for 48 h.

5.2 Bromophenol blue solution

Dissolve 4 mg of bromophenol blue in 1 000 ml of water.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Test mill, of an appropriate type[1] (see annex A).

6.2 Woven metal wire sieve[2], in accordance with the specifications of ISO 565, having a nominal aperture size of 150 µm, 200 mm in diameter, moved by an appropriate automatic vibration device of eccentricity 50 mm and rotational frequency 200 min^{-1}.

6.3 Perforated metal plate sieve, with slots 1 mm wide.

6.4 Flat-bottom cylinders, of 100 ml capacity, graduated in millilitres, with a distance of 180 mm to 185 mm between the bottom and the 100 ml mark, and equipped with plastic or glass stoppers.

6.5 Cylinder shaker, fitted with a time-switch and providing a shaking frequency of 40 min^{-1}; each cycle shall be through 60° (30° above and below the horizontal).

6.6 One-mark pipettes, of 25 ml and 50 ml capacity, conforming to ISO 648, or **automatic dispensers** emptying in 10 s to 15 s.

6.7 Stop-clock

6.8 Balance, accurate to 0,01 g.

7 Sampling

It is important that the laboratory receive a sample which is truly representative and has not 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 950[3].

8 Procedure

8.1 Moisture content of the grain

If the moisture content of the grain is unknown, determine it in accordance with the practical method specified in ISO 712.

If the moisture content determined is not between 14,5 % (m/m) and 15 % (m/m), reduce it or increase it so that it lies between these limits, either by drying the grain at laboratory temperature or by placing it in an atmosphere with a high relative humidity.

8.2 Ash content of the test flour

Determine the ash content of the test flour by the incineration method at 900 °C specified in ISO 2171. The ash content shall not exceed 0,6 % of the dry matter content of the flour.

If this is not the case, it is impossible to obtain exact results for the sedimentation index.

8.3 Preparation of the test flour

Take a sample of 100 g, 150 g or 200 g of the grain, according to the type of mill (6.1) used for grinding (see annex A).

Separate all impurities from the grain, removing the coarsest particles by hand and smaller particles by means of the perforated metal plate sieve (6.3).

Grind the grain and sieve the ground product as described in annex A.

After sieving, thoroughly mix all the test flour obtained, the mass of which shall be at least 10 % of the mass of the sample taken for grinding.

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1) At present, the following five types of mill are suitable: Miag-Grobschrotmühle; Brabender-Sedimat; Strand-Roll, model SRM; Straube, model W.1; Tag-Heppenstall. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the apparatus named. Equivalent apparatus may be used if it can be shown to lead to the same results.

2) In the case of the Brabender-Sedimat mill, the sieving device is built into the appliance (see A.3).

3) ISO 950:1979, *Cereals — Sampling (as grain)*.

8.4 Test portion

Weigh, to the nearest 0,05 g, 3,2 g of the test flour (8.3).

NOTE 2 If there is any reason to think that the moisture content of the test flour is outside the range 13 % (*m/m*) to 15 % (*m/m*), determine its value in accordance with ISO 712 then weigh a quantity of the test flour corresponding to 3,20 g ± 0,05 g at 14 % (*m/m*) moisture content (i.e. 2,75 g ± 0,04 g of dry matter).

8.5 Determination

8.5.1 Carry out the sedimentation procedure specified in 8.5.2 to 8.5.6 twice on separate test portions taken from the same test flour (8.3).

8.5.2 The operations specified in 8.5.3 to 8.5.6 shall be carried out under normal lighting conditions, out of direct sunlight.

The time taken to pour each reagent into the cylinder (see 8.5.3 and 8.5.4) shall not exceed 15 s.

8.5.3 Place the test portion (8.4) in a graduated cylinder (6.4).

Add 50 ml of the bromophenol blue solution (5.2) to the test portion. Close the cylinder with a stopper and, holding the cylinder in a horizontal position, shake it left and right, through approximately 18 cm, 12 times in each direction over approximately 5 s.

