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Cereals and cereal products — Common wheat (*Triticum aestivum* L.) — Determination of alveograph properties of dough at constant hydration from commercial or test flours and test milling methodology

*Céréales et produits céréaliers — Blé tendre (*Triticum aestivum *L.) — Détermination des propriétés alvéographiques d'une pâte à hydratation constante de farine industrielle ou d'essai et méthodologie pour la mouture d'essai*

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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.

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 27971 was prepared by the European Committee for Standardization (CEN) Technical Committee CEN/TC 338, *Cereal and cereal products*, in collaboration with Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 4, *Cereals and pulses*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This first edition of ISO 27971 cancels and replaces ISO 5530-4:2002, which has been technically revised to specify the preparation of a test flour, to present complete precision data, and to add one annex giving alveograph maintenance advice and another for the assessment of proteolytic activity in wheat or flour.

Introduction

The end-use value of wheat is determined by a number of properties that are useful in the manufacture of baked products such as bread, rusks, and biscuits.

Such properties include the important viscoelastic (rheological) properties of dough formed as a result of flour hydration and kneading. An alveograph is used to study the main parameters by subjecting a dough test piece to biaxial extension (producing a dough bubble) by inflating it with air which is similar to the deformation to which it is subjected during panary fermentation.

Recording the pressure generated inside the bubble throughout the deformation of the dough test piece until rupture provides information on:

— the resistance of the dough to deformation, or its strength (stiffness). It is expressed by the maximum pressure parameter, P;

— the extensibility or the possibility of inflating the dough to form a bubble. It is expressed by the parameters of extensibility, L, or swelling, G;

— the elasticity of the dough during biaxial extension. It is expressed by the elasticity index, I_e;

— the energy required to deform the dough bubble until it bursts, which is proportional to the area of the alveogram (sum of the pressures throughout the deformation process). It is expressed by the parameter, W.

The P/L ratio is a measurement of the balance between tenacity and extensibility.

Alveographs are commonly used throughout the wheat and flour industry, for the following purposes:

— selecting and assessing different varieties of wheat and marketing batches of wheat;

— blending different batches of wheat or flour to produce a batch with given values for the alveographic criteria (W, P, and L) complying with the proportional laws of blending.

Alveographs are used both on the upstream side of the industry for marketing, selecting and assessing the different varieties and on the downstream side throughout the baking industries (see Bibliography).

Cereals and cereal products — Common wheat (*Triticum aestivum* L.) — Determination of alveograph properties of dough at constant hydration from commercial or test flours and test milling methodology

1 Scope

This International Standard specifies a method of using an alveograph to determine the rheological properties of different types of dough obtained from "soft" to "hard" wheat flour (*Triticum aestivum* L.) produced by industrial milling or laboratory test milling.

It describes the alveograph test and how to use a laboratory mill to produce flour in two stages:

Stage 1: preparation of the wheat grain for milling to make it easier to separate the bran from the endosperm (see Clause 7);

Stage 2: the milling process itself, including the break system involving three fluted rollers, reduction of particle size between two smooth rollers and the use of a centrifugal sieving machine to grade the products (see Clause 8).

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 385, *Laboratory glassware — Burettes*

ISO 660, *Animal and vegetable fats and oils — Determination of acid value and acidity*

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

ISO 835, *Laboratory glassware — Graduated pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 7700-1, *Check of the calibration of moisture meters — Part 1: Moisture meters for cereals*

3 Principle

The behaviour of dough obtained from a mixture of different types of flour and salt water is evaluated during deformation. A dough disk is subjected to a constant air flow; which at first it withstands. Subsequently, it swells into a bubble, according to its extensibility, and ruptures. The change in the dough is measured and recorded in the form of a curve called an alveogram.

4 Reagents

Unless otherwise specified, use only reagents of recognized analytical grade, and only distilled or demineralized water or water of equivalent purity.

4.1 Sodium chloride solution, obtained by dissolving (25 ± 0,2) g of NaCl in water and then making the volume up to 1 000 ml. This solution shall not be stored for more than 15 days and its temperature shall be (20 ± 2) °C when used.

4.2 Refined vegetable oil, low in polyunsaturates, such as peanut oil. It is possible to use olive oil if its acid index value is less than 0,4 (determined according to ISO 660). Store in a dark place in a closed container and replace regularly (at least every 3 months).

Alternatively, **liquid paraffin** (also known as "soft petroleum paraffin"), with an acid index value less than or equal to 0,05 and the lowest possible viscosity [maximum 60 mPa s (60 cP) at 20 °C].

4.3 Cold degreasing agent, optimum safety[1)].

5 Apparatus

Usual laboratory apparatus, and in particular the following.

5.1 Mechanical cleaner, fitted with sieves for wheat cleaning, in accordance with the manufacturer's requirements.

5.2 Conical or riffle sample divider.

5.3 Analytical balance, accurate to 0,01 g.

5.4 Glass burette, of capacity 50 ml, complying with the requirements of ISO 385, Class A, graduated in 0,1 ml divisions, stand-mounted.

5.5 Rotary blender[2)], for grain conditioning and flour homogenization, including the following components:

5.5.1 Constant speed stirrer.

5.5.2 Two worm screws integral with the flask, possibly via the stopper (one for wheat preparation, the other for flour homogenization).

5.5.3 Several wide-necked **plastic flasks**, of capacity 2 l.

5.6 Test mill[3)] (laboratory mill) manually operated (see Annex A).

1) ITECMA "Securclean ER" is an example of a suitable product 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.

2) The Chopin MR 2 l rotary blender is an example of a suitable product 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.

3) The Chopin-Dubois CD1 test mill is an example of a suitable product 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.

5.7 Complete alveograph system (see Table 1 for specifications and characteristics of the accessories) including the following devices:

5.7.1 Kneading machine [for models MA 82, MA 87 and MA 95, see Figure 1a); for model NG, see labels a in Figure 2 and Figure 3], with accurate temperature control, for dough sample preparation.

5.7.2 Hydraulic manometer or **Alveolink**4) [for models MA 82, MA 87 and MA 95, see Figure 1 b); for model NG, see labels b in Figure 2 and Figure 3] for recording the pressure curve.

5.7.3 Alveograph5) [for models MA 82, MA 87 and MA 95, see Figure 1 c); for model NG, see labels c in Figures 2 and Figure 3] with accurate temperature control, for test piece biaxial deformation of the dough pieces. It has two rest chambers, each containing five plates on which the dough test pieces can be arranged prior to deformation.

5.8 Burette, supplied with the apparatus, of capacity 160 ml, graduated in divisions of 0,1 % of moisture content6) .

5.9 Timer, for use with model MA 82 only.

5.10 Planimetric scales, supplied with the apparatus where an Alveolink is not included.

5.11 System for recording the test environment conditions (temperature and relative air humidity) as specified in 8.1 and 9.1.

5.12 Volumetric flask, of capacity 1 000 ml, complying with the requirements of ISO 1042, class A.

5.13 Pipette, of capacity 25 ml, graduated in divisions of 0,1 ml, complying with the requirements of ISO 835, class A.

6 Sampling

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

Sampling is not part of the method specified in this International Standard. Recommended sampling methods are given in ISO 2170[1], ISO 6644[6], and ISO 13690[7].

7 Preparation of the wheat for test milling

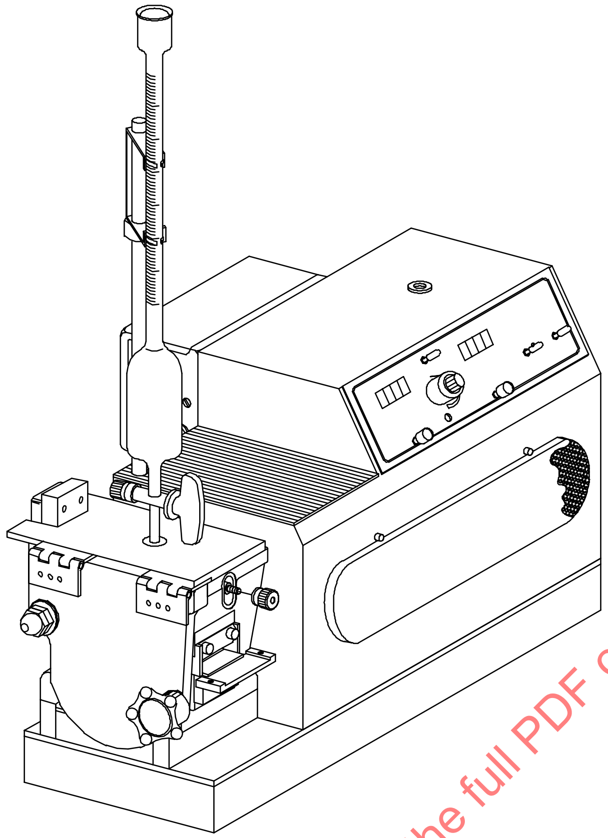
7.1 Cleaning the laboratory sample

Pass the laboratory sample through a mechanical cleaner (5.1) to ensure that all stones and metal fragments are removed and to avoid damaging the rollers during milling. A magnetic device can also be used to remove ferrous metal fragments.

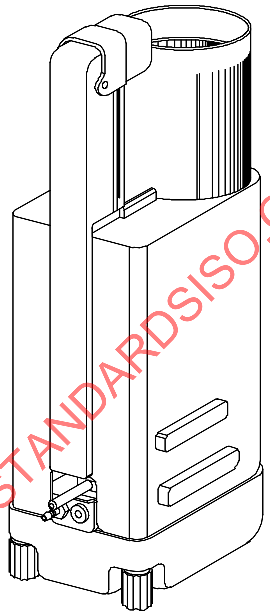
4) Example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

5) The methods specified in this International Standard are based on the use of the MA 82, MA 87, MA 95 and NG models of Chopin alveograph.

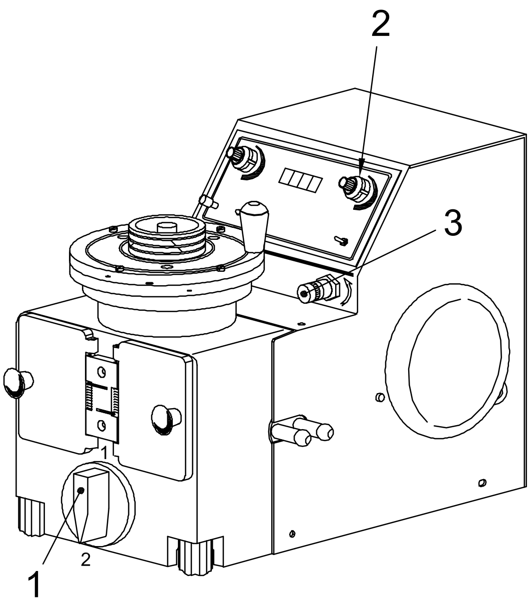
6) Throughout this International Standard, "content" is to be understood as a "mass fraction" (see ISO 80000-9:—[8], 12), i.e. the ratio of the mass of substance in a mixture to the mass of the mixture.



