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Wheat flour — Physical characteristics of doughs —

Part 4:

Determination of rheological properties using an
alveograph

Farines de blé tendre — Caractéristiques physiques des pâtes —

*Partie 4: Détermination des caractéristiques rhéologiques au moyen de
l'alvéographe*



Reference number
ISO 5530-4:1991(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 5530-4 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 5530-4:1983), of which it constitutes a technical revision.

This part of ISO 5530 is based on Standard No. 121 of the International Association for Cereal Science and Technology (ICC).

ISO 5530 consists of the following parts, under the general title *Wheat flour — Physical characteristics of doughs*:

- *Part 1: Determination of water absorption and rheological properties using a farinograph*
- *Part 2: Determination of rheological properties using an extensograph*
- *Part 3: Determination of water absorption and rheological properties using a valorigraph*
- *Part 4: Determination of rheological properties using an alveograph*

Annex A forms an integral part of this part of ISO 5530. Annex B is for information only.

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Wheat flour — Physical characteristics of doughs —

Part 4:

Determination of rheological properties using an alveograph

1 Scope

This part of ISO 5530 specifies a method, using an alveograph, for determining certain rheological properties, in particular, the maximum overpressure p , the index of swelling G , the average abscissa at rupture l and the deformation energy W .

The method is applicable to doughs made from wheat flour (*Triticum aestivum* Linnaeus).

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this part of ISO 5530. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5530 are encouraged to investigate the possibility of applying the most recent edition of the standard 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)*.

3 Principle

Preparation of a dough of constant moisture content from wheat flour and sodium chloride solution under specified conditions. Preparation of test pieces of specified thickness from the dough. After a period of rest, biaxial extension of the test pieces by inflating them into the form of a bubble. Plotting of the variation in pressure inside the bubble as a function of time. Assessment of the properties of the dough from the shape and area of the diagrams obtained.

4 Reagents

Use only reagents of recognized analytical quality, unless otherwise specified, and distilled or demineralized water or water of equivalent purity.

4.1 Sodium chloride, solution, prepared as follows.

Dissolve 25 g of sodium chloride, food grade, in the water and make up to 1 000 ml.

4.2 Refined vegetable oil, low in polyunsaturates, with an acid value less than 0,4 (see ISO 660), such as olive oil or groundnut oil; store in a dark place in a stoppered container and replace regularly (every 3 months). Alternatively, **paraffin oil** (also known as vaseline oil), sold by pharmacists under the name of *petrolatum liquidum* (liquid paraffin), which is a purified mixture of natural, liquid, saturated hydrocarbons obtained from petroleum, with an acid value less than or equal to 0,05. Use paraffin oil with the lowest possible viscosity [not more than 60 mPa·s (60 cP) at 20 °C].

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Alveograph¹⁾, with a temperature regulator, and having the following characteristics.

NOTE 1 Unless otherwise specified in the text, the characteristics given apply to both the old and the new model.

- Rotational frequency of the mixer blade: $(60 \pm 1) \text{ min}^{-1}$

NOTE 2 Some older models have a rotational frequency of the mixer blade of $(59 \pm 1) \text{ min}^{-1}$. This different frequency will not, however, have any effect on the results.

- Height of the sheeting guides: $(12,0 \pm 0,1) \text{ mm}$
- Diameter of the sheeting roller
 - large diameter: $(40,0 \pm 0,1) \text{ mm}$
 - small diameter: $(33,3 \pm 0,1) \text{ mm}$
- Inner diameter of the dough cutter: $(46,0 \pm 0,5) \text{ mm}$
- Diameter of the opening in the moving plate (diameter of the portion of the test piece subjected to inflation): $(55,0 \pm 0,1) \text{ mm}$
- Theoretical distance between the fixed and moving plates after clamping (equal to the thickness of the test piece before inflation): $(2,67 \pm 0,01) \text{ mm}$
- Volume of the pear-shaped rubber bulb: $(18 \pm 2) \text{ ml}$
- Linear speed of the periphery of the recording drum: $(5,5 \pm 0,1) \text{ mm/s}$

Old model

- Volume of the burette between the marks 0 and 25: $(625 \pm 10) \text{ ml}$
- Flow-time in the burette between the marks 0 and 25: $(23,0 \pm 0,5) \text{ s}$

New model

- Flow rate of air generator after adjustment of load loss created by the calibrated nozzle No. 12 C: $(96 \pm 2) \text{ l/h}$

5.2 Burette, of 160 ml capacity, graduated in 0,25 ml intervals, or a **burette**, graduated directly in percentages of moisture content of the flour from 11,6 % to 17,8 % (accuracy 0,1 %).