8.5.4 Place the cylinder in the shaker (6.5), and start the stop-clock (6.7) and the shaker. After 5 min, remove the cylinder from the shaker and add to its contents 25 ml of the sedimentation test reagent (5.1).

Replace the cylinder and continue the shaking.

8.5.5 After a total time of 10 min, remove the cylinder from the shaker and place it in an upright position.

8.5.6 Leave the contents of the cylinder to stand for exactly 5 min, and then note the volume of the deposit to the nearest 0,5 ml.

9 Expression of results

The number indicating the volume, expressed in millilitres, of the deposit noted in accordance with 8.5.6 represents the sedimentation index.

Take as the result the arithmetic mean of the results of two determinations (8.5.1), provided that their difference does not exceed 2 units. If their difference does exceed 2 units, discard the results of the two determinations and repeat the operations given in 8.4 and 8.5.

Report the result as a whole number.

10 Precision

10.1 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, should not be greater than 2 units.

10.2 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, should not be greater than

— 2 (absolute value) for a sedimentation index of less than 20

— 10 % (relative value) of the mean value for a sedimentation index of greater than 20.

11 Test report

The test report shall specify

— the method in accordance with which sampling was carried out (if known),

— the type of mill used,

— the method used,

— the test result(s) obtained, and

— if the repeatability has been checked, the final quoted result obtained.

It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the result.

The test report shall include all information necessary for the complete identification of the sample.

Annex A
(normative)

Grinding and sieving of the sample

A.1 General

The sample specified in A.2 to A.6 as appropriate, freed from impurities, shall be ground under the conditions specified below, according to the type of mill used.

A.2 Miag-Grobschrotmühle mill

Sample: 100 g

— Carry out a first grinding with the rolls 1 mm apart and at a rotational frequency of about 30 min^{-1}.

— Regrind all the material obtained from the first grinding, but with the rolls 0,1 mm apart, then repeat this regrinding procedure a further three times.

— Using a sieve (6.2) having a nominal aperture size of 150 µm, sieve the product of these three successive grindings for 5 min.

A.3 Brabender-Sedimat mill

Sample: 100 g

— Set the timing device of the mill at 3 min.

— Carry out the grinding with a gap of 1 mm between the feed roll and the first grinding roll, and a gap of about 0,5 mm between the other grinding rolls, at a rotational frequency of about 1 000 min^{-1}.

NOTES

3 On the Brabender-Sedimat mill, the roll gaps and speed are not adjustable. These details are given to ensure that the rolls are not badly worn and that the motor runs at the correct speed.

4 The products of the milling pass directly into the sifter.

— If the mass of the ground product is less than 10 g, continue sieving until that quantity is obtained.

A.4 Tag-Heppenstall mill

Sample: 200 g

— Carry out a first grinding with the rolls 0,6 mm apart and at a rotational frequency of about 30 min^{-1}.

— Regrind all the material obtained from the first grinding, with the same gap between the rolls, then repeat this regrinding procedure a further three times.

— Using a sieve (6.2) having a nominal aperture size of 150 µm, sieve the product of the five successive grindings for 1,5 min.

A.5 Strand-Roll mill, model SRM

Sample: 150 g

— Carry out a first grinding with the rolls 0,8 mm apart and at a rotational frequency of about 30 min^{-1}.

— Regrind all the material obtained from the first grinding, with the same gap between the rolls, then repeat this regrinding procedure a further three times.

— Using a sieve (6.2) having a nominal aperture size of 150 µm, sieve the product of the five successive grindings for 1,5 min.

A.6 Straube mill, model W.1

Sample: 150 g

Proceed as in A.5, carrying out the five grindings with the rolls 1,10 mm apart and at a rotational frequency of 60 min^{-1}.

A.7 Cleaning of apparatus

Between successive grinding and sieving operations with different samples of wheat, the mills and sieves shall be suitably cleaned.

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