a) kneading machine



b) manometer



Key

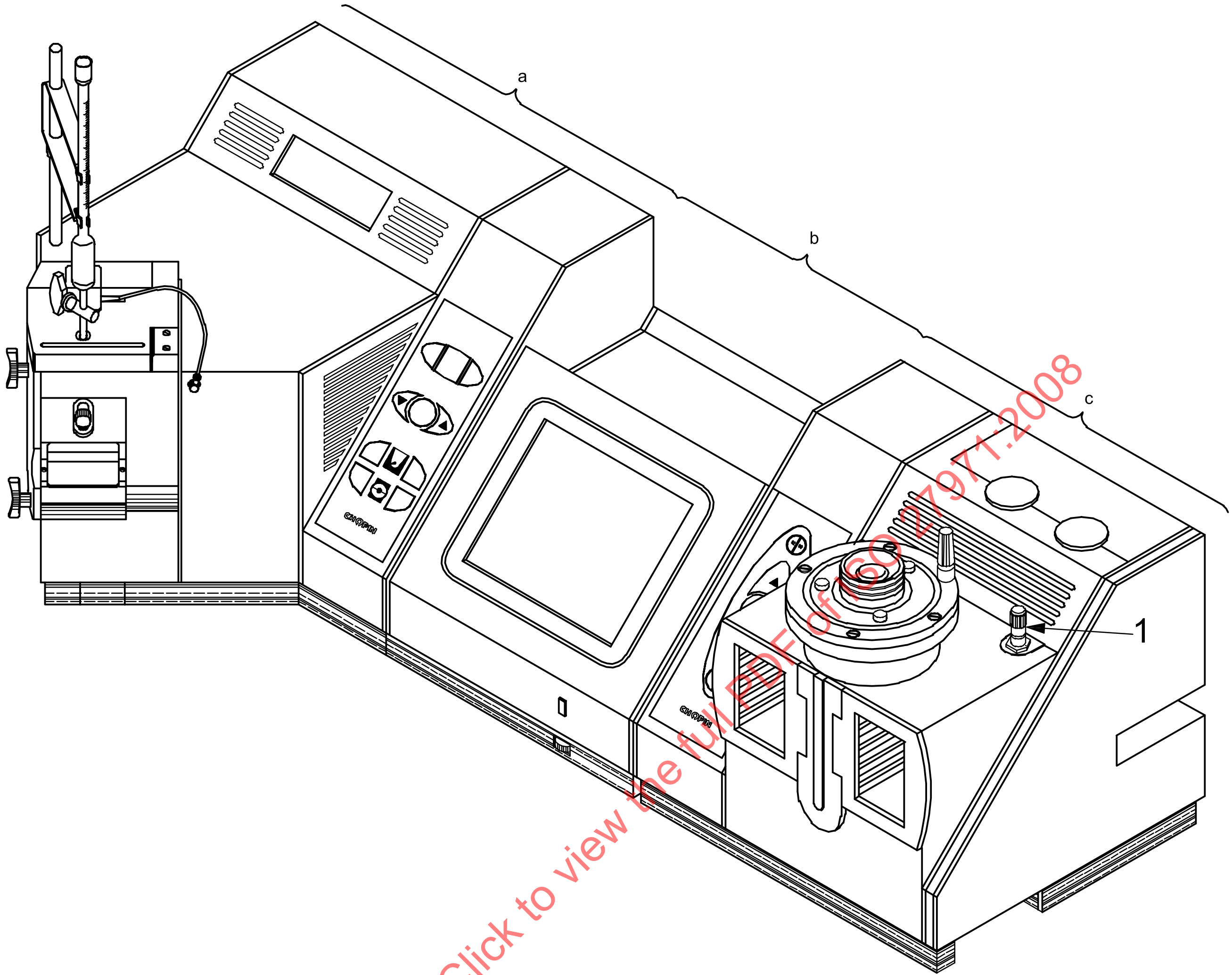
1 handle A in position 2

2 pump potentiometer

3 micrometric valve for air flow adjustment

c) alveograph

Figure 1 — Alveograph model MA 82, MA 87 and MA 95 assemblies



Key

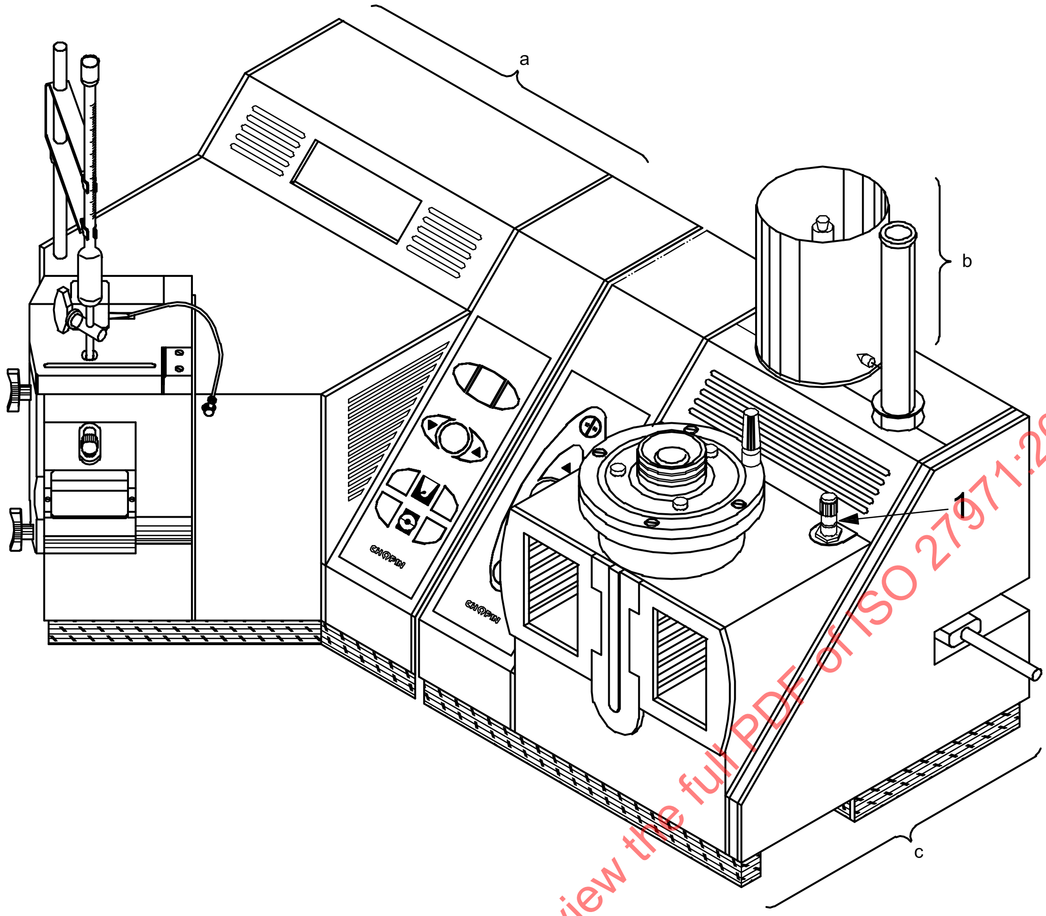
1 micrometric valve for air flow adjustment

a kneading machine

b integrator-recorder

c alveograph (with Alveolink integrator-recorder)

Figure 2 — Alveograph model NG assembly with an Alveolink integrator-recorder



Key

1 micrometric valve for air flow adjustment

a kneading machine

b recording manometer

c alveograph (with hydraulic recording manometer)

Figure 3 — Alveograph model NG assembly with a hydraulic recording manometer

Table 1 — Specifications and characteristics of the accessories required for the test

Quantity	Value and tolerance
Rotational frequency of the kneading machine blade	(60 ± 2) Hz
Height of sheeting guides	(12,0 ± 0,1) mm
Large diameter of the sheeting roller	(40,0 ± 0,1) mm
Small diameter of the sheeting roller	(33,3 ± 0,1) mm
Inside diameter of the dough cutter	(46,0 ± 0,5) mm
Diameter of the aperture created when the moving plate opens (which determines the effective diameter of the test piece)	(55,0 ± 0,1) mm
Theoretical distance between the fixed and moving plates after clamping (equal to the thickness of the test piece before inflation)	(2,67 ± 0,01) mm
Volume of air automatically injected to detach the test piece prior to inflating the bubble[a]	(18 ± 2) ml
Linear speed of the periphery of the recording drum	(5,5 ± 0,1) mm/s
Air flow[b] ensuring inflation	(96 ± 2) l/h
Rotation of the manometer drum (from stop to stop)	(55 ± 1) s

[a] Some older devices are fitted with a pear-shaped rubber bulb for manual injection of the 18 ml required to detach the test piece.

[b] To adjust the flow rate of the air generator used to inflate the bubble, fit the nozzle (Figure 4) to create a specified pressure drop (and obtain a pressure corresponding to a height of 92 mmHg (12,3 kPa) on the manometer chart). The air flow rate is set with the standardized pressure drop to obtain a pressure corresponding to a height of 60 mmHg (8,0 kPa) on the manometer chart, i.e. (96 ± 2) l/h (see Figure 4 and Figure 5).

7.2 Test portion

The test portion shall be representative of the initial wheat mass. Use the sample divider (5.2) to homogenize and divide the laboratory sample until the mass required for test milling plus moisture content determination is obtained. The minimum wheat mass of the test portion for milling shall be 800 g.

7.3 Wheat moisture content determination

Determine the moisture content of the test portion as specified in ISO 712, or using a rapid device whose measurement does not differ from the reference value by ± 0,4 g water per 100 g of sample (see ISO 7700-1).

7.4 Wheat conditioning

7.4.1 General

Conditioning the wheat for milling makes it easier to separate the bran from the endosperm. The target moisture content is (16 ± 0,5) %.

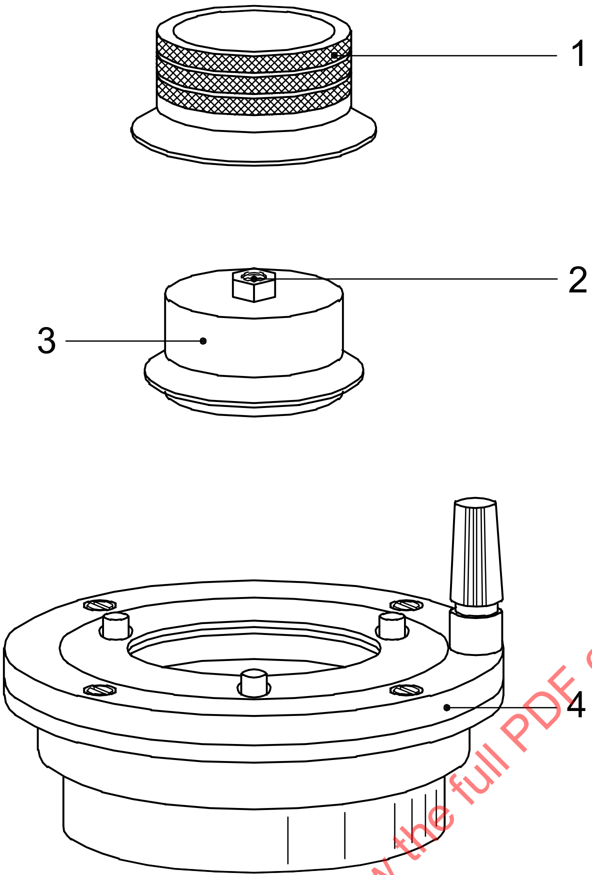
7.4.2 Wheat with initial moisture content between 13 % and 15 % (one-stage moistening)

Weigh, using the balance (5.3), a test portion of (800 ± 1) g of wheat and pour it into the blender.

Add the required amount of water (see Table B.1) to the grain from the burette (5.4) directly, or after weighing it to the nearest 0,1 g.

Immediately after adding the water, insert the stopper fitted with the worm screw provided for use with wheat into the flask, shake vigorously for a few seconds and place on the rotary blender (5.5).

Run the rotary blender for (30 ± 5) min (time required to distribute the water evenly across the surface of the grains).



Key

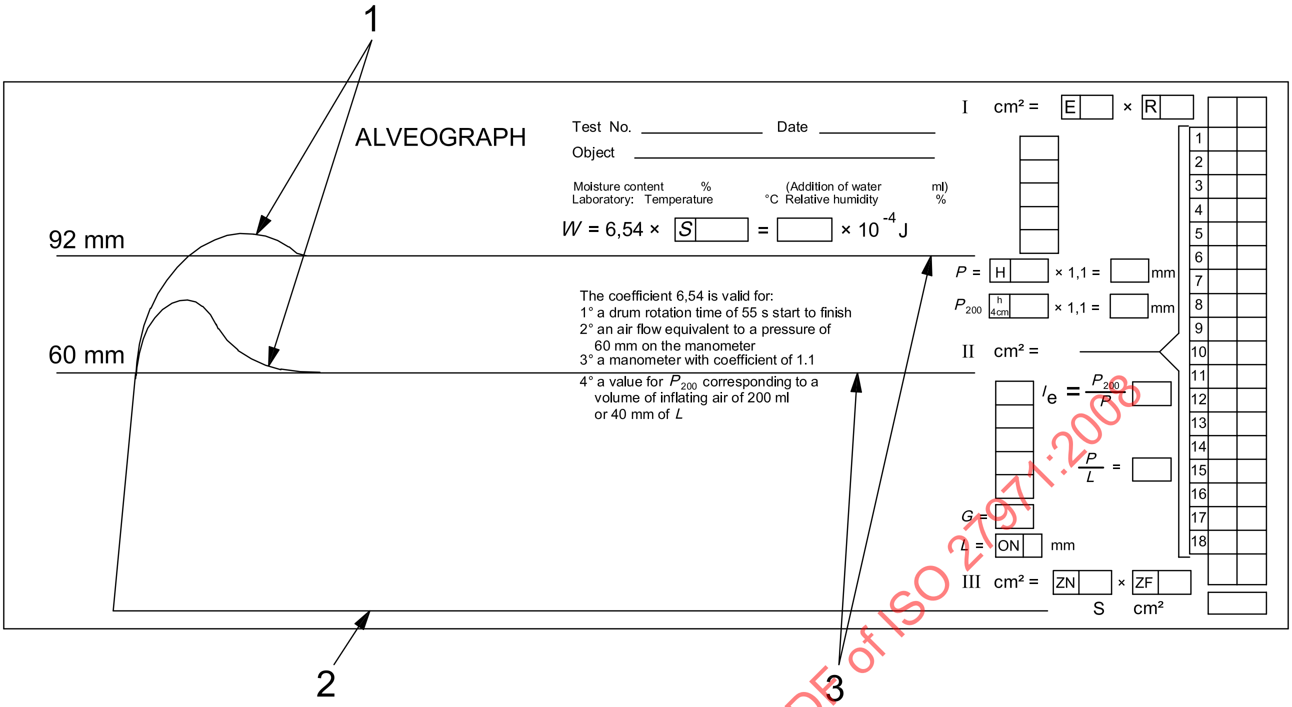
1 knurled ring

2 nozzle

3 nozzle holder

4 top plate

Figure 4 — Flow control system



Key

1 floater curve

2 zero pressure baseline

3 parallel lines

Figure 5 — Measurement pressure adjustment

Allow the flask to rest for a period that brings the total time of operations of moistening, shaking and resting to (24 ± 1) h.

7.4.3 Wheat with a moisture content less than 13 % (two-stage moistening)

As a larger volume of water is required, divide it into two halves and add in two stages during the conditioning period.

Proceed as described in 7.4.2, using only half the total quantity of water required (see Table B.1).

Shake the flask as described in 7.4.2 and allow it to rest for at least 6 h.

Then add the second half of the total quantity of water between the 6th hour and the 7th hour.

After adding the second half, shake the flask again for (30 ± 5) min, then allow it to rest for a period that brings the total time of operations of moistening, shaking and resting to (24 ± 1) h.

7.4.4 Wheat with a moisture content greater than 15 % (preliminary drying followed by conditioning, as described above)

The wheat shall be dried to produce a moisture content lower than 15 %.

Spread the laboratory sample in a thin layer to optimize the exchange between the grain and the air. Allow to dry in the open air in a dry place for at least 15 h.

Proceed to a new moisture determination (7.3).

Then condition the wheat as specified in 7.4.2 or 7.4.3, depending on the new moisture content.