5.3 Balance, accurate to 0,5 g.

5.4 Timer.

5.5 Planimeter and/or planimetric scale.

6 Sampling

Sampling should have been carried out in accordance with ISO 2470.

7 Procedure

NOTE 3 Unless otherwise stated in the text, the operations specified apply to both the old and the new model.

7.1 Preliminary checks

7.1.1 Both models

Before each test, check that the temperatures of the mixer and the alveograph are $(24,0 \pm 0,2) ^\circ\text{C}$ and $(25,0 \pm 0,2) ^\circ\text{C}$ respectively. Regulate the thermostat a sufficient time before use so that the mixer and alveograph have stabilized at these temperatures. Also check these temperatures while the apparatus is in use.

If the temperature of the mixer rises above $25 ^\circ\text{C}$, follow the manufacturer's instructions for methods of cooling it.

1) This part of ISO 5530 has been drawn up on the basis of the *Alvéographe Chopin*, which is the only apparatus of this type presently available. It takes into account both the older hydraulic model (see figure 1) and the new constant-flow air generator model (see figure 2). The manufacturer also offers an integrated computer for the display and printing of the values W , p , l and p/l ; the computer also provides automatic detachment of the piece of dough (volume of air, 18 ml).

The manufacturer provides, with the apparatus, a burette graduated in percentages for the moisture content of flour, a knife/spatula, a planimetric scale and a scale for measuring the index of inflation and various sets of instructions for use.

Different types of pressure gauges may be supplied with the alveograph: the traditional type with $K = 1,1$ (see 8.2) and others with $K = 1,6$ (old model) and $K = 2,0$ (new model) for flours where the pressure p may rise to 132 mm ($K = 1,1$), 192 mm ($K = 1,6$) or 240 mm ($K = 2,0$).

7.1.2 Old model

7.1.2.1 Check regularly that the apparatus is sealed (no hydraulic leakage or air leakage).

7.1.2.2 Check that the water level in the burette is at index mark 0.

7.1.2.3 Check regularly the rate at which water rises in the burette. The time for water to flow between index marks 0 and 25 shall be $(23 \pm 0,5)$ s.

7.1.3 New model

7.1.3.1 Check regularly that the apparatus is sealed (no air leakage).

7.1.3.2 Using the calibrated nozzle No.12 C, adjust the flow rates to obtain the following pressure readings:

- adjustment of the air generator: 92 mm pressure on the chart of the water pressure gauge or the computer display;
- adjustment of the flow-rate valve: 60 mm pressure on the chart of the water pressure gauge or the computer display.

7.1.4 Both models

Use the timer to check the rotation time of the recording drum, which shall be exactly 60 s for one revolution with a current of frequency 50 Hz (or of 60 Hz for apparatus with a motor of this type) (or 55 s from stop to stop). This corresponds to a linear chart travel of 302,5 mm in 55 s.

7.2 Preliminary operations

7.2.1 Determine the moisture content of the flour by the method specified in ISO 712.

7.2.2 Check that the temperature of the flour and that of the sodium chloride solution (4.1) is (20 ± 5) °C. The apparatus shall be used in a room where the temperature is between 18 °C and 22 °C and the relative humidity is (65 ± 15) %.

7.2.3 Determine from table 1 the volume of sodium chloride solution (4.1) to be used in 7.3.1 to prepare the dough.

The values given in table 1 have been calculated to obtain constant hydration of the dough, i.e. a dough made from 50 ml of sodium chloride solution (4.1) and 100 g of flour with a moisture content of 15 %.

