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Cereals and milled cereal products — Determination of the viscosity of flour — Method using an amylograph

Céréales et produits de mouture des céréales — Détermination de la viscosité de la farine — Méthode utilisant un amylographe

Reference number
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Foreword

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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 7973 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 4, *Cereals and pulses*.

This International Standard takes into account Standard No. 126 of the International Association for Cereal Science and Technology (ICC).

Annex A forms an integral part of this International Standard. Annexes B and C are for information only.

International Organization for Standardization
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Cereals and milled cereal products — Determination of the viscosity of flour — Method using an amylograph

1 Scope

This International Standard specifies a method using an amylograph for determining the viscosity of a suspension of flour in water, in which the starch is gelatinized by heating, in order to assess the conditions of gelatinization of the flour and so judge whether there is any alpha-amylase activity.

This method is applicable to wheat and rye flour and also to wheat and rye grain.

NOTES

1 This International Standard has been prepared on the basis of the Brabender-type amylograph.

2 This method applies strictly to an amylograph and not to a viscograph, since an amylograph possesses the following characteristics:

- it is possible to change the torque-measuring head;
- the heating coils are located around the bowl of the apparatus and at the bottom;
- there is no cooling rod for lowering the gel temperature.

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

ISO 3093:1982, *Cereals — Determination of falling number.*

3 Definition

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

3.1 amylograph viscosity: Maximum viscosity reached by a suspension of flour and water which is gelatinized by heating under the conditions set out in this International Standard.

It is expressed as an arbitrary unit: amylograph unit (AU).

4 Principle

Preparation of a suspension of flour in water, followed by recording of the viscosity of the suspension which is heated at a constant rate from 30 °C to the temperature corresponding to the moment at which viscosity starts to decrease, having reached its maximum (approximately 95 °C).

The increase in viscosity due to gelatinization of the starch is dependent upon the increase in temperature, the mechanical action of stirring and the activity of alpha-amylase already present or added to the flour.

5 Reagent

5.1 Distilled water, or water of equivalent purity.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Amylograph, having the following characteristics:

Speed of rotation of the spindle	(75 ± 1) rev/min
Torque exerted per amylograph unit (AU), using a standard measuring cartridge	$(6{,}86 \pm 0{,}14) \times 10^{-5}$ N.m/AU [(0,700 ± 0,015) gf.cm/AU)]
Rate of temperature rise	(1,50 ± 0,03) °C/min
Linear speed of the recorder	(0,50 ± 0,01) cm/min

Position the sensing pins and the pins of the bowl so that they penetrate freely into the jig provided by the manufacturer.

Adjust the pressure of the pen on the paper as follows. Remove the pen, fill it with ink and weigh it. At the end of the arm which normally holds the pen, place a mass 0,5 g to 1 g less than that of the completely filled pen. Adjust the position of the counterweight to obtain equilibrium then remove the mass and replace the pen.

6.2 Analytical balance, accurate to within 0,1 g.

6.3 Burette, of 450 ml capacity, with an automatic zeroing system.

6.4 Shallow beaker, of capacity 600 ml to 1000 ml, or a **conical flask** with **stopper**.

6.5 Spatula, with a rubber or plastic end.

6.6 Mill[1], complying with the requirements of ISO 3093 in the case of wheat and rye grains; i.e. capable of grinding a product of moisture content up to 30 % (*m*/*m*) and with adjustment to obtain a meal meeting the requirements of table 1.

Table 1 — Particle size requirements

Mesh opening of sieve µm	Meal passing through the sieve %
710	100
500	95 to 100
210 to 200	80 or less

7 Sampling

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

Sampling is not part of the method specified in this International Standard. Recommended sampling methods are given in ISO 950 (for grains) and ISO 2170 (for milled products).

8 Procedure

8.1 Preparation of test sample

8.1.1 Flour

Use the laboratory sample as provided, after thorough mixing, taking test samples as required.

8.1.2 Grain

Remove dust and coarse impurities from the laboratory sample and then take approximately 300 g of grain.

Grind the sample carefully in the mill (6.6), avoiding heating or overloading.

Continue grinding for 30 s to 40 s after the last of the sample has been fed into the mill. Bran particles up to 1 % remaining on the screen may be discarded.