8 Laboratory milling

8.1 General

The test mill (5.6) shall be used with the manufacturer's settings. Additional weights shall not be used and the tension on the converter side of the spring shall not be changed.

The quality of the milling process depends on several factors:

a) environmental conditions which allow the final moisture content of the flour to be between 15,0 % and 15,8 % (wheat should be milled in an ambient temperature between 18 °C and 23 °C with a relative air humidity between 50 % and 75 %);

b) condition of the sieves: the sieving area shall remain uniform — if a sieve is pierced, it shall immediately be replaced;

c) beater condition and setting: worn blades reduce the extraction rate;

d) compliance with flow rates: the efficiency of the roll and the efficiency of the sieving process are strictly dependent on a regular feed rate. The speed at which the products pass through the sieving drum can be set by adjusting the position of the blades[7)] on the beaters.

8.2 Milling procedure

8.2.1 Break system

Switch on the device.

Set the feed rate to allow the conditioned wheat to pass through the mill in (5 ± 1) min.

Pour the conditioned wheat (7.4) into the mill feed hopper and, at the same time, start the timer to check milling time.

After the last grains of wheat have passed through, let the mill continue to operate for (180 ± 30) s to completely clear out the sieve.

When the mill stops, weigh (5.3), to the nearest 0,1 g, the bran, the semolina, and the flour fractions.

Calculate the percentage of semolina obtained compared with the mass of wheat used, expressing the result to one decimal place.

8.2.2 Reduction system

Switch on the device.

Adjust the feed rate to allow the semolina produced in 8.2.1 to pass through the mill in (5 ± 1) min.

Pour the semolina into the feed hopper and, at the same time, start the timer to check the milling time.

After the last grains of semolina have passed through, let the mill continue to operate for (180 ± 30) s to completely clear out the sieve.

7) Two adjustable blades in the middle and at the end of the beater on the break side, four blades at the end of the reduction side.

Repeat the above reduction procedure if the mass of semolina obtained from the break system is greater than or equal to 48,0 %[8] of the mass of conditioned wheat.

When the mill stops, weigh to the nearest 0,1 g (5.3) the middlings fraction collected and the converted flour fractions.

Ensure that the milling ratio, BM (sum of the masses of the milled products divided by the total conditioned wheat mass), is equal to at least 98 %.

NOTE A result of less than 98 % indicates excessively worn beaters or an obstruction in the sieves, causing some of the product to remain inside the sieving drum.

8.2.3 Flour homogenization

Pour the flour obtained from the break and reduction processes into the blender flask (5.5.3).

Insert the stopper fitted with the worm screw (5.5.2) provided for use with flour into the flask and place the flask on the blender (5.5).

Mix for (20 ± 2) min.

Remove the worm screw (5.5.2) and replace it with the flask stopper. The flour is now ready for the alveograph test.

8.2.4 Storage of the flour

The flask containing the flour shall be kept in the room where the alveograph test is performed.

8.3 Expression of milling results

Calculate the extraction rate, ER, of flour extracted from the cleaned wheat on the dry matter basis, as a percentage, using Equation (1):

$$ER = \frac{(100 - H_f) \times M_f}{(100 - H_b) \times M_b} \times 100 \qquad (1)$$

where

H_f is the moisture content, as a percentage, of the flour obtained (determined according to ISO 712);

H_b is the moisture content, as a percentage, of the wheat test portion for milling before conditioning (determined according to ISO 712);

M_f is the mass, in grams, of the total flour obtained;

M_b is the wheat mass, in grams, of the test portion for milling before moistening.

Express the result to the nearest 0,1 % by mass.

Calculate the percentage of bran, S, using Equation (2):

$$S = [M_s/(M_b + M_e)] \times 100 \qquad (2)$$

8) Round up the values: 47,5 becomes 48 and 48,5 becomes 49.

Calculate the percentage of middlings, R, using Equation (3):

$$R = [M_r/(M_b + M_e)] \times 100 \qquad (3)$$

where

M_s is the mass, in grams, of bran;

M_r is the mass, in grams, of middlings;

M_b is the mass, in grams, of the wheat test portion for milling before moistening (7.2);

M_e is the mass, in grams, of water added (numerically equal to the volume, V_e, in millilitres, of water added).

Express the results to the nearest integer.

9 Preparation and alveograph test

9.1 Preliminary checks

Ensure that the ambient temperature is between 18 °C and 22 °C with a relative humidity between 50 % and 80 %.

Ensure that the various components of the apparatus (kneading machine, alveograph, recorder, burette, tools, etc.) are clean.

Check that the F-register is in place in the extrusion aperture to prevent any loss of flour or salt solution leakage.

Ensure that the temperature of the kneading machine (5.7.1) at the start of the test is (24 ± 0,5) °C; the temperature of the alveograph shall be continuously set to (25 ± 0,5) °C.

NOTE A rise in the kneading machine temperature during the kneading process is normal and specific to the flour under test. The continuous control feature provided on the NG models should not be used.

Regularly check that the pneumatic circuit on the apparatus is sealed (no air leakage) by following the manufacturer's recommended procedure.

Check the air flow settings using the nozzle (see Table 1, Note b), creating the specified loss of pressure [see Figure 1c), Labels 1 in Figures 2 and 3, and Figures 4 and 5] by setting:

a) the air generator to a pressure corresponding to 92 mmHg (12,3 kPa) on the hydraulic manometer chart or on the recorder screen;

b) the micrometer flow rate valve to a pressure corresponding to 60 mmHg (8,0 kPa) on the manometer chart or the recorder screen.

Check that the alveograph plate is horizontal.

If a recording manometer is employed, use the timer (5.9) to check the rotation time of the recording drum according to the manufacturer's recommended procedure.

9.2 Preliminary operations

At the beginning of the test, the temperature of the flour shall be the ambient temperature.

Determine the moisture content of the flour according to the method specified in ISO 712. From Table 2, find the quantity of sodium chloride solution (4.1) to be used in 9.3 to prepare the dough.

Table 2 — Volume of sodium chloride solution (4.1) to be added during kneading

Moisture content of the flour %	Volume of solution to be added ml	Moisture content of the flour %	Volume of solution to be added ml	Moisture content of the flour %	Volume of solution to be added ml
8,0	155,9	11,0	142,6	14,0	129,4
8,1	155,4	11,1	142,2	14,1	129,0
8,2	155,0	11,2	141,8	14,2	128,5
8,3	154,6	11,3	141,3	14,3	128,1
8,4	154,1	11,4	140,9	14,4	127,6
8,5	153,7	11,5	140,4	14,5	127,2
8,6	153,2	11,6	140,0	14,6	126,8
8,7	152,8	11,7	139,6	14,7	126,3
8,8	152,4	11,8	139,1	14,8	125,9
8,9	151,9	11,9	138,7	14,9	125,4
9,0	151,5	12,0	138,2	15,0	125,0
9,1	151,0	12,1	137,8	15,1	124,6
9,2	150,6	12,2	137,4	15,2	124,1
9,3	150,1	12,3	136,9	15,3	123,7
9,4	149,7	12,4	136,5	15,4	123,2
9,5	149,3	12,5	136,0	15,5	122,8
9,6	148,8	12,6	135,6	15,6	122,4
9,7	148,4	12,7	135,1	15,7	121,9
9,8	147,9	12,8	134,7	15,8	121,5
9,9	147,5	12,9	134,3	15,9	121,0
10,0	147,1	13,0	133,8	16,0	120,6
10,1	146,6	13,1	133,4		
10,2	146,2	13,2	132,9		
10,3	145,7	13,3	132,5		
10,4	145,3	13,4	132,1		
10,5	144,9	13,5	131,6		
10,6	144,4	13,6	131,2		
10,7	144,0	13,7	130,7		
10,8	143,5	13,8	130,3		
10,9	143,1	13,9	129,9		

NOTE Volume of sodium chloride solution (4.1), V_{NaCl}, to be added during kneading is calculated from the equation:

$V_{NaCl} = 191{,}175 - 4{,}411\ 75\ H_f$

where H_f is the moisture content of the flour.

These values have been calculated to obtain constant hydration, i.e. equivalent to a dough made from 50 ml of sodium chloride solution (4.1) and 100 g of flour with a moisture content of 15 %.

9.3 Kneading

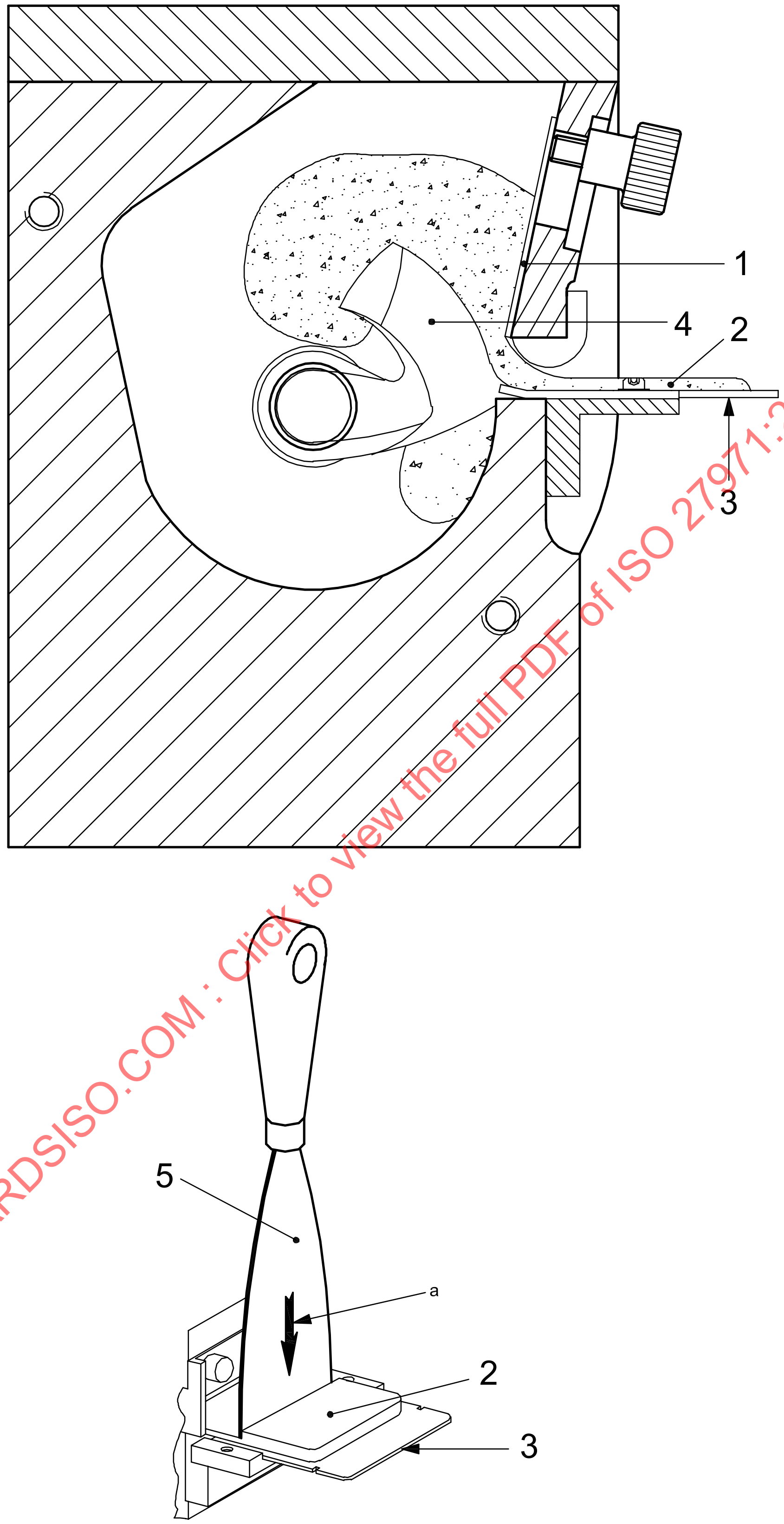
Place 250 g of flour, weighed (5.3) to within 0,5 g, in the kneading machine (5.7.1). Secure the lid with the locking device.

At the same time, switch on the motor, start the timer on the MA 82 models and use a burette (5.8) to deliver the appropriate quantity of sodium chloride solution (4.1) through the hole in the cover.

If the moisture content of the flour is less than 10,5 %, use the burette (5.8) to add a quantity of sodium chloride solution equivalent to a moisture content of 12 %, i.e. 138,2 ml. With a pipette (5.13), add a quantity of sodium chloride solution equal to the difference between the value given in Table 2 and the 138,2 ml already in the machine.

Allow the dough to form for 1 min, then switch off the motor, open the lid and, with the help of the plastic spatula provided, reincorporate any flour and dough adhering to the F-register (see Figure 6) and to the corners of the kneading machine. This operation should take less than 1 min.

NOTE This operation can be performed in two parts, allowing the kneading machine to rotate about 10 times between the first and second operations.



Key

1 F-register
2 dough
3 receiving plate
4 blender blade
5 knife/spatula
[a] Extruded dough strip cutting direction.

Figure 6 — Kneading machine

Close the cover, then restart the motor and knead for 6 min. During this time, oil the accessories required for extrusion.

Stop kneading after a total of 8 min (corresponding to the sum of dough formation and reincorporation times), then extrude the dough pieces (the NG has an automatic stop).

9.4 Preparation of dough test pieces

Reverse the direction of rotation of the kneader blade. Open the extrusion aperture by raising the F-register and place a few drops of oil (4.2) on the previously installed receiving plate. Remove the first centimetre of dough using the knife/spatula in a clean, downward movement, close to the guide (see Figure 6).