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Table 1 — Volume of sodium chloride solution to be added as a function of the moisture content of the flour

Moisture content of flour	Volume of sodium chloride to be added to 250 g of flour	Moisture content of flour	Volume of sodium chloride to be added to 250 g of flour	Moisture content of flour	Volume of sodium chloride to be added to 250 g of flour
%	ml	%	ml	%	ml
5,0	169,6	10,0	147,2	15,0	125,0
5,1	169,2	10,1	146,8	15,1	124,6
5,2	168,7	10,2	146,3	15,2	124,1
5,3	168,3	10,3	145,9	15,3	123,7
5,4	167,8	10,4	145,5	15,4	123,2
5,5	167,4	10,5	145,1	15,5	122,8
5,6	166,9	10,6	144,6	15,6	122,3
5,7	166,5	10,7	144,2	15,7	121,9
5,8	166,0	10,8	143,7	15,8	121,4
5,9	165,6	10,9	143,3	15,9	121,0
6,0	165,1	11,0	142,8	16,0	120,6
6,1	164,7	11,1	142,4	16,1	120,2
6,2	164,2	11,2	141,9	16,2	119,7
6,3	163,8	11,3	141,5	16,3	119,3
6,4	163,3	11,4	141,0	16,4	118,8
6,5	162,9	11,5	140,6	16,5	118,4
6,6	162,4	11,6	140,1	16,6	117,9
6,7	162,0	11,7	139,7	16,7	117,5
6,8	161,5	11,8	139,2	16,8	117,0
6,9	161,1	11,9	138,8	16,9	116,6
7,0	160,6	12,0	138,3	17,0	116,1
7,1	160,2	12,1	137,9	17,1	115,7
7,2	159,7	12,2	137,5	17,2	115,2
7,3	159,3	12,3	137,1	17,3	114,8
7,4	158,8	12,4	136,6	17,4	114,3
7,5	158,4	12,5	136,2	17,5	113,9
7,6	157,9	12,6	135,7	17,6	113,4
7,7	157,5	12,7	135,3	17,7	113,0
7,8	157,0	12,8	134,8	17,8	112,5
7,9	156,6	12,9	134,4	17,9	112,1
8,0	156,1	13,0	133,9	18,0	111,7
8,1	155,7	13,1	133,5	18,1	111,3
8,2	155,2	13,2	133,0	18,2	110,8
8,3	154,8	13,3	132,6	18,3	110,4
8,4	154,4	13,4	132,1	18,4	109,9
8,5	153,9	13,5	131,7	18,5	109,5
8,6	153,5	13,6	131,2	18,6	109,0
8,7	153,1	13,7	130,8	18,7	108,6
8,8	152,6	13,8	130,3	18,8	108,1
8,9	152,2	13,9	129,9	18,9	107,7
9,0	151,7	14,0	129,4	19,0	107,2
9,1	151,3	14,1	128,9	19,1	106,8
9,2	150,8	14,2	128,6	19,2	106,3
9,3	150,4	14,3	128,2	19,3	105,9
9,4	149,9	14,4	127,7	19,4	105,4
9,5	149,5	14,5	127,3	19,5	105,0
9,6	149,0	14,6	126,8	19,6	104,5
9,7	148,6	14,7	126,4	19,7	104,1
9,8	148,1	14,8	125,9	19,8	103,7
9,9	147,7	14,9	125,5	19,9	103,3

7.3 Mixing the dough

7.3.1 Place 250 g of flour, weighed to the nearest 0,5 g, in the mixer. Secure the lid by tightening the two screws. If the old model of alveograph is used, connect the mixing blade to the speed reducer. Start the motor and the timer. Pour the required volume of sodium chloride solution (4.1) (see table 1) through the hole in the lid. The pouring time shall be 20 s to 30 s.

Leave the dough to form for 1 min (inclusive of the pouring time for the sodium chloride solution).

The capacity of the burette, which is graduated in percentage of moisture content of the flour, supplied by the manufacturer is not sufficient for the volume of solution it is necessary to add to a flour having a moisture content of less than 11,6 %. Therefore, if the flour under test has a moisture content of less than 11,6 %, pour in the volume of solution corresponding to a moisture content of 12,0 % (i.e. 138,3 ml), then, using a 25 ml pipette graduated in 0,1 ml divisions, pour in a second volume of solution equal to the difference between the value given in table 1 and the 138,3 ml already added.

7.3.2 After this 1 min period, stop the motor and remove the lid. Using a spatula, mix in any flour and dough adhering to the lid or in the corners, so that all the dough undergoes hydration. Complete this operation in 1 min and replace the lid.

7.3.3 At the end of this second minute, re-start the motor. Allow mixing to continue for a further 6 min.

7.3.4 At the end of the eighth minute, stop mixing and proceed with the extrusion.

7.4 Preparation of test pieces

IMPORTANT — The preparation of the test pieces and the operations specified in 7.5.1 have to be completed in less than 20 min since the test has to be started 28 min after starting the mixing (see 7.5.2).

