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Wheat flour — Physical characteristics of doughs —

Part 3:

Determination of water absorption and rheological properties
using a valorigraph

Farines de blé tendre — Caractéristiques physiques des pâtes —

*Partie 3: Détermination de l'absorption d'eau et des caractéristiques rhéologiques au moyen
du valorigraphe*

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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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 5530-3 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*.

This part of ISO 5530 is based on Standard No. 115 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 of this part of ISO 5530 is for information only.

Wheat flour — Physical characteristics of doughs —

Part 3: Determination of water absorption and rheological properties using a valorigraph

1 Scope

This part of ISO 5530 specifies a method, using a valorigraph¹⁾, for the determination of the water absorption of flours and the mixing behaviour of doughs made from them.

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

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5530. At the time of publication, the editions indicated were 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 editions of the standards listed 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 2170 : 1980, *Cereals and pulses — Sampling of milled products*.

3 Definitions

For the purposes of this part of ISO 5530, the following definitions apply.

3.1 consistency: The resistance, expressed in arbitrary units (valorigraph units, VU), of a dough to being mixed in the valorigraph at a specified constant speed.

3.2 water absorption (of flour): The volume of water, expressed in millilitres per 100 g of flour at 14 % (*m/m*) moisture content, required to produce a dough with a maximum consistency of 500 VU, under the operating conditions specified in this part of ISO 5530.

4 Principle

Measurement and recording, by means of a valorigraph, of the consistency of a dough as it is formed from flour and water, as it is developed, and as it is broken down.

NOTE — The maximum consistency of the dough is adjusted to a fixed value by adapting the quantity of water added. The correct water addition, which is called the water absorption, is used to obtain a complete mixing curve, the various features of which are a guide to the strength of the flour.

5 Reagent

Distilled water, or water of equivalent purity.

6 Apparatus

Usual laboratory equipment and, in particular, the following.

6.1 Valorigraph (see annex A).

6.1.1 Operating characteristics

— Slow blade rotational frequency: $(64 \pm 2) \text{ min}^{-1}$

The ratio of the rotational frequencies of the mixing blades shall be $1,50 \pm 0,01$.

1) This part of ISO 5530 has been drawn up on the basis of the Labor-MIM Valorigraph.

This information is given for the convenience of users of this part of ISO 5530 and does not constitute an endorsement by ISO of this product.

— Deflection of the recorder pen, when the lever arm is loaded, at the appropriate position (75 mm from the pivot), with the calibrating weight (900 g, accessory of the instrument), with the mixing blades rotating in the empty bowl: (500 ± 10) VU

— Chart speed: (1,00 ± 0,03) cm/min

6.1.2 Burettes

— burette graduated from 25 ml to 35 ml in 0,1 ml divisions, and

— burette graduated from 32 ml to 42 ml in 0,1 ml divisions.

The time to flow from 0 ml to 35 ml or 0 ml to 42 ml respectively shall be not more than 20 s.

6.2 Balance, accurate to 0,1 g.

6.3 Plastic spatula.

6.4 Water-bath, capable of being controlled at a temperature of (31 ± 1) °C.

7 Sampling

Carry out sampling by the method specified in ISO 2170.

8 Procedure

8.1 Determination of the moisture content of the flour

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

8.2 Preparation of sample and apparatus

8.2.1 If necessary, bring the flour to a temperature of (25 ± 5) °C. Fill the flask of the valorigraph with water. Switch on the preheating and the thermostatically controlled heating. When the air temperature has reached 28 °C, switch off the preheating. Start a flow of tap water through the cooling tubes of the valorigraph. Before and during use, check the temperature of the air in the centre part of the cabinet. The air temperature shall be between 28 °C and 30 °C, with the mixer mounted, for at least 10 min before the first test portion is placed into the mixer. During use, the air temperature shall be (30 ± 0,2) °C.

8.2.2 Adjust the arm of the recorder pen so as to obtain zero deflection with the mixing blades rotating in the empty, clean bowl.

Adjust the damper so that, with the motor running, the time required for the recorder pen to go from 1000 VU to 100 VU is (1,0 ± 0,2) s.

8.2.3 Fill the burette with water. Fill the tip of the burette by letting about 1 ml of water flow into a vessel; fill the burette again.

8.3 Test portion

Weigh, to the nearest 0,1 g, the equivalent of 50 g of flour having a moisture content of 14 % (*m/m*). Let this mass, in grams, be *m*; see table 1 for *m* as a function of moisture content.

Switch off the heating. Place the flour into the mixer. Cover the mixer, and keep it covered until the end of mixing (8.4.1), except for the shortest possible time, when water has to be added, and the dough scraped down (see A.2.2). Switch on the thermostatically controlled heating.

8.4 Determination

8.4.1 Mix for 1 min or slightly longer. Start adding water from the burette into the mixer, when a whole-minute line on the recorder paper passes by the pen.

NOTE — In order to reduce the waiting time, the recorder paper may be moved forward during the mixing of the flour. Do not move it backwards.