When the strip of dough is level with the notches on the extrusion plate, quickly cut the dough with the knife/spatula. Slide the piece of dough onto the previously oiled stainless steel plate on the sheeting table (first dough piece) (see Figure 6).

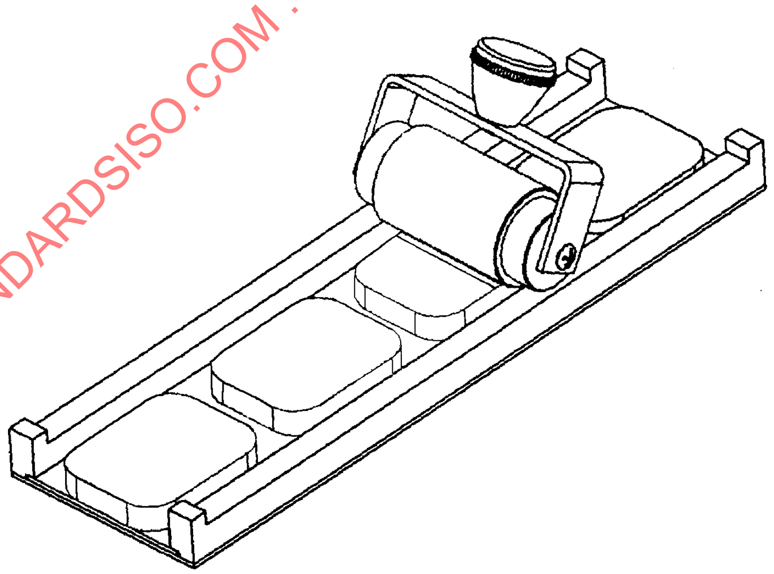
Successively extrude five dough pieces without stopping the motor, replacing the previously oiled receiving plate each time. Arrange the first four dough pieces on the sheeting table so that their direction of extrusion corresponds to its major axis [Figure 7a)]. Leave the fifth dough piece on the extrusion plate. Stop the motor.

NOTE Experienced operators are able to sheet, cut, and transfer each dough piece to the rest chamber in the same amount of time it takes to extrude the next dough piece.

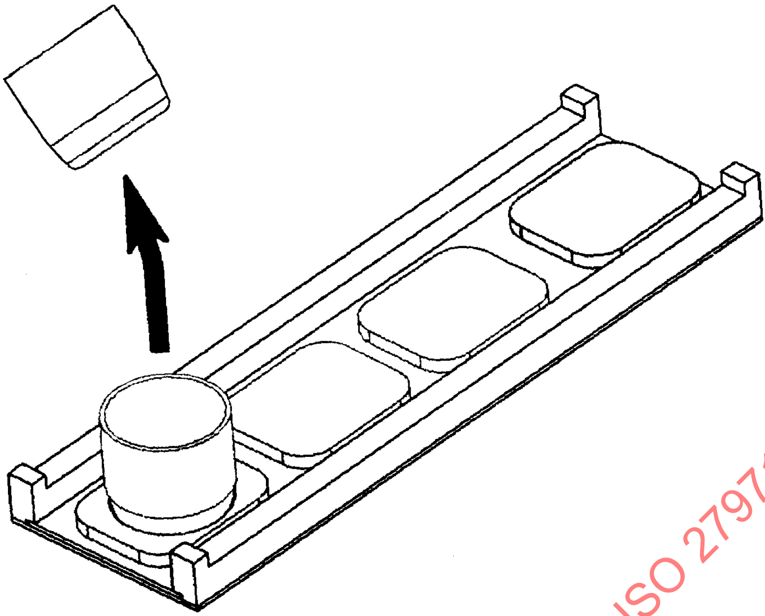
Sheet the four dough pieces using the previously oiled steel roller; run the roller backwards and forwards along the rails 12 times in succession, six times in each direction [see Figure 7a)].

Using the cutter, cut a test piece from each strip of dough in one clean movement [see Figure 7b)]. Remove any surplus dough.

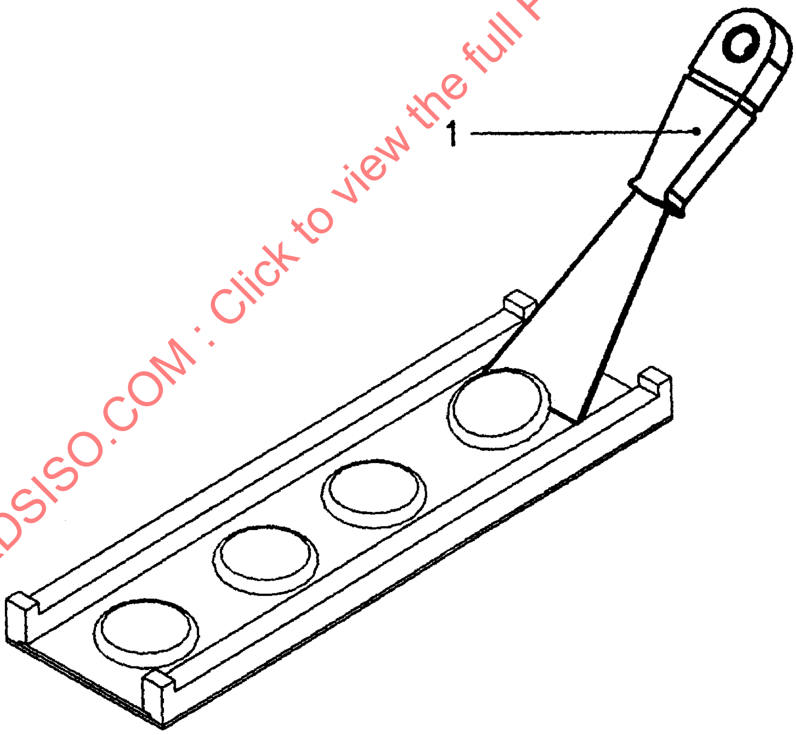
Hold the cutter containing the dough test piece above the previously oiled resting plate to which it is to be transferred and incline the cutter. If the dough sticks to the sides of the cutter, free it by tapping the work surface with the side of the palm of the hand (do not touch with fingers). If the test piece remains on the stainless steel plate on the sheeting table, raise it gently with the spatula [see Figure 7c)] and slide the resting plate under it.



a) sheeting the dough test pieces



b) cutting the dough test pieces



Key

1 spatula

c) transferring the dough test pieces

Figure 7 — Preparation of the dough test pieces

Immediately place each resting plate containing a dough piece into the thermostatically controlled compartment heated to 25 °C. Proceed by order of extrusion, carefully noting the location of the first test piece.

Repeat the operations described above with the fifth dough piece.

9.5 Alveograph test

9.5.1 Initial preparations

If a hydraulic manometer is used, distance the recording pen from the drum and place a recording chart on the drum. Fill the pen with ink. Turn the cylinder until it comes into contact with its stop. Bring the pen into contact with the cylinder and turn the cylinder so that it draws the zero line. Move the pen aside again to move the drum against the stop in its original position.

Perform the test 28 min after kneading begins. Check that the piston is in the raised position, proceed in the order of extraction of the test pieces.

9.5.2 Stage 1: Adjusting the dough test piece

Raise the handle to the vertical position on the NG model [Figure 8a)].

Raise the top plate by unscrewing it two turns to bring it level with the three guide studs [Figure 8a)].

Remove the ring and the plug [Figure 8a)].

Oil the bottom plate and the inner surface of the plug [Figure 8b)].

Slide the dough test piece onto the centre of the bottom plate.

Replace the plug and the ring [Figure 8c)].

Calibrate the test piece by slowly tightening the top plate by two turns in approximately 20 s [Figure 8d)].

Remove the ring and plug to free the dough test piece.

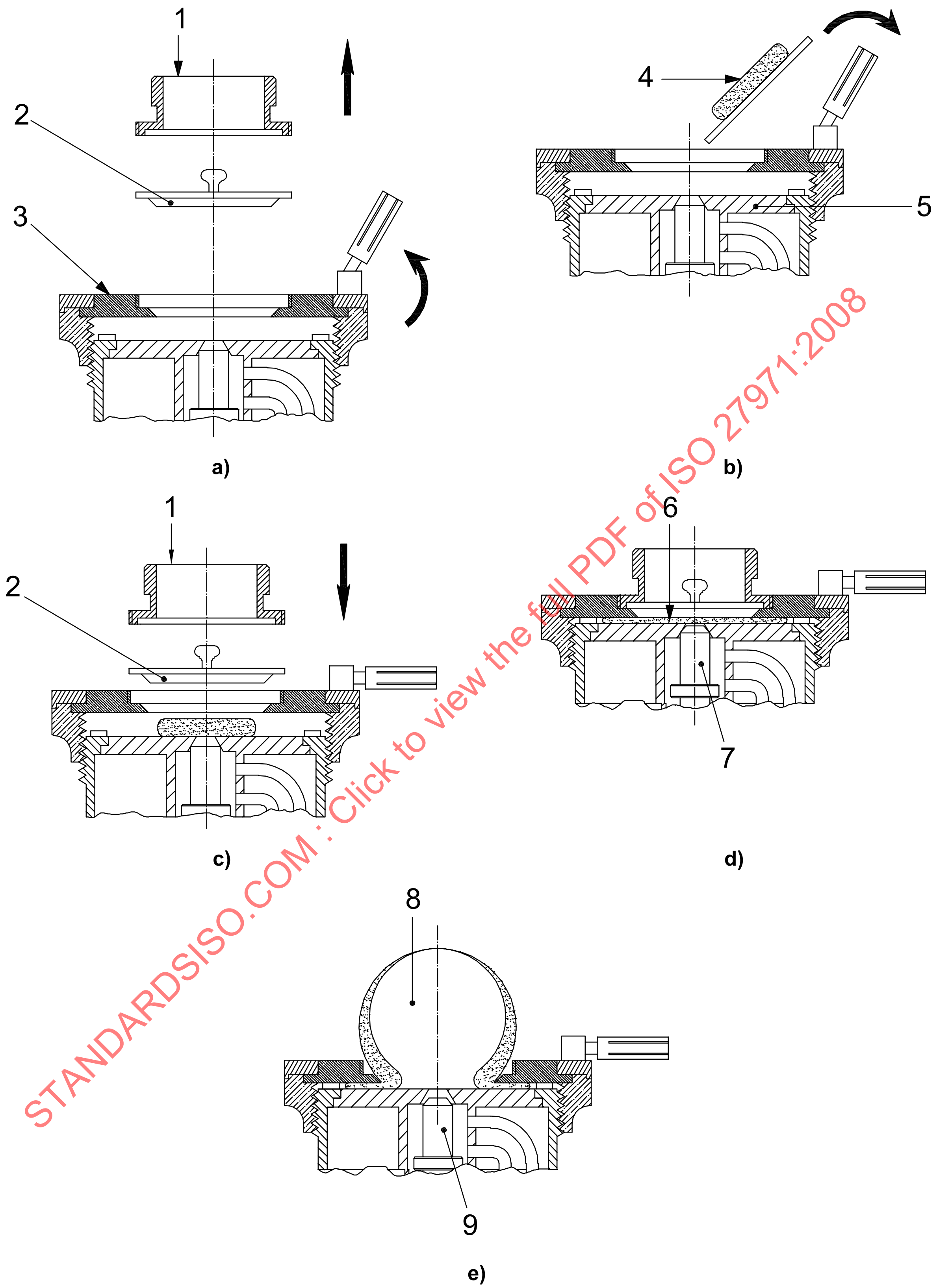
9.5.3 Stage 2: Biaxial extension

On the NG model, press the ON/OFF button to start the test.

On the MA 95 model, set handle A to position 2 [see Figure 1c)] to release the test piece automatically, inflate the dough bubble and start the recording drum.

On other models, the dough piece is released by setting the handle to an intermediate position. Turn the tap to the horizontal position, squeeze the rubber pear-shaped switch between the thumb and the index finger and without releasing the pressure, turn the tap to the vertical position. Set handle A to position 3 to begin inflation.

Watch as the bubble inflates, in order to determine the exact moment of rupture and immediately stop measuring by turning handle A to position 1, or by pressing the ON/OFF button on the NG models.



Key

1 ring
2 plug
3 top plate
4 dough test piece
5 bottom plate
6 calibrated dough test piece
7 piston in the raised position
8 inflated dough test piece
9 piston in low position

Figure 8 — Alveograph test

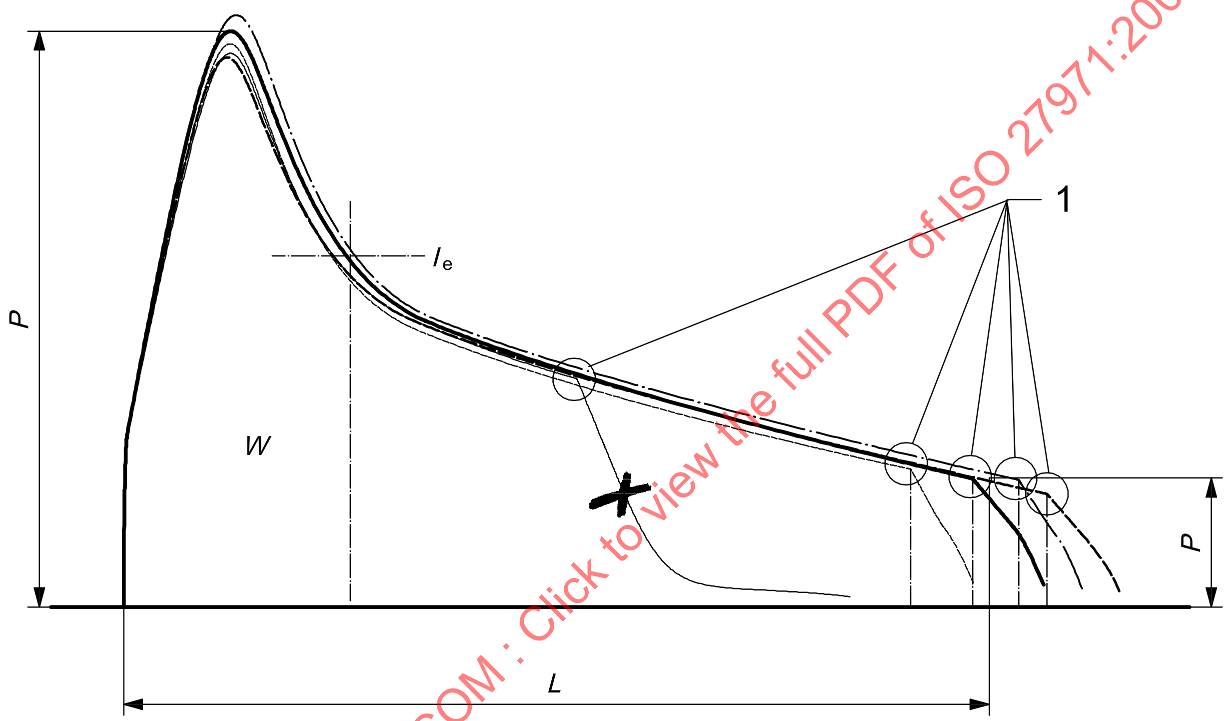
Where a recording drum is used, draw the five curves on the same sheet taking care to return the drum to its initial position before every trial.