Thoroughly mix the milled product.

8.2 Preliminary operations

8.2.1 Determination of the moisture content of the test sample

Determine the moisture content of the test sample (8.1) in accordance with ISO 712.

8.2.2 Adjustment of the amylograph and no-load test

Manually adjust the starting temperature of the temperature regulator to 30 °C, with the clutch in the neutral position. Fill the pen with ink. Place the spindle in the bowl, connect the spindle to the shaft and lower the amylograph head into position. Start the motor and check that the pen moves over the baseline of the recording paper. If necessary, adjust the position of the pen on its arm. Stop the motor, disconnect the spindle, lift and turn the head of the apparatus. Remove the spindle.

1) Kamas Slago 200 A and Falling Number type KT 120 mills are examples of suitable commercially available products. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the products named. Equivalent products may be used if they can be shown to lead to the same results.

CAUTION — Refer to annex A for the calibration of the amylograph. Particular care shall be taken when adjusting the contact thermometer as its position has a marked influence on the results.

8.3 Test portion

8.3.1 For flour

Weigh, to the nearest 0,1 g, the equivalent of 80,0 g of flour with a moisture content of 14,0 % (*m*/*m*). This mass, *m*, in grams, is given in table 2 as a function of moisture content.

8.3.2 For grain

Adjust the mass of the test portion so that 90,0 g of milled product corresponds to a moisture content of 14,0 % (*m*/*m*). This mass, *m*, in grams, is also given in table 2 as a function of moisture content.

8.4 Preparation of suspension

8.4.1 Fill the burette (6.3) with distilled water up to the zero mark.

8.4.2 Place the test portion in the beaker (6.4) and add 100 ml of water from the burette. Mix with the spatula (6.5) for approximately 20 s in order to obtain a uniform suspension.

NOTE 3 A longer mixing time may be necessary for rye flour.

While continuing to stir, gradually add the water (in four stages) until there are approximately 100 ml remaining in the burette and check that the suspension is free from lumps and there is as little foaming as possible, then pour the suspension into the amylograph bowl.

8.4.3 Using the spatula, scrape up any residue of suspension which might be sticking to the sides and bottom of the beaker and dilute with half the water remaining in the burette. Pour all of this suspension into the amylograph bowl.

Pour the remaining water into the beaker to rinse it and collect a quantity of rinsing solution in the amylograph bowl, such that the total mass of the suspension is 530,0 g ± 0,5 g.

8.4.4 It is important that operations 8.4.1, 8.4.2 and 8.4.3 be completed within 2 min.

8.4.5 In the case of grain, the mass of the test portion plus the mass of water should equal 540,0 g ± 0,5 g (corresponding to 90 g of flour and 450 g of water).

8.5 Amylograph test

8.5.1 Place the spindle in the amylograph bowl. Connect it to the shaft and carefully lower the head of the apparatus.

8.5.2 Start the motor and switch on the heater and timer. As soon as the heater cuts out (automatically), mark the next line up on the recording paper. At the moment that this line passes under the pen, place the clutch in the up position.

Set the timer to ring at the desired time. For wheat the time taken to obtain the viscosity curve is 40 min to 45 min or less, and for rye it is 30 min to 40 min or less.

8.5.3 When the curve has reached its maximum and has started to drop again, stop the motor, switch off the heater and read the temperature on the thermometer. Disconnect the spindle from its shaft. Raise the head of the apparatus, leaving the spindle in the bowl. Remove and clean the bowl and spindle under running water from the tap. Clean the thermoregulator with a warm damp cloth.

8.5.4 If a viscosity in excess of 1 000 AU is reached, add a supplementary weight provided for this purpose, so increasing the recording range of the curve by 500 AU or 1 000 AU.

If this is not possible, repeat the procedure from 8.3, using a smaller test portion (e.g. 70 g).

9 Calculation

9.1 Determination of the maximum amylograph viscosity

The maximum amylograph viscosity, expressed in amylograph units (AU), is given by the *y*-axis of the curve at its maximum (see figure 1).

Express this viscosity to the nearest 5 AU.