Add a volume of water close to that expected to produce a maximum consistency (9.1) of 500 VU. When the dough forms, scrape down the sides of the bowl with the spatula (6.3), adding any adhering particles to the dough, without stopping the mixer. If the consistency is too high, add a little more water to obtain a maximum consistency (9.1) of approximately 500 VU. Stop mixing and clean the mixer.

8.4.2 After cleaning, place the parts of the mixer for at least 2 min in the water-bath (6.4) at a temperature of (31 ± 1) °C. Dry and mount them rapidly. Wait for at least 5 min, with the temperature control operating, before the next mixing.

8.4.3 Make further mixings as necessary, until two mixings are available

- in which the water addition has been completed within 25 s;
- the maximum consistencies (9.1) of which are between 480 VU and 520 VU; and
- the recording of which has been continued for at least 12 min after the end of the development time (9.2), if the degree of softening is to be reported.

Stop mixing and clean the mixer. Between successive mixings, repeat the operations specified in 8.4.2.

9 Expression of results

9.1 Water absorption

From each of the mixings with maximum consistencies between 480 VU and 520 VU, derive the corrected volume V_c , in millilitres, of water corresponding to a maximum consistency of 500 VU, by means of the formula

$$V_c = V + 0,016 (c - 500)$$

where

V is the volume, in millilitres, of water added;

c is the maximum consistency, in valorigraph units, (see figure 1) given by

$$c = \frac{c_1 + c_2}{2}$$

where

c_1 is the maximum height of the upper contour of the curve, in valorigraph units;

c_2 is the maximum height of the lower contour of the curve, in valorigraph units.

NOTE — In the relatively infrequent case where two maxima are observed, use the height of the higher maximum.

Use for the calculation the mean value of duplicate determinations of V_c , provided that the difference between them does not exceed 0,5 ml of water.

The valorigraph water absorption, expressed in millilitres per 100 g of flour at 14 % (m/m) moisture content, is equal to

$$(\bar{V}_c + m - 50) \times 2$$

where

\bar{V}_c is the numerical value of the mean value of the duplicate determinations of the corrected volume, in millilitres, of water corresponding to a maximum consistency of 500 VU

m is the numerical value of the mass, in grams, of the test portion derived from table 1.

Report the result to the nearest 0,1 ml per 100 g.

Table 1 — Mass of flour, in grams, equivalent to 50 g, at a moisture content of 14 % (m/m)

| Moisture content % (m/m) | Mass m of flour | Moisture content % (m/m) | Mass m of flour | Moisture content % (m/m) | Mass m of flour |
|--------------------------|-------------------|--------------------------|-------------------|--------------------------|-------------------|
| 9,0 | 47,3 | 12,1 | 48,9 | 15,1 | 50,6 |
| 9,1 | 47,3 | 12,2 | 49,0 | 15,2 | 50,7 |
| 9,2 | 47,4 | 12,3 | 49,0 | 15,3 | 50,8 |
| 9,3 | 47,4 | 12,4 | 49,1 | 15,4 | 50,8 |
| 9,4 | 47,5 | 12,5 | 49,1 | 15,5 | 50,9 |
| 9,5 | 47,5 | 12,6 | 49,2 | 15,6 | 50,9 |
| 9,6 | 47,6 | 12,7 | 49,3 | 15,7 | 51,0 |
| 9,7 | 47,6 | 12,8 | 49,3 | 15,8 | 51,1 |
| 9,8 | 47,7 | 12,9 | 49,4 | 15,9 | 51,1 |
| 9,9 | 47,7 | 13,0 | 49,4 | 16,0 | 51,2 |
| 10,0 | 47,8 | 13,1 | 49,5 | 16,1 | 51,3 |
| 10,1 | 47,8 | 13,2 | 49,5 | 16,2 | 51,3 |
| 10,2 | 47,9 | 13,3 | 49,6 | 16,3 | 51,4 |
| 10,3 | 47,9 | 13,4 | 49,7 | 16,4 | 51,4 |
| 10,4 | 48,0 | 13,5 | 49,7 | 16,5 | 51,5 |
| 10,5 | 48,0 | 13,6 | 49,8 | 16,6 | 51,6 |
| 10,6 | 48,1 | 13,7 | 49,8 | 16,7 | 51,6 |
| 10,7 | 48,2 | 13,8 | 49,9 | 16,8 | 51,7 |
| 10,8 | 48,2 | 13,9 | 49,9 | 16,9 | 51,7 |
| 10,9 | 48,3 | 14,0 | 50,0 | 17,0 | 51,8 |
| 11,0 | 48,3 | 14,1 | 50,1 | 17,1 | 51,9 |
| 11,1 | 48,4 | 14,2 | 50,1 | 17,2 | 51,9 |
| 11,2 | 48,4 | 14,3 | 50,2 | 17,3 | 52,0 |
| 11,3 | 48,5 | 14,4 | 50,2 | 17,4 | 52,1 |
| 11,4 | 48,5 | 14,5 | 50,3 | 17,5 | 52,1 |
| 11,5 | 48,6 | 14,6 | 50,4 | 17,6 | 52,2 |
| 11,6 | 48,6 | 14,7 | 50,4 | 17,7 | 52,2 |
| 11,7 | 48,7 | 14,8 | 50,5 | 17,8 | 52,3 |
| 11,8 | 48,8 | 14,9 | 50,5 | 17,9 | 52,4 |
| 11,9 | 48,8 | 15,0 | 50,6 | 18,0 | 52,4 |
| 12,0 | 48,9 | | | | |