Repeat the operations specified in 9.5.1 to 9.5.3 on the four remaining dough test pieces.

9.6 Expression of the results of the alveograph test

9.6.1 General

The results are measured or calculated from the five curves obtained. However, if one (and only one) of the curves deviates greatly from the other four, it should not be taken into account in the expression of results (see Figure 9).



Key

1 rupture points

I_e elasticity index, $(P_{200}/P) \times 100$, %

L mean abscissa at rupture

P maximum pressure parameter (mean of maximum ordinates times 1,1)

W deformation energy

Figure 9 — Alveograph curve

9.6.2 Maximum pressure parameter, P

P corresponds to the maximum pressure within the bubble, which is related to the deformation resistance (stiffness). The value of P equals the mean of the maximum ordinates, in millimetres, multiplied by a factor, $K = 1{,}1$.

Record P to the nearest integer.

9.6.3 Mean abscissa at rupture, L

The mean of the abscissa values at rupture for the curves represents the length, L. These abscissa values are measured, in millimetres, for each curve along the basis line, from the origin of the curves to the point corresponding vertically to where the pressure starts to drop abruptly.

Record L to the nearest integer.

9.6.4 Index of swelling, G

The mean of the abscissa values at rupture, L, converted into the index of swelling, G, represents the extensibility of the dough. This value is the square root of the volume of air, in millilitres, required to inflate the bubble until it ruptures. It is calculated using Equation (4):

$$G = 2{,}226\sqrt{L} \tag{4}$$

Annex D gives a conversion table from L to G.

Record G to the nearest first place of decimals.

9.6.5 Elasticity index

The elasticity index, I_e, expressed as a percentage, is calculated using Equation (5):

$$I_e = \left(\frac{P_{200}}{P}\right) \times 100 \tag{5}$$

where P_{200} is the pressure inside the bubble when a volume of 200 ml of air has been insufflated into the test piece.

Record I_e to the nearest first place of decimals.

9.6.6 Curve configuration ratio P/L

The term “curve configuration” is conventional.

Record P/L to the nearest second place of decimals.

9.6.7 Deformation energy, W

W represents the baking strength of the flour and the deformation energy of 1 g of dough obtained by the method described. It is expressed in units of 10^{-4} J. W is calculated, as indicated in Equation (6), from the alveogram parameters and various experimental factors.

$$W = 6{,}54\ S \tag{6}$$

where S is the area under the mean curve.

The coefficient 6,54 is valid for:

a) a drum rotation time of 55 s from stop to stop;

b) a constant air flow rate of 96 l/h;

c) a hydraulic manometer with the coefficient, $K = 1{,}1$.

Record W to the nearest integer.

10 Precision

10.1 Interlaboratory tests

The repeatability and reproducibility limits of the method used for commercial flour were established within the context of an interlaboratory test in which six laboratories participated. The test was performed on three samples of flour and repeated four times on each sample. The statistical analysis was performed as specified in ISO 5725 (Parts 2, 3, 6) [3], [4], [5]. The statistical results of the test are given in Annex E.

The repeatability and reproducibility limits of the method used for flour obtained by test milling were established by two interlaboratory tests.

Participants in the first round of tests numbered 10 and in the second 12. In total, 14 wheat samples were analysed and the test was repeated three times for each sample. The statistical analysis was performed as specified in ISO 5725 (Parts 2, 3, 6) [3], [4], [5]. The statistical results of the tests are given in Annex F.

The values obtained from each analysis apply to the concentration ranges and to the matrices tested.

10.2 Repeatability limits

10.2.1 General

Repeatability is the value below which there is a 95 % probability that the absolute value of the difference between two test results obtained under repeatability conditions will lie.

The repeatability limits, r, are obtained using Equations (7) to (17). To make it easier to use them, practical application tables are given in Annexes E and F.

10.2.2 Commercial flour

For W:

$$r = (0{,}054\,1\ W - 1{,}571\,5) \times 2{,}77 \quad (7)$$

For P:

$$r = (0{,}017\,3\ P + 0{,}310\,7) \times 2{,}77 \quad (8)$$

For L:

$$r = (0{,}144\,9\ L - 7{,}083) \times 2{,}77 \quad (9)$$

For G:

$$r = (0{,}121\,8G - 1{,}861\,7) \times 2{,}77 \quad (10)$$

For P/L:

$$r = [0{,}125(P/L) - 0{,}06] \times 2{,}77 \quad (11)$$

10.2.3 Flour obtained from test milling

For W:

$$r = (0{,}034\,4\ W + 3{,}903\,8) \times 2{,}77 \quad (12)$$

For P:

$$r = (0{,}026\,8\ P + 0{,}535) \times 2{,}77 \quad (13)$$

For L:

$$r = (0,049\ L + 2,347\ 1) \times 2,77 \quad (14)$$

For G:

$$r = 0,81 \times 2,77 = 2,25 \quad (15)$$

For P/L:

$$r = [0,121\ 5(P/L) - 0,015\ 4] \times 2,77 \quad (16)$$

For the extraction rate, ER:

$$r = 0,83 \times 2,77 = 2,29 \quad (17)$$

10.3 Reproducibility limits

10.3.1 General

Reproducibility is the value below which there is a 95 % probability that the absolute value of the difference between two test results obtained under reproducibility conditions will lie.

The reproducibility limits, R, are obtained using Equations (18) to (28). To make them easier to use, practical application tables are given in Annexes E and F.

10.3.2 Commercial flour

For W:

$$R = (0,059\ 5\ W + 0,569\ 6) \times 2,77 \quad (18)$$

For P:

$$R = (0,032\ 9\ P - 0,568\ 6) \times 2,77 \quad (19)$$

For L:

$$R = (0,139\ 3\ L - 5,132\ 1) \times 2,77 \quad (20)$$

For G:

$$R = (0,115\ 7\ G - 1,560\ 8) \times 2,77 \quad (21)$$

For P/L:

$$R = [0,125(P/L) - 0,04] \times 2,77 \quad (22)$$

10.3.3 Flour obtained from test milling

For W:

$$R = (0,053\ 4\ W + 4,195\ 1) \times 2,77 \quad (23)$$

For P:

$$R = (0,063\ 7\ P + 1,579\ 9) \times 2,77 \quad (24)$$

For L:

$$R = (0{,}099\,8\,L + 1{,}331\,1) \times 2{,}77 \qquad (25)$$

For G:

$$R = 1{,}25 \times 2{,}77 = 3{,}46 \qquad (26)$$

For P/L:

$$R = [0{,}210\,7(P/L) - 0{,}015\,4] \times 2{,}77 \qquad (27)$$

For the extraction rate, ER:

$$R = 3{,}79 \times 2{,}77 = 10{,}50 \qquad (28)$$

10.4 Uncertainty

10.4.1 General

Uncertainty, u, is a parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand (ISO Guide 98:1995 [8], 2.2.3). This uncertainty is given by a statistical distribution of the results from the interlaboratory test and is characterized by the experimental standard deviation.

For every parameter, the uncertainty is equal to plus or minus twice the reproducibility standard deviation given in this International Standard.

10.4.2 Commercial flour

For W:

$$u = \pm (0{,}059\,5\,W + 0{,}569\,6) \times 2 \qquad (29)$$

For P:

$$u = \pm (0{,}032\,9\,P - 0{,}568\,6) \times 2 \qquad (30)$$

For L:

$$u = \pm (0{,}139\,3\,L - 5{,}132\,1) \times 2 \qquad (31)$$

For G:

$$u = \pm (0{,}115\,7\,G - 1{,}560\,8) \times 2 \qquad (32)$$

For P/L:

$$u = \pm [0{,}125(P/L) - 0{,}04] \times 2 \qquad (33)$$

10.4.3 Flour obtained from test milling

For W:

$$u = \pm (0{,}053\,4\,W + 4{,}195\,1) \times 2 \qquad (34)$$

For P:

$$u = \pm (0{,}063\,7\,P - 1{,}579\,9) \times 2 \qquad (35)$$

For L:

$$u = \pm (0{,}099\,8\,L - 1{,}331\,1) \times 2 \quad (36)$$

For G:

$$u = \pm 1{,}25 \times 2 = \pm 2{,}50 \quad (37)$$

For P/L:

$$u = \pm [0{,}210\,7(P/L) - 0{,}015\,4] \times 2 \quad (38)$$

For the extraction rate, ER:

$$u = \pm 3{,}79 \times 2 = \pm 7{,}58 \quad (39)$$

11 Test report

The test report shall specify:

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

b) the sampling method used, if known;

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

d) when a test milling is included in the test, all the information necessary for a full identification of the mill used;

e) when a test milling is included in the test: the extraction rate expressed on the dry matter basis, the percentage of bran obtained from the break process, and the percentage of middlings obtained from the conversion process, or, if the repeatability has been checked, the final quoted results obtained for these parameters;

f) the alveograph parameters, or, if the repeatability has been checked, the final quoted results obtained for these parameters;

g) all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents noted during the milling process and alveograph test which may have influenced the test results.

Annex A
(informative)

Characteristics of the Chopin-Dubois CD1 mill

A.1 Break system

Three stacked hardened-steel fluted rollers with oblique teeth (2 passages).

Non-adjustable clearance:	First passage	1,00 mm
	Second passage	0,10 mm
Non-adjustable roller speeds:	Top roller	200 r/min
	Intermediate roller	450 r/min
	Bottom roller	200 r/min

A.2 Reduction system

Two smooth cast-iron rollers in contact (1 run) are cleaned by two scrapers; the pressure can be adjusted by adding or removing additional weights or by means of the spring pressure on the roller load.

Roller speed:	Top roller	325 r/min
	Bottom roller	325 r/min

A.3 Sieve material

A.3.1 Post-break

A.3.1.1 Stainless steel flour sieve, of wire diameter, 110 µm; of mesh aperture, 160 µm; and sieving area, 38 %.

A.3.1.2 Galvanized steel semolina sieve, of wire diameter, 315 µm; of mesh aperture, 800 µm; and sieving area, 51 %.

A.3.2 Post-reduction

As for A.3.1.1.

A.4 Milling

Break time: adjust the feed rate to allow 800 g of wheat to pass through the mill in (5 ± 1) min.

Reduction time: adjust the feed rate to allow the quantity of semolina obtained from the break system to pass through the mill in (5 ± 1) min.

Sieving time: continue sieving for (180 ± 30) s after the break system has finished. Do the same for the reduction system.

A.5 Break performance indicator

Irrespective of the type of wheat milled, the percentage of bran obtained from the break system shall be between 17 % and 23 % of the wheat mass used. A percentage outside this range indicates an incorrect setting or inadequate device maintenance.

A.6 Conversion performance indicator

Irrespective of the type of wheat milled, the percentage of middlings obtained from the conversion process shall be between 9 % and 17 % of the wheat mass used. A percentage outside this range indicates an incorrect setting or inadequate device maintenance.

A.7 Maintenance operations

Check the sieve screens regularly. The frequency recommended by the manufacturer is once per month. Replace sieves immediately if they are damaged, for example if they become detached or if any holes appear (do not patch them up). If they become obstructed, it is best to clean them with compressed air. Never wet the sieves.

Remove any metal particles from the safety magnet.

Use a shim to measure the amount of wear on the beater blades every 6 months. The space between the blade and the casing shall be less than 2 mm. Otherwise, replace the beater.

Check the amount of wear on the scrapers every year.

Replace the foam O-rings at least once a year or as soon as they become detached or begin to deteriorate.

It is recommended that every 2 years, a qualified engineer check the mechanical condition of the mill, and where necessary take remedial action, for:

a) the amount of wear on the bearings and scrapers;

b) the condition of the sieves;

c) the slope of the blades on the break and reduction sides;

d) the condition of the roll surface;

e) the tension of the reduction compression spring;

f) the condition of the wheat and semolina feed systems.

Annex B
(normative)

Quantity of water to be added to wheat for conditioning

The mass of water, M_e, in grams, to be added to wheat for the purposes of conditioning is calculated according to Equation (B.1):

$$M_e = [M_b(H_s - H_b)]/ (100 - H_s) \quad (B.1)$$

where

M_b is the wheat mass, in grams, to be conditioned;

H_b is the moisture content, as a percentage, of the wheat prior to conditioning;

H_s is the required moisture content, as a percentage, of the wheat after conditioning.