Table 2 — Mass of the test portion, in grams, corresponding to 80 g and 90 g, at 14 % (m/m) moisture content

Moisture content % (m/m)	Mass of the test portion corresponding to		Moisture content % (m/m)	Mass of the test portion corresponding to	
	80 g	90 g		80 g	90 g
9,0	75,6	85,1	13,6	79,6	89,6
9,1	75,7	85,1	13,7	79,7	89,7
9,2	75,8	85,2	13,8	79,8	89,8
9,3	75,9	85,3	13,9	79,9	89,9
9,4	75,9	85,4	14,0	80,0	90,0
9,5	76,0	85,5	14,1	80,1	90,1
9,6	76,1	85,6	14,2	80,2	90,2
9,7	76,2	85,7	14,3	80,3	90,3
9,8	76,3	85,8	14,4	80,4	90,4
9,9	76,4	85,9	14,5	80,5	90,5
10,0	76,4	86,0	14,6	80,6	90,6
10,1	76,5	86,1	14,7	80,7	90,7
10,2	76,6	86,2	14,8	80,8	90,8
10,3	76,7	86,3	14,9	80,8	91,0
10,4	76,8	86,4	15,0	80,9	91,1
10,5	76,9	86,5	15,1	81,0	91,2
10,6	77,0	86,6	15,2	81,1	91,3
10,7	77,0	86,7	15,3	81,2	91,4
10,8	77,1	86,8	15,4	81,3	91,5
10,9	77,2	86,9	15,5	81,4	91,6
11,0	77,3	87,0	15,6	81,5	91,7
11,1	77,4	87,1	15,7	81,6	91,8
11,2	77,5	87,2	15,8	81,7	91,9
11,3	77,6	87,3	15,9	81,8	92,0
11,4	77,7	87,4	16,0	81,9	92,1
11,5	77,7	87,5	16,1	82,0	92,3
11,6	77,8	87,6	16,2	82,1	92,4
11,7	77,9	87,7	16,3	82,2	92,5
11,8	78,0	87,8	16,4	82,3	92,6
11,9	78,1	87,9	16,5	82,4	92,7
12,0	78,2	88,0	16,6	82,5	92,8
12,1	78,3	88,1	16,7	82,6	92,9
12,2	78,4	88,2	16,8	82,7	93,0
12,3	78,4	88,3	16,9	82,8	93,1
12,4	78,5	88,4	17,0	82,9	93,3
12,5	78,6	88,5	17,1	83,0	93,4
12,6	78,7	88,6	17,2	83,1	93,5
12,7	78,8	88,7	17,3	83,3	93,6
12,8	78,9	88,8	17,4	83,3	93,7
12,9	79,0	88,9	17,5	83,4	93,8
13,0	79,1	89,0	17,6	83,5	93,9
13,1	79,2	89,1	17,7	83,6	94,0
13,2	79,3	89,2	17,8	83,7	94,2
13,3	79,4	89,3	17,9	83,8	94,3
13,4	79,4	89,4	18,0	83,9	94,4
13,5	79,5	89,5			

NOTE — The values in this table have been calculated using the formula

$$m = m' \times 86/(100 - H)$$

where

m is the mass of the test portion, in grams;

H is the moisture content of the sample, expressed as a percentage by mass;

m' is the mass, in grams, of a test portion of moisture content 14 % ($m' = 80$ g or $m' = 90$ g).

9.2 Determination of temperature at maximum viscosity

The temperature θ, at maximum viscosity, expressed in degrees Celsius, is given by the formula

$$\theta = 30{,}0 + (t_f - 30{,}0) \times a/b$$

where

t_f is the temperature, in degrees Celsius, read from the thermometer at the end of the test (approximately 95 °C);

a is the length, in centimetres, of the recording from the mark made at the start of the test to maximum viscosity;

b is the length, in centimetres, of the recording from the mark made at the start of the test to the end of the test.

Express the temperature θ to the nearest 0,5 °C.

If the apparatus is correctly adjusted

$$(t_f - 30{,}0)/b = (3{,}0 \pm 0{,}1)\ °C/cm$$

and thus $\theta = 30{,}0 + 3a$.

NOTE 4 It may also be of interest to note the temperature at which gelatinization starts (marked change in the slope of the recorded curve), and then use a formula similar to the above.