7.4.1 Reverse the direction of rotation of the mixing blade. Open the extrusion aperture by raising the slide of the shutter and place a few drops of oil (4.2) on the receiving plate, previously positioned in place. Discard the first 20 mm of dough extruded by rapidly cutting it off using the knife/spatula supplied by the manufacturer.

7.4.2 When the strip of extruded dough reaches the line indicated by the small indented notches on the plate, rapidly cut the dough, using the knife/spatula supplied by the manufacturer, against the guide.

Slide the piece of dough onto the glass plate of one of the sheeting systems, which shall have been previously oiled (first piece).

7.4.3 Repeat the operation described in 7.4.2 three times (second, third and fourth pieces), distributing the pieces of dough so that there are two pieces on each glass plate of each sheeting system, and leave the fifth piece of dough on the receiving plate. Stop the motor of the mixer.

7.4.4 Sheet the first two pieces of dough which were placed on the sheeting plates using the steel roller which has been previously oiled, running it along the rails 12 times in succession (three rapid backwards-and-forwards movements followed by three slow ones). Repeat these operations with the two other pieces of dough on the other sheeting system.

In one clean movement, cut dough test pieces from the pieces of dough using the cutter. Cut away all surplus dough. Lift the cutter containing the dough test piece, tilting it above the resting plate intended to hold it. If the dough piece sticks to the sides of the cutter, free it by tapping the table with the heel of the hand that is holding the cutter, taking care not to touch the dough with the fingers. If the dough piece sticks to the glass, lift it gently with a spatula and slide the resting plate underneath it. Immediately place each plate in the isothermal compartment (at 25 °C) of the alveograph in the order of extrusion, the first test piece being placed on top. Remove the fifth dough piece from the receiving plate and repeat the operations described above.

NOTE 4 After some experience, it is possible and preferable to carry out the operations described in 7.4.3 and 7.4.4 continuously on one sheeting system, while the strip of dough is being extruded.

7.5 Alveograph test on test pieces

7.5.1 During the period of rest, place a sheet of recording paper on the recording drum. Fill the pen with ink, trace the zero pressure line and replace the drum in its starting position.

7.5.2 Start the test 28 min after mixing began as follows.

a) First operation

Rotate knob A to position 1 (see figures 1 and 2).

Raise the upper plate B by rotating it through two revolutions.