Express M_e to the nearest 0,1 g. The value of M_e is numerically equivalent to the volume, V_e, in millilitres, of water required.

Table B.1 — Moisture conditioning to 16 % by mass for 800 g of wheat

Initial wheat moisture content (before conditioning) H_b %	**Water mass or volume** M_e or V_e g or ml	**Initial wheat moisture content** (before conditioning) H_b %	**Water mass or volume** M_e or V_e g or ml	**Initial wheat moisture content** (before conditioning) H_b %	**Water mass or volume** M_e or V_e g or ml	**Initial wheat moisture content** (before conditioning) H_b %	**Water mass or volume** M_e or V_e g or ml
9,0	66,7	11,0	47,6	13,0	28,6	15,0	9.5
9,1	65,7	11,1	46,7	13,1	27,6		
9,2	64,8	11,2	45,7	13,2	26,7		
9,3	63,8	11,3	44,8	13,3	25,7		
9,4	62,9	11,4	43,8	13,4	24,8	If the moisture content of the wheat exceeds 15 %, dry the wheat before conditioning (see 7.4.4)	
9,5	61,9	11,5	42,9	13,5	23,8		
9,6	61,0	11,6	41,9	13,6	22,9		
9,7	60,0	11,7	41,0	13,7	21,9		
9,8	59,0	11,8	40,0	13,8	21,0		
9,9	58,1	11,9	39,0	13,9	20,0		
10,0	57,1	12,0	38,1	14,0	19,0	If the moisture content of the wheat is less than 13 %, add the conditioning water in two stages (see 7.4.3)	
10,1	56,2	12,1	37,1	14,1	18,1		
10,2	55,2	12,2	36,2	14,2	17,1		
10,3	54,3	12,3	35,2	14,3	16,2		
10,4	53,3	12,4	34,3	14,4	15,2		
10,5	52,4	12,5	33,3	14,5	14,3		
10,6	51,4	12,6	32,4	14,6	13,3		
10,7	50,5	12,7	31,4	14,7	12,4		
10,8	49,5	12,8	30,5	14,8	11,4		
10,9	48,6	12,9	29,5	14,9	10,5		

Annex C
(informative)

Sample milling sheet

Users of this International Standard may copy this sheet for practical use.

		WHEAT SAMPLE REFERENCE:
Initial moisture content (%), of cleaned wheat	H_{b0}	
→ if $H_{b0} > 15$ %, pre-dry (see 7.4.4)		start of predrying: date: ______ time: ______ end of predrying: date: ______ time: ______
Moisture content (%) of cleaned wheat after pre-drying, if any (if no pre-drying, $H_{b0} = H_b$)	H_b	
Quantity of water (g or ml) to be added to condition to 16 % (see Annex B)	M_e or V_e	
A. if $H_b < 13$ % by mass, add the water in two stages (see 7.4.3)		1. add M_1 g of water M_1 = ___ g: date: ___ time: ___ 2. add M_2 g of water M_2 = ___ g: date: ___ time: ___ $M_1 + M_2 = M_e$
B. if 15 % $\geqslant H_b \geqslant$ 13 %, add all the water at the same time (see 7.4.2)		add M_e g of water = _____ g: date: _____ time: _____
Cleaned wheat mass for milling (g)	M_b	
Cleaned wheat mass for milling expressed as dry matter (g)	MS_b	$= [M_b \times (100 - H_b)/100] =$
Total mass for milling (g)	T_1	$= M_b + M_e =$
Test mill identification:		
Start of milling:		date: __________ time: ________
Flour mass after break system (g)	M_{br}	
Semolina mass after break system (g)	M_{sem}	
Reduction flour mass (g)	M_{conv}	
Number of reductions	N	
Total flour mass (g)	M_f	$= M_{br} + M_{conv} =$
Bran mass (g)	M_s	i.e. $S =$ %
Middling mass (g)	M_r	i.e. $R =$ %
Total mass of milled products (g)	M_{tot}	$= M_f + M_s + M_r =$
Flour moisture content (%)	H_f	
Total flour mass expressed as dry matter (g)	MS_f	$= M_f \times (100 - H_f)/100 =$
Extraction rate, dry matter/dry matter (%)	ER	$= (MS_f/MS_b) \times 100 =$
TOTAL RECOVERY (%)	BM	$= (T_2/T_1) \times 100 =$

Ash yield of the flour (% dry matter):		*(if determined)*
Damaged starch content of the flour:		*(if determined)*
Breaking time (min):		
Reduction time (min):		

Annex D
(informative)

Conversion table for L to G

Table D.1 — Conversion of the length, L, into swelling, G, according to Equation (4): $G = 2{,}226\sqrt{L}$

Length L mm	Swelling G ml	Length L mm	Swelling G ml	Length L mm	Swelling G ml	Length L mm	Swelling G ml	Length L mm	Swelling G ml
13,0	8,0	63,0	17,7	113,0	23,7	163,0	28,4	213,0	32,5
14,0	8,3	64,0	17,8	114,0	23,8	164,0	28,5	214,0	32,6
15,0	8,6	65,0	17,9	115,0	23,9	165,0	28,6	215,0	32,6
16,0	8,9	66,0	18,1	116,0	24,0	166,0	28,7	216,0	32,7
17,0	9,2	67,0	18,2	117,0	24,1	167,0	28,8	217,0	32,8
18,0	9,4	68,0	18,4	118,0	24,2	168,0	28,9	218,0	32,9
19,0	9,7	69,0	18,5	119,0	24,3	169,0	28,9	219,0	32,9
20,0	10,0	70,0	18,6	120,0	24,4	170,0	29,0	220,0	33,0
21,0	10,2	71,0	18,8	121,0	24,5	171,0	29,1	221,0	33,1
22,0	10,4	72,0	18,9	122,0	24,6	172,0	29,2	222,0	33,2
23,0	10,7	73,0	19,0	123,0	24,7	173,0	29,3	223,0	33,2
24,0	10,9	74,0	19,1	124,0	24,8	174,0	29,4	224,0	33,3
25,0	11,1	75,0	19,3	125,0	24,9	175,0	29,4	225,0	33,4
26,0	11,4	76,0	19,4	126,0	25,0	176,0	29,5	226,0	33,5
27,0	11,6	77,0	19,5	127,0	25,1	177,0	29,6	227,0	33,5
28,0	11,8	78,0	19,7	128,0	25,2	178,0	29,7	228,0	33,6
29,0	12,0	79,0	19,8	129,0	25,3	179,0	29,8	229,0	33,7
30,0	12,2	80,0	19,9	130,0	25,4	180,0	29,9	230,0	33,8
31,0	12,4	81,0	20,0	131,0	25,5	181,0	29,9	231,0	33,8
32,0	12,6	82,0	20,2	132,0	25,6	182,0	30,0	232,0	33,9
33,0	12,8	83,0	20,3	133,0	25,7	183,0	30,1	233,0	34,0
34,0	13,0	84,0	20,4	134,0	25,8	184,0	30,2	234,0	34,1
35,0	13,2	85,0	20,5	135,0	25,9	185,0	30,3	235,0	34,1
36,0	13,4	86,0	20,6	136,0	26,0	186,0	30,4	236,0	34,2
37,0	13,5	87,0	20,8	137,0	26,1	187,0	30,4	237,0	34,3
38,0	13,7	88,0	20,9	138,0	26,1	188,0	30,5	238,0	34,3
39,0	13,9	89,0	21,0	139,0	26,2	189,0	30,6	239,0	34,4
40,0	14,1	90,0	21,1	140,0	26,3	190,0	30,7	240,0	34,5
41,0	14,3	91,0	21,2	141,0	26,4	191,0	30,8	241,0	34,6
42,0	14,4	92,0	21,4	142,0	26,5	192,0	30,8	242,0	34,6

Table D.1 *(continued)*

Length L mm	Swelling G ml	Length L mm	Swelling G ml	Length L mm	Swelling G ml	Length L mm	Swelling G ml	Length L mm	Swelling G ml
43,0	14,6	93,0	21,5	143,0	26,6	193,0	30,9	243,0	34,7
44,0	14,8	94,0	21,6	144,0	26,7	194,0	31,0	244,0	34,8
45,0	14,9	95,0	21,7	145,0	26,8	195,0	31,1	245,0	34,8
46,0	15,1	96,0	21,8	146,0	26,9	196,0	31,2	246,0	34,9
47,0	15,3	97,0	21,9	147,0	27,0	197,0	31,2	247,0	35,0
48,0	15,4	98,0	22,0	148,0	27,1	198,0	31,3	248,0	35,1
49,0	15,6	99,0	22,1	149,0	27,2	199,0	31,4	249,0	35,1
50,0	15,7	100,0	22,3	150,0	27,3	200,0	31,5	250,0	35,2
51,0	15,9	101,0	22,4	151,0	27,4	201,0	31,6	251,0	35,3
52,0	16,1	102,0	22,5	152,0	27,4	202,0	31,6	252,0	35,3
53,0	16,2	103,0	22,6	153,0	27,5	203,0	31,7	253,0	35,4
54,0	16,4	104,0	22,7	154,0	27,6	204,0	31,8	254,0	35,5
55,0	16,5	105,0	22,8	155,0	27,7	205,0	31,9	255,0	35,5
56,0	16,7	106,0	22,9	156,0	27,8	206,0	31,9	256,0	35,6
57,0	16,8	107,0	23,0	157,0	27,9	207,0	32,0	257,0	35,7
58,0	17,0	108,0	23,1	158,0	28,0	208,0	32,1	258,0	35,8
59,0	17,1	109,0	23,2	159,0	28,1	209,0	32,2	259,0	35,8
60,0	17,2	110,0	23,3	160,0	28,2	210,0	32,3	260,0	35,9
61,0	17,4	111,0	23,5	161,0	28,2	211,0	32,3	261,0	36,0
62,0	17,5	112,0	23,6	162,0	28,3	212,0	32,4	262,0	36,0

Annex E
(informative)

Interlaboratory data for commercial flour

Table E.1 — Statistical results on commercial flour

Parameter	Flour 1					Flour 2					Flour 3				
	W	P	L	P/L	G	W	P	L	P/L	G	W	P	L	P/L	G
	10^{-4} J	mm	mm	—	ml	10^{-4} J	mm	mm	—	ml	10^{-4} J	mm	mm	—	ml
No. laboratories (after outlier removal)	6	5	6	6	6	6	6	6	6	6	6	6	6	6	6
Overall mean	191,04	69,95	77,87	0,92	19,60	235,93	80,67	88,21	0,92	20,85	413,67	117,96	93,33	1,28	21,43
Standard deviation of repeatability, s_r	6,56	1,10	4,15	0,05	0,52	13,96	2,24	5,84	0,06	0,70	20,26	2,23	6,34	0,10	0,74
Repeatability limit, r (= 2,77 × s_r)	18,17	3,05	11,50	0,14	1,44	38,66	6,20	16,18	0,17	1,94	56,12	6,18	17,56	0,29	2,05
Coefficient of variation of repeatability, CV(r) (%)	3,43	1,58	5,33	5,95	2,65	5,92	2,77	6,62	6,66	3,34	4,90	1,89	6,79	7,95	3,44
Standard deviation of reproducibility, s_R	10,85	1,44	5,67	0,07	0,70	15,94	2,45	7,31	0,08	0,87	24,90	3,23	7,77	0,12	0,90
Reproducibility limit, R (= 2,77 × s_R)	30,05	3,99	15,71	0,19	1,94	44,15	6,79	20,25	0,22	2,41	68,97	8,95	21,52	0,33	2,49
Coefficient of variation of reproducibility CV(R) (%)	5,68	2,06	7,28	7,98	3,57	6,76	3,04	8,28	8,90	4,18	6,02	2,73	8,33	9,58	4,22

Table E.2 — Practical illustration of repeatability equations for commercial flour

W **Validity range:** **190 to 415** $s_r = 0{,}054\,1\,W - 1{,}571\,5$		P **Validity range:** **70 to 118** $s_r = 0{,}017\,3\,P + 0{,}310\,7$		L **Validity range:** **78 to 98** $s_r = 0{,}144\,9\,L - 7{,}083$		G **Validity range:** **19,5 to 21,5** $s_r = 0{,}121\,8\,G - 1{,}861\,7$		P/L **Validity range:** **0,92 to 1,28** $s_r = 0{,}125(P/L) - 0{,}06$	
W 10^{-4} J	**Repeatability limit** ($r = 2{,}77\ s_r$)	P mm	**Repeatability limit** ($r = 2{,}77\ s_r$)	L mm	**Repeatability limit** ($r = 2{,}77\ s_r$)	G ml	**Repeatability limit** ($r = 2{,}77\ s_r$)	P/L	**Repeatability limit** ($r = 2{,}77\ s_r$)
190	24	70	4	78	12	19,5	1,4	0,92	0,15
200	26	72	4	79	12	19,6	1,5	0,94	0,16
210	27	74	4	80	12	19,7	1,5	0,95	0,16
220	29	76	5	81	13	19,8	1,5	0,97	0,17
230	30	78	5	82	13	19,9	1,6	0,98	0,17
240	32	80	5	83	14	20,0	1,6	1,00	0,18
250	33	82	5	84	14	20,1	1,6	1,01	0,18
260	35	84	5	85	14	20,2	1,7	1,03	0,19
270	36	86	5	86	15	20,3	1,7	1,04	0,19
280	38	88	5	87	15	20,4	1,7	1,06	0,20
290	39	90	5	88	16	20,5	1,8	1,07	0,20
300	41	92	5	89	16	20,6	1,8	1,09	0,21
310	42	94	5	90	17	20,7	1,8	1,10	0,21
320	44	96	5	91	17	20,8	1,9	1,12	0,22
330	45	98	6	92	17	20,9	1,9	1,13	0,23
340	47	100	6	93	18	21,0	1,9	1,15	0,23
350	48	102	6	94	18	21,1	2,0	1,16	0,24
360	50	104	6	95	19	21,2	2,0	1,18	0,24
370	51	106	6	96	19	21,3	2,0	1,19	0,25
380	53	108	6	97	19	21,4	2,1	1,21	0,25
390	54	110	6	98	20	21,5	2,1	1,22	0,26
400	56	112	6					1,24	0,26
410	57	114	6					1,25	0,27
415	58	116	6					1,27	0,27
		118	7					1,28	0,28

Table E.3 — Practical illustration of reproducibility equations for commercial flour

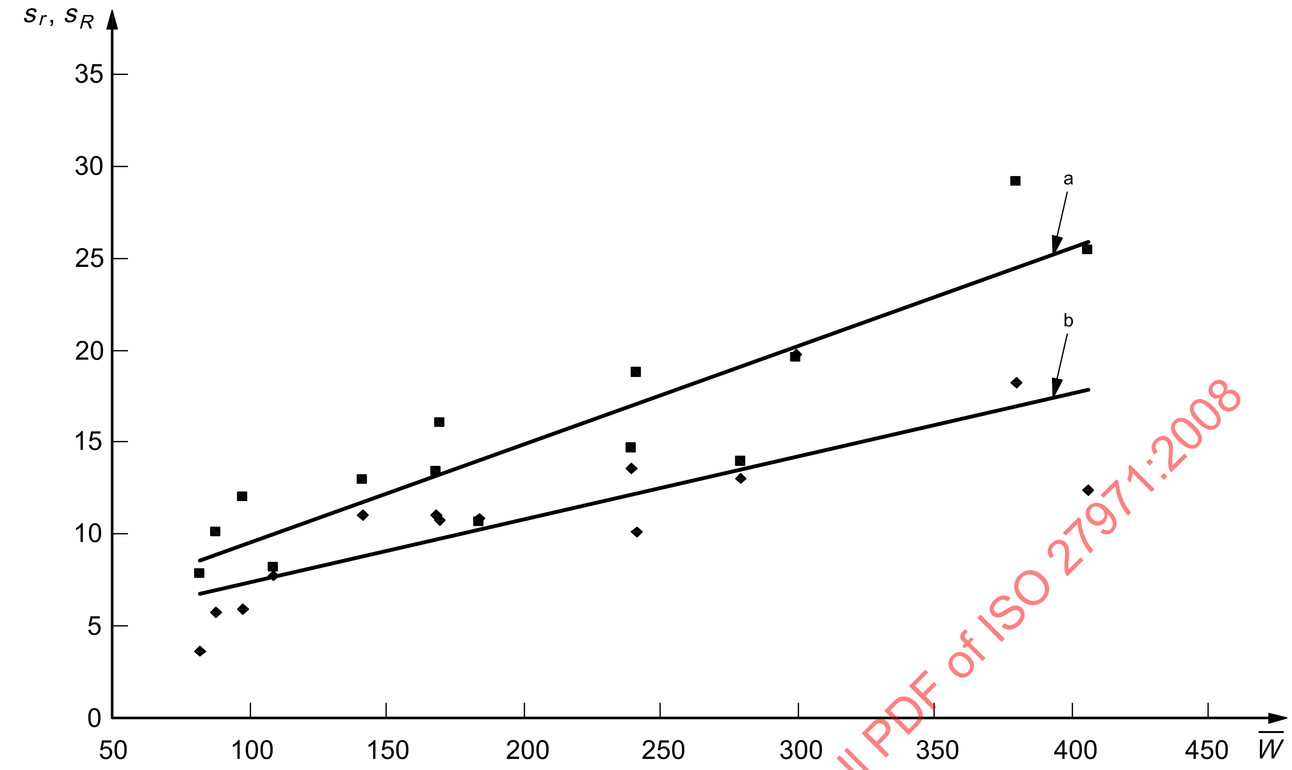
W **Validity range:** **190 to 415** $s_R = 0{,}059\,5\,W + 0{,}569\,6$		*P* **Validity range:** **70 to 118** $s_R = 0{,}032\,9\,P - 0{,}568\,6$		*L* **Validity range:** **78 to 98** $s_R = 0{,}139\,3\,L - 5{,}132\,1$		*G* **Validity range:** **19,5 to 21,5** $s_R = 0{,}115\,7\,G - 1{,}560\,8$		*P/L* **Validity range:** **0,92 to 1,28** $s_R = 0{,}125(P/L) - 0{,}04$	
W 10^{-4} J	**Reproducibility limit** ($R = 2{,}77\ s_R$)	*P* mm	**Reproducibility limit** ($R = 2{,}77\ s_R$)	*L* mm	**Reproducibility limit** ($R = 2{,}77\ s_R$)	*G* ml	**Reproducibility limit** ($R = 2{,}77\ s_R$)	*P/L*	**Reproducibility limit** ($R = 2{,}77\ s_R$)
190	33	70	5	78	16	19,5	1,9	0,92	0,21
200	35	72	5	79	16	19,6	2,0	0,94	0,21
210	36	74	5	80	17	19,7	2,0	0,95	0,22
220	38	76	5	81	17	19,8	2,0	0,97	0,22
230	39	78	6	82	17	19,9	2,1	0,98	0,23
240	41	80	6	83	18	20,0	2,1	1,00	0,23
250	43	82	6	84	18	20,1	2,1	1,01	0,24
260	44	84	6	85	19	20,2	2,2	1,03	0,24
270	46	86	6	86	19	20,3	2,2	1,04	0,25
280	48	88	6	87	19	20,4	2,2	1,06	0,25
290	49	90	7	88	20	20,5	2,2	1,07	0,26
300	51	92	7	89	20	20,6	2,3	1,09	0,26
310	53	94	7	90	21	20,7	2,3	1,10	0,27
320	54	96	7	91	21	20,8	2,3	1,12	0,28
330	56	98	7	92	21	20,9	2,4	1,13	0,28
340	58	100	8	93	22	21,0	2,4	1,15	0,29
350	59	102	8	94	22	21,1	2,4	1,16	0,29
360	61	104	8	95	22	21,2	2,5	1,18	0,30
370	63	106	8	96	23	21,3	2,5	1,19	0,30
380	64	108	8	97	23	21,4	2,5	1,21	0,31
390	66	110	8	98	24	21,5	2,6	1,22	0,31
400	68	112	9					1,24	0,32
410	69	114	9					1,25	0,32
415	70	116	9					1,27	0,33
		118	9					1,28	0,33

Annex F
(informative)

Interlaboratory data for laboratory milled flour

Table F.1 — Statistical results for W on laboratory milled flour

Wheat	10	7	8	5	1	2	12	3	11	4	14	9	13	6
No. laboratories	10	10	10	10	9	10	10	10	10	9	10	10	10	10
Mean, $\overline{W}$	82	88	98	109	141	168	170	184	240	242	279	300	380	406
s_r	4	6	6	8	11	11	11	11	14	10	13	20	18	12
CV(r) %	4	6	6	7	8	7	6	6	6	4	5	7	5	3
r	10	16	16	22	31	31	30	30	38	28	36	55	51	34
s_R	8	10	12	8	13	13	16	11	15	19	14	20	29	25
CV(R) %	10	12	12	7	9	8	9	6	6	8	5	7	8	6
R	22	28	33	23	36	37	45	30	41	52	39	54	81	70



Key

$\overline{W}$ mean of W

s_r standard deviation of repeatability

s_R standard deviation of reproducibility

a s_r; $y = 0{,}034\,4\,x + 3{,}903\,8$; $R^2 = 0{,}655$ (correlation coefficient)

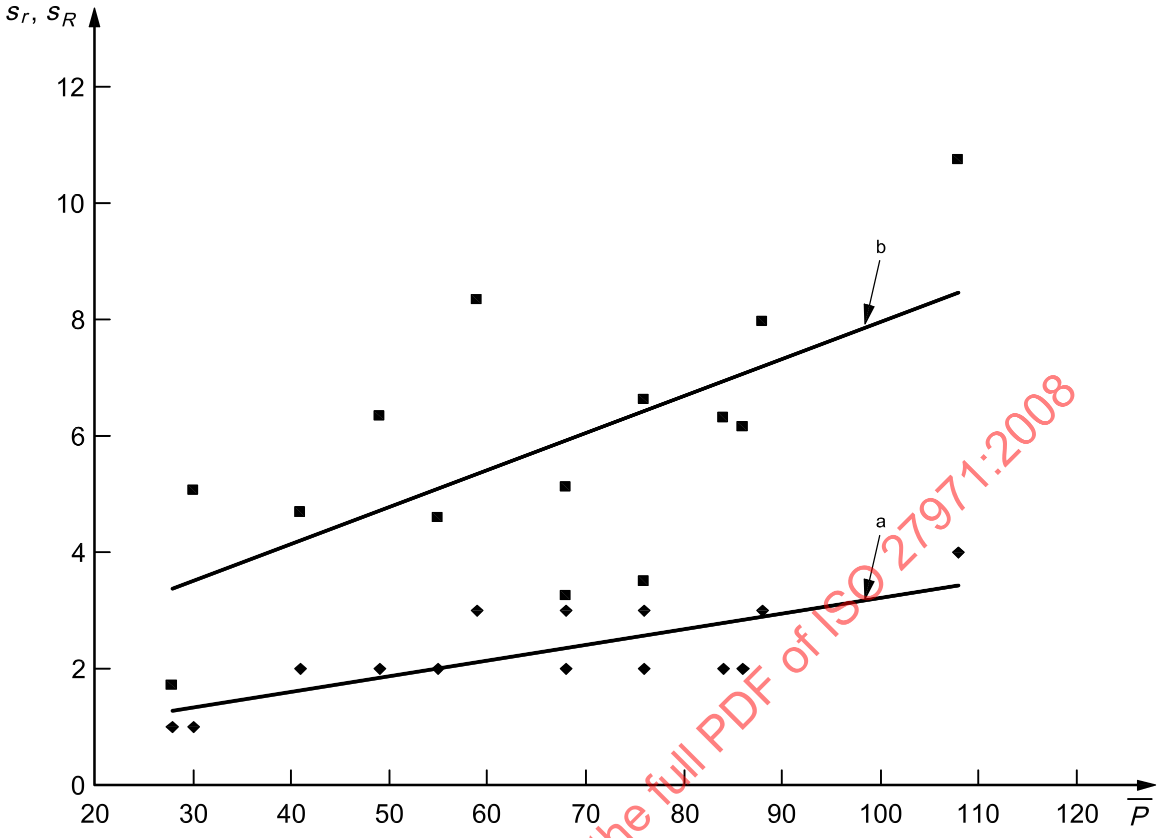
b s_R; $y = 0{,}053\,4\,x + 4{,}195\,1$; $R^2 = 0{,}821\,1$ (correlation coefficient)

Figure F.1 — Relationship between precision standard deviation and mean values of W

Figure F.1 shows that the standard deviations of repeatability and reproducibility are dependent on the arithmetic average value of W.

Table F.2 — Statistical results for P on laboratory milled flour

Wheat	5	8	10	12	4	7	1	2	3	13	14	6	11	9
No. laboratories	10	10	10	10	9	10	9	9	10	10	10	10	10	10
Mean, $\overline{P}$	28	30	41	49	55	59	68	68	76	76	84	86	88	108
s_r	1	1	2	2	2	3	3	2	2	3	2	2	3	4
CV(r) %	2	4	4	3	3	5	5	3	3	4	3	2	4	4
r	2	4	5	4	4	9	9	5	6	8	7	6	10	11
s_R	2	5	5	6	5	8	5	3	4	7	6	6	8	11
CV(R) %	6	17	11	13	8	14	8	5	5	9	8	7	9	10
R	5	14	13	18	13	23	14	9	10	18	17	17	22	30



Key

$\overline{P}$ mean of P

s_r standard deviation of repeatability

s_R standard deviation of reproducibility

[a] s_r; $y = 0{,}026\,8\,x + 0{,}535$; $R^2 = 0{,}565\,8$ (correlation coefficient)