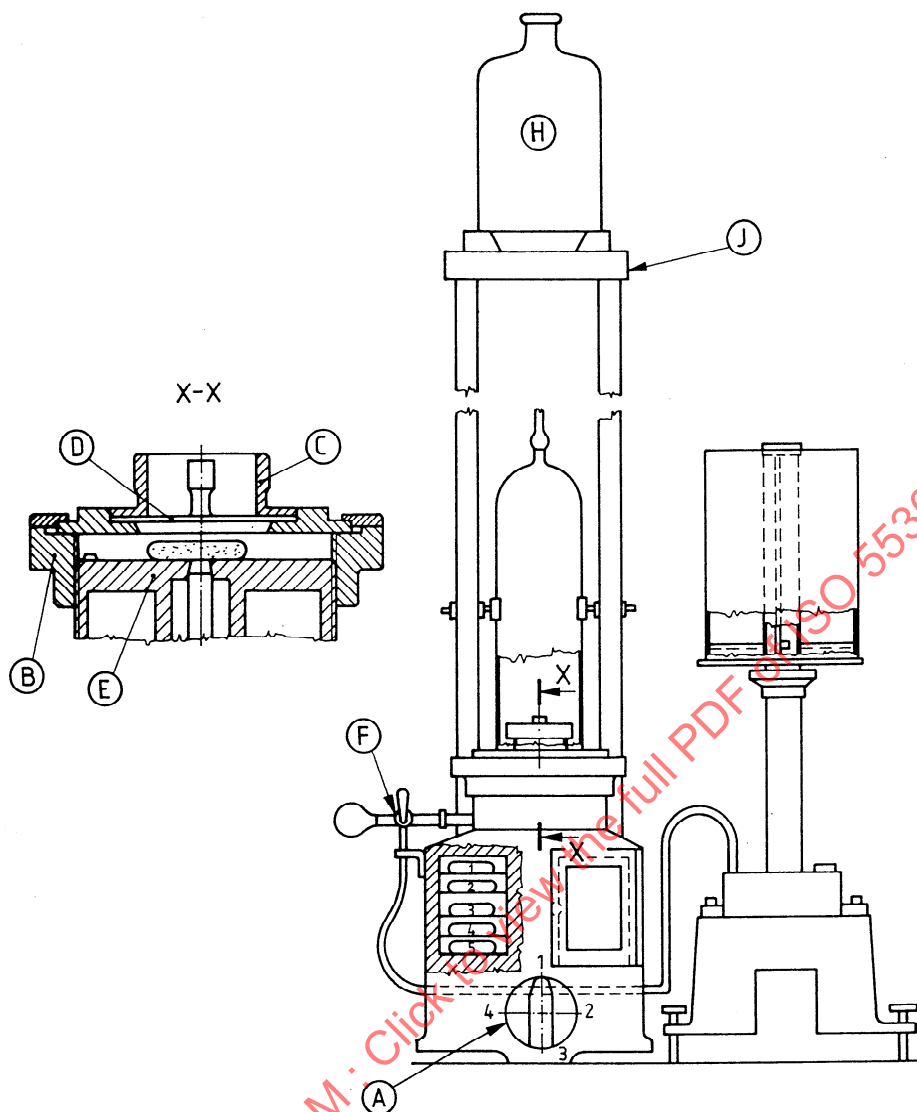


Figure 1 — Alveograph (old model)

Remove ring C and stopper D.
 Oil the plate E and the inner face of stopper D.
 Slide the dough test piece to the centre of E.
 Replace D and C.
 Flatten the test piece by slowly lowering B (two turns in 20 s).
 Remove ring C and stopper D to free the dough test piece.

b) Second operation

Rotate knob A to position 2.
 Open tap F.
 Firmly squeeze the pear-shaped rubber bulb between the thumb and index finger and maintain

the pressure. The dough test piece should then detach itself from the plate E.

Close tap F and release the bulb.

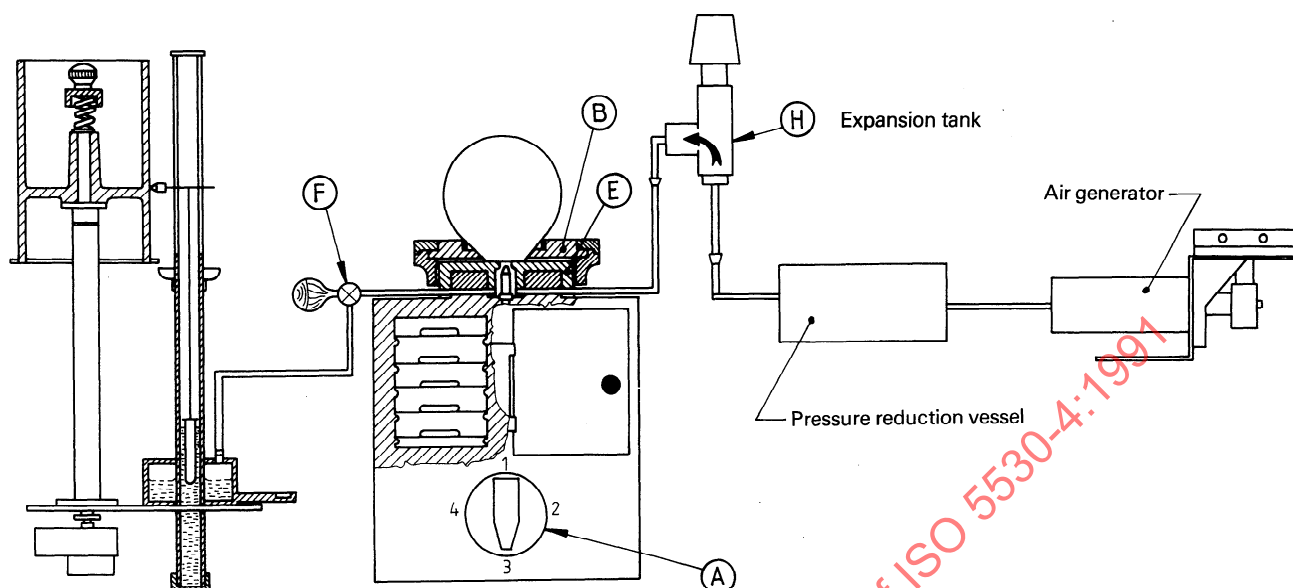
Place the water tank H on platform J (old model).

c) Third operation

Rotate knob A to position 3 so that the test piece starts to inflate and the recording drum starts to revolve.

NOTE 5 Knob A in position 3 directly controls the starting of the air generator in the new model.

Place knob A in position 4 (old model) or position 1 (new model) as soon as the dough bubble ruptures.



NOTE — Ring C and stopper D are not shown in the figure, which is a schematic representation of the apparatus during use.

Figure 2 — Alveograph (new model)

Replace water tank H on the work table (old model).

Return knob A to position 1 (old model) and the recording drum to its original position.

7.5.3 Repeat the operations described in 7.5.2 on the four remaining test pieces.

This gives five curves with the same origin.

8 Expression of results

8.1 General

The results are measured or calculated from the five curves obtained. However, if one of the curves deviates greatly from the other four, particularly as a result of premature rupture of the bubble, it shall not be taken into account in the expression of results (see, for example, figures 3 and 4).

8.2 Method by calculation

NOTE 6 For determination of the results for values of K other than 1,1, the user should refer to the manufacturer's instructions.

8.2.1 Average ordinate of maximum overpressure p

The maximum overpressure p (which is related to

the resistance of the dough to deformation) is given by the average of the maximum ordinates, measured in millimetres, multiplied by $K = 1,1$.

Express the result to the nearest whole millimetre.

8.2.2 Average abscissa at rupture, l

The average of the abscissae at the rupture points, l , is determined as follows. Measure the abscissa at rupture of each curve, in millimetres, on the zero line, starting from the origin of the curves up to the point corresponding vertically with the clear drop in pressure due to rupture of the bubble.

Express the result to the nearest whole millimetre.

8.2.3 Index of swelling, G

The index of swelling, G , is the average of the swelling indices read from the swelling scale and corresponding to the rupture abscissae. Its value is the square root of the volume of air, expressed in millilitres, necessary to inflate the bubble until it ruptures (not including the volume of air necessary to detach the dough test piece).

Annex A contains a conversion table giving G as a function of l .

Express the result to the nearest 0,5 unit (e.g. 23; 23,5; 24; ...).