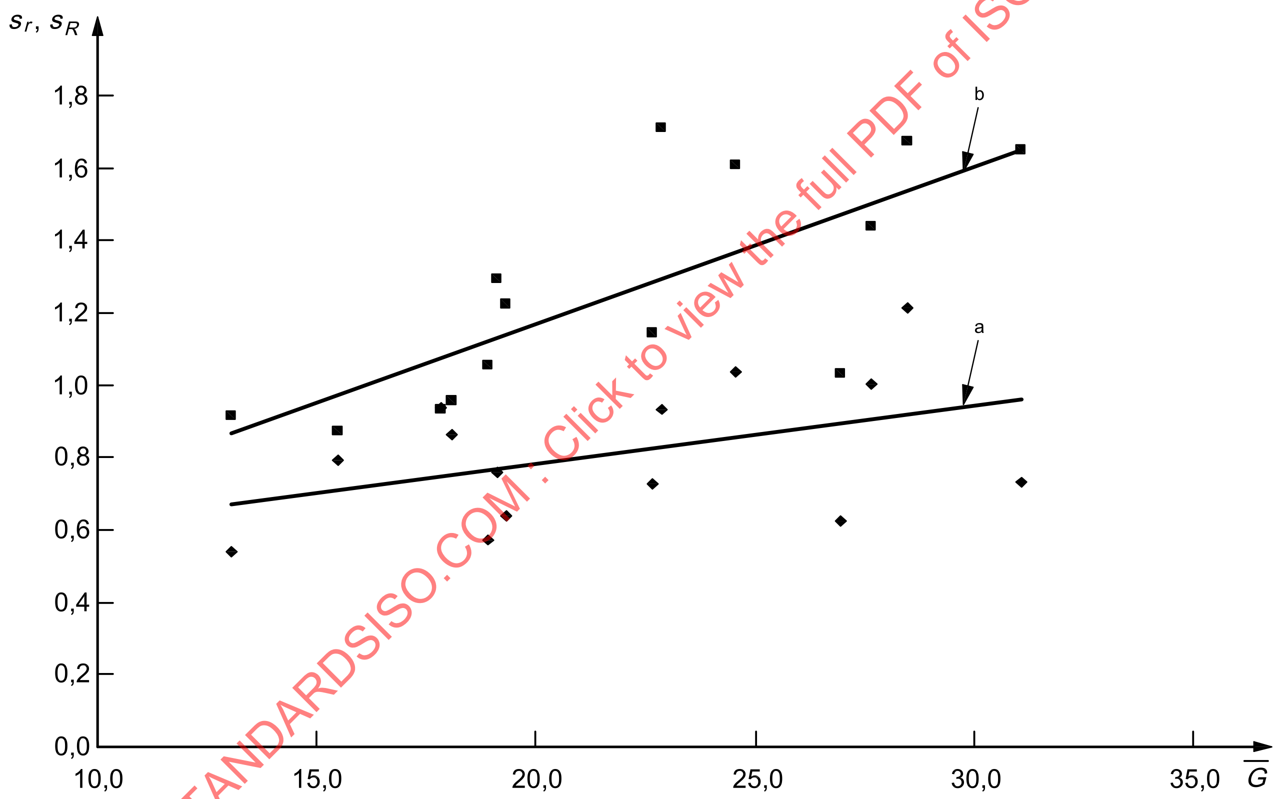
[b] s_R; $y = 0{,}063\,7\,x + 1{,}579\,9$; $R^2 = 0{,}411\,8$ (correlation coefficient)

Figure F.2 — Relationship between precision standard deviation and mean values of *P*

Figure F.2 shows that the standard deviations of repeatability and reproducibility are dependent on the arithmetic average value of P.

Table F.3 — Statistical results for G on laboratory milled flour

Wheat	7	1	3	2	11	9	10	14	8	12	6	5	13	4
No. laboratories	10	9	10	10	10	10	10	10	10	10	10	10	10	9
Mean, $\overline{G}$	13,1	15,5	17,8	18,1	18,9	19,1	19,3	22,7	22,9	24,6	26,9	27,7	28,5	31,1
s_r	0,5	0,8	0,9	0,9	0,6	0,8	0,6	0,7	0,9	1,0	0,6	1,0	1,2	0,7
CV(r) %	4,1	5,1	5,3	4,8	3,0	4,0	3,3	3,2	4,1	4,2	2,3	3,6	4,3	2,4
r	1,5	2,2	2,6	2,4	1,6	2,1	1,8	2,0	2,6	2,9	1,7	2,8	3,4	2,0
s_R	0,9	0,9	0,9	1,0	1,1	1,3	1,2	1,1	1,7	1,6	1,0	1,4	1,7	1,6
CV(R) %	7,0	5,6	5,2	5,3	5,6	6,8	6,3	5,0	7,5	6,5	3,8	5,2	5,9	5,3
R	2,5	2,4	2,6	2,7	2,9	3,6	3,4	3,2	4,7	4,5	2,9	4,0	4,6	4,6



Key

$\overline{G}$ mean of G

s_r standard deviation of repeatability

s_R standard deviation of reproducibility

[a] s_r; $y = 0{,}016\,x + 0{,}462\,6$; $R^2 = 0{,}19$ (correlation coefficient)

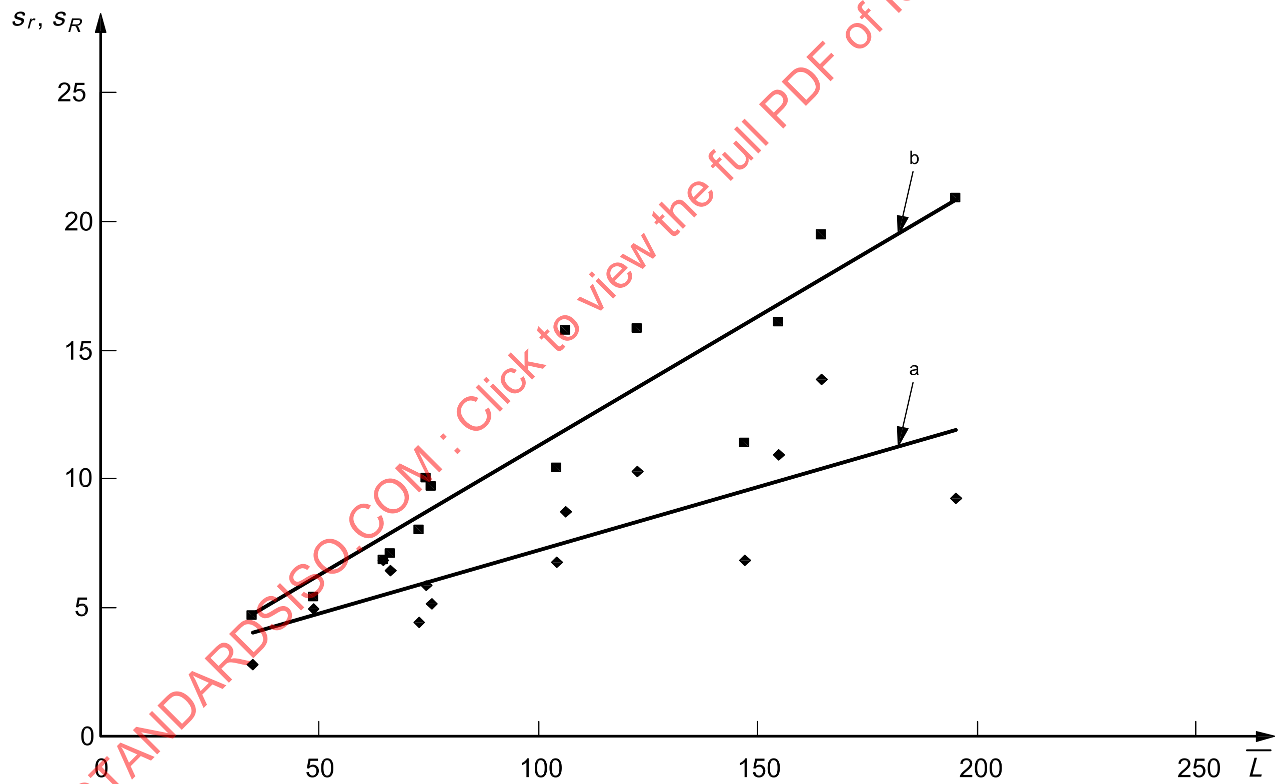
[b] s_R; $y = 0{,}043\,7\,x + 0{,}295\,2$; $R^2 = 0{,}559\,4$ (correlation coefficient)

Figure F.3 — Relationship between precision standard deviation and mean values of G

The standard deviations of repeatability and reproducibility are considered to be independent of the mean of G (see Figure F.3).

Table F.4 — Statistical results for L on laboratory milled flour

Wheat	7	1	3	2	11	9	10	14	8	12	6	5	13	4
No. laboratories	10	9	10	10	10	10	10	10	10	10	10	10	10	9
Mean, $\overline{L}$	35	49	64	66	73	74	76	104	106	123	147	155	164	195
s_r	3	5	7	6	4	6	5	7	9	10	7	11	14	9
CV(r) %	8	10	11	10	6	8	7	7	8	8	5	7	8	5
r	8	14	19	18	12	16	14	19	24	28	19	30	38	26
s_R	5	5	7	7	8	10	10	10	16	16	11	16	19	21
CV(R) %	14	11	11	11	11	13	13	10	15	13	8	10	12	11
R	13	15	19	20	22	28	27	29	44	44	32	44	54	58



Key

$\overline{L}$ mean of L

s_r standard deviation of repeatability

s_R standard deviation of reproducibility

a s_r; $y = 0{,}049\ x + 2{,}347\ 1$; $R^2 = 0{,}644$ (correlation coefficient)

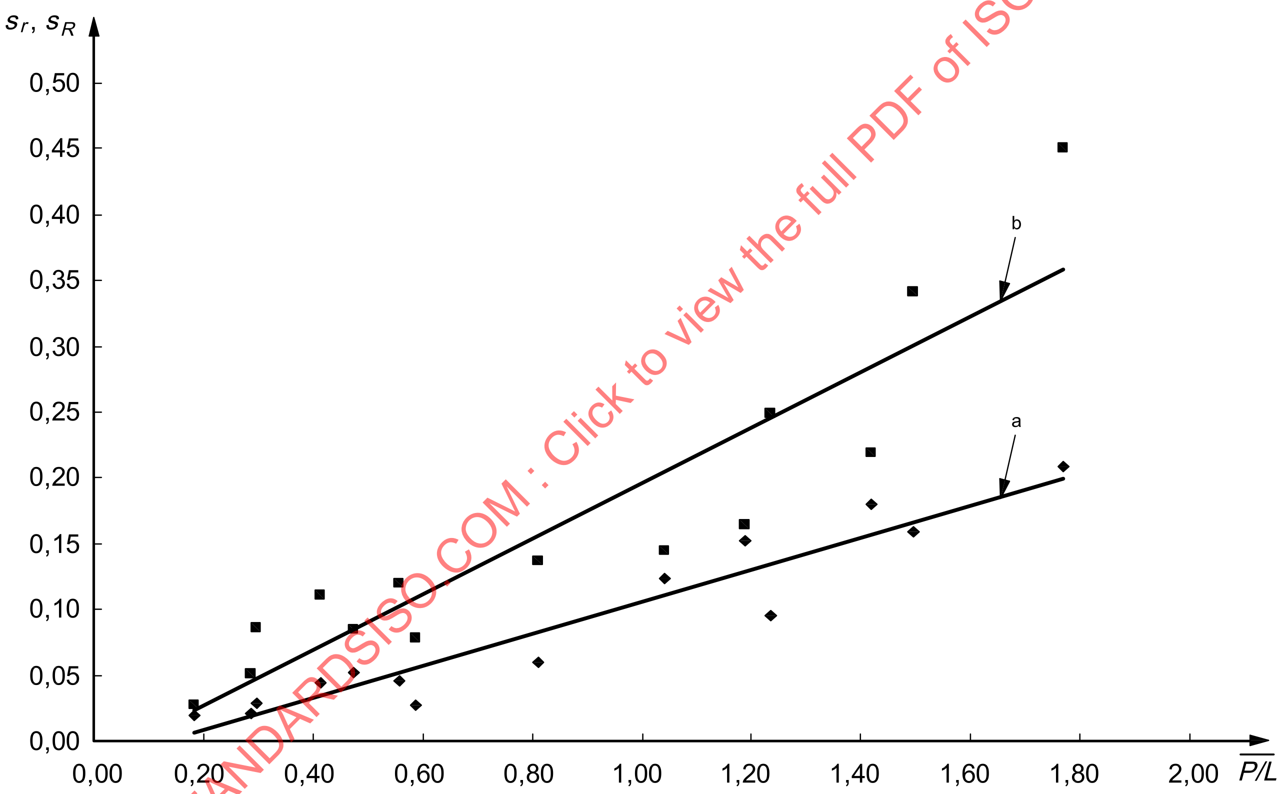
b s_R; $y = 0{,}099\ 8\ x + 1{,}331\ 1$; $R^2 = 0{,}857\ 1$ (correlation coefficient)

Figure F.4 — Relationship between precision standard deviation and mean values of L

Figure F.4 shows that the standard deviations of repeatability and reproducibility are dependent on the arithmetic average value of L.

Table F.5 — Statistical results for P/L on laboratory milled flour

Wheat	5	4	8	12	13	10	6	14	2	3	11	1	9	7
No. laboratories	10	9	10	10	10	10	10	10	10	10	10	9	10	10
Mean, $\overline{P/L}$	0,18	0,29	0,30	0,41	0,47	0,56	0,59	0,81	1,04	1,19	1,24	1,42	1,49	1,77
s_r	0,02	0,02	0,03	0,04	0,05	0,05	0,03	0,06	0,12	0,15	0,10	0,18	0,16	0,21
CV(r) %	10,28	7,15	9,62	10,66	10,98	8,26	4,72	7,29	11,83	12,87	7,71	12,64	10,60	11,75
r	0,05	0,06	0,08	0,12	0,14	0,13	0,08	0,16	0,34	0,42	0,26	0,50	0,44	0,58
s_R	0,03	0,05	0,09	0,11	0,09	0,12	0,08	0,14	0,14	0,16	0,25	0,22	0,34	0,45
CV(R) %	15,11	17,74	28,88	26,83	17,93	21,58	13,23	16,74	13,90	13,82	20,08	15,46	22,84	25,42
R	0,08	0,14	0,24	0,31	0,24	0,33	0,22	0,38	0,40	0,46	0,69	0,61	0,95	1,25



Key

$\overline{P/L}$ mean of P/L

s_r standard deviation of repeatability

s_R standard deviation of reproducibility

a s_r; $y = 0{,}121\,5\,x - 0{,}015\,4$; $R^2 = 0{,}915\,1$ (correlation coefficient)

b s_R; $y = 0{,}210\,7\,x - 0{,}015\,4$; $R^2 = 0{,}846\,4$ (correlation coefficient)

Figure F.5 — Relationship between precision standard deviation and mean values of P/L

Figure F.5 shows that the standard deviations of repeatability and reproducibility are dependent on the arithmetic average value of P/L.