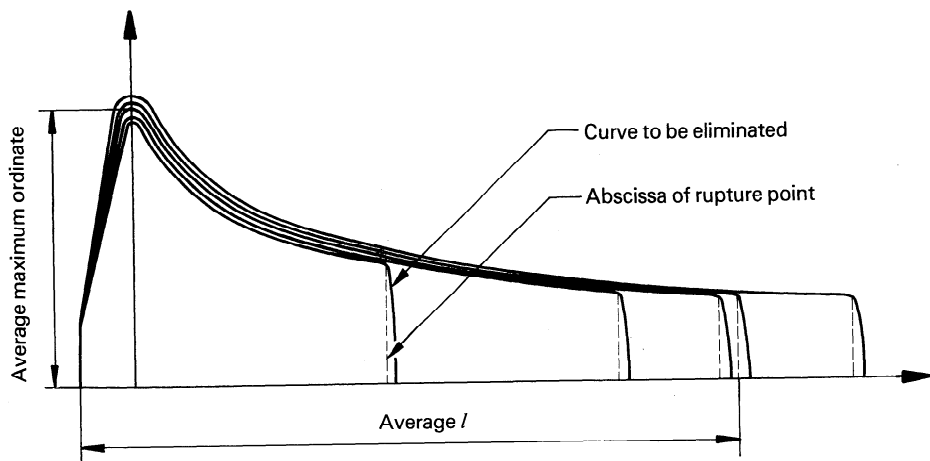


Figure 3 — Curves obtained by using a water pressure gauge

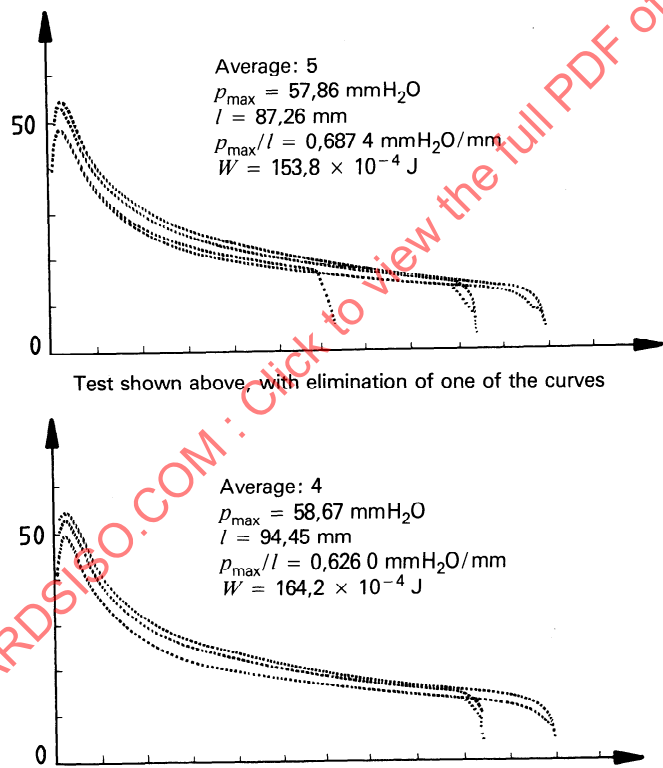


Figure 4 — Curves obtained on the printer of the integral RCV 4 computer

8.2.4 Curve configuration ratio

The curve configuration ratio is given by the formula p/l .

8.2.5 Deformation energy, W

Draw an average curve on the basis of the averages of the ordinates up to the average abscissa at rupture, l ; this is used in place of the real curves in the calculations. Measure the area on the chart, in square centimetres, using a planimetric scale or a planimeter.

Determine the deformation energy, W , in ten to the power minus four joules (10^{-4} J), of the dough necessary for inflation of the bubble until it ruptures, related to 1 g of dough, using either the reference calculation or the practical calculation given below.

8.2.5.1 Reference calculation

$$W = 1,32 \times \frac{V}{l} \times S$$

where

- V is the numerical value of the volume of air, in millilitres, equal to the square of the swelling index G to which it is necessary to add 10 ml, corresponding to the mean volume of air required to detach the dough piece;
- l is the numerical value of the average abscissa at rupture, in millimetres;
- S is the numerical value of the area under the curve, in square centimetres;
- 1,32 is a coefficient linking various factors [i.e. the curve ordinate and pressure correspondence, coefficient of pressure gauge ($K = 1,1$ in this case), average mass of the test piece, coefficient of correlation between first generation apparatus and current apparatus].

Express the result to the nearest 5 units for flours with values of $W < 200$ (e.g. scale of values: 150, 155, 160, 165, ...), or to the nearest 10 units for flours with values of $W > 200$ (e.g. scale of values: 250, 260, 280, ...).

8.2.5.2 Practical calculation

For most commonly used flours with indices G between 12 and 26 (or l between 29,2 mm and 137,3 mm), the indices being measured by means of the scale, the following simplified formula may be used

$$W = 6,54 \times S$$

where S is the numerical value of the area under the curve, in square centimetres.

The coefficient 6,54 is valid for

- a) a drum rotation time of 55 s from stop to stop;
- b) a water flow period in the burette of 23 s between marks 0 and 25 (old model);
- c) a constant air flow rate of 96 l/h (new model);
- d) a traditional-type pressure gauge with $K = 1,1$.

8.3 Method with integral RCV 4 computer (see figure 4)

The integral RCV 4 computer performs the following operations:

- calculation and display of the average values of p , l , W and p/l (with possibility of deletion of clearly abnormal tests);
- output onto a printer, at the end of the test, of the five memorized curves and the average values of p , l , W and p/l .

The values of W , p and l supplied by the integral RCV 4 computer are identical to those supplied by the use of the water pressure gauge. However, the area of the curves recorded on the printer is smaller because of the automatic detachment of the dough piece and the much more rapid response of the electronic sensor to the increase in pressure. The calculation of W can be verified using the simplified formula

$$W = 7,16 \times S$$

where S is the numerical value of the area under the curve, in square centimetres.

9 Precision

The reproducibility and repeatability of the results depend on four principal factors:

- strict adherence to the experimental procedure and to the checks detailed in the instructions for use provided with the apparatus;
- accuracy of the determination of the moisture content of the flour;
- the degree of adherence to the method specified in this part of ISO 5530;
- good mechanical state of the various parts of the apparatus.

NOTE 7 Under normal conditions, the coefficients of variation of reproducibility are of the order

8 % for W

8 % for p

5 % for G

The coefficients of variation of repeatability are substantially lower than the coefficients of variation of reproducibility.

10 Test report

The test report shall specify the method used, the model of apparatus used and the results obtained. It shall also mention all operating details not specified in this part of ISO 5530, or regarded as optional, together with details of any incidents which may have influenced the results.

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

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