NOTE — The values in this table were calculated using the formula

$$m = \frac{4\ 300}{100 - H}$$

where H is the moisture content of the sample, as a percentage by mass.

9.2 Dough development time

The dough development time is the time from the beginning of addition of water to the point on the curve immediately before the first signs of the decrease of consistency (see figure 1).

NOTE — In the relatively infrequent case where two maxima are observed, use the second maximum to measure the development time.

Take as the result the mean dough development time from the two curves to the nearest 0,5 min, provided that the difference between them does not exceed 1 min for development times of up to 4 min, or 25 % of their mean value for longer development times.

9.3 Degree of softening

The degree of softening is the difference in height between the centre of the curve at the end of the dough development time and the centre of the curve 12 min after this point (see figure 1).

Take as the result the mean degree of softening from the two curves to the nearest 5 VU, provided that the difference

between them does not exceed 20 VU for degrees of softening up to 80 VU, or 25 % of their mean value for larger values.

9.4 Repetition

If one or more of the differences between measurements on the two curves exceed the values specified in 9.1 to 9.3 inclusive, make another two mixings that meet the requirements of 8.4.3.

9.5 Precision

The precision data were determined from an experiment conducted in 1985 involving 11 laboratories, 4 levels, and determinations in duplicate as specified in the method (see table 2).

10 Test report

The test report shall specify the method 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.

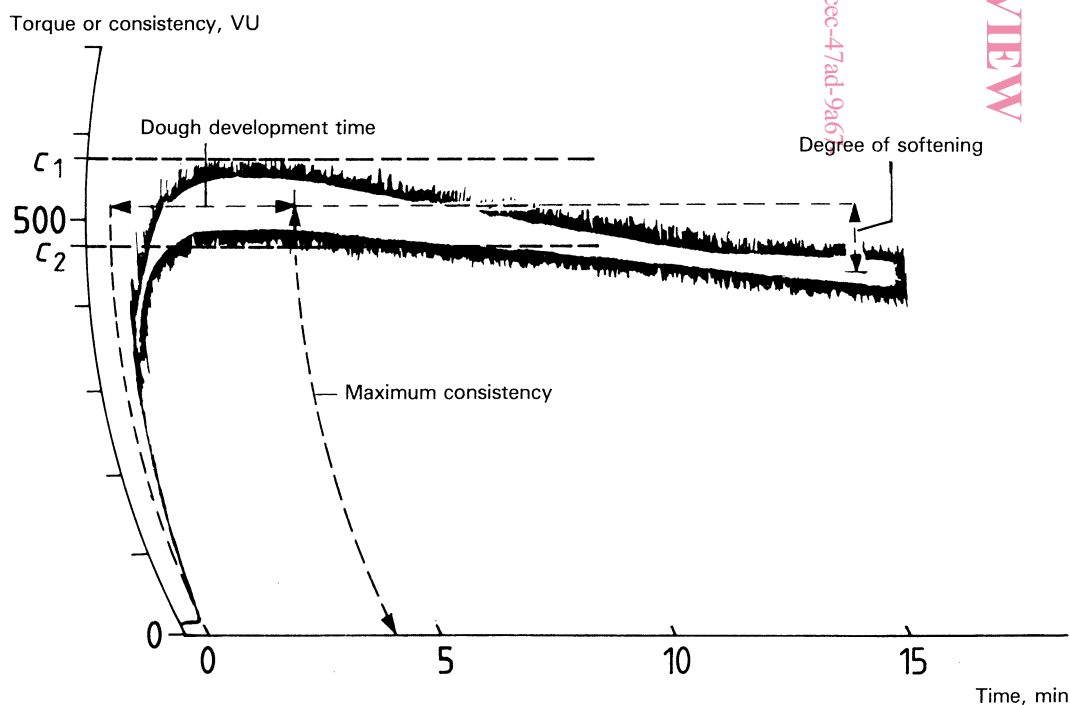


Figure 1 — Representative valorigram showing the commonly measured indices

Table 2 — Precision of valorigraph measurements

| | Repeatability | Reproducibility |
|------------------------|---|-------------------------|
| Water absorption | 0,7 ml per 100 g of flour | 5 ml per 100 g of flour |
| Dough development time | 1,0 min | Results inconclusive |
| Degree of softening | up to 80 VU : 20 VU above 80 VU : 25 % of mean value | 60 % of mean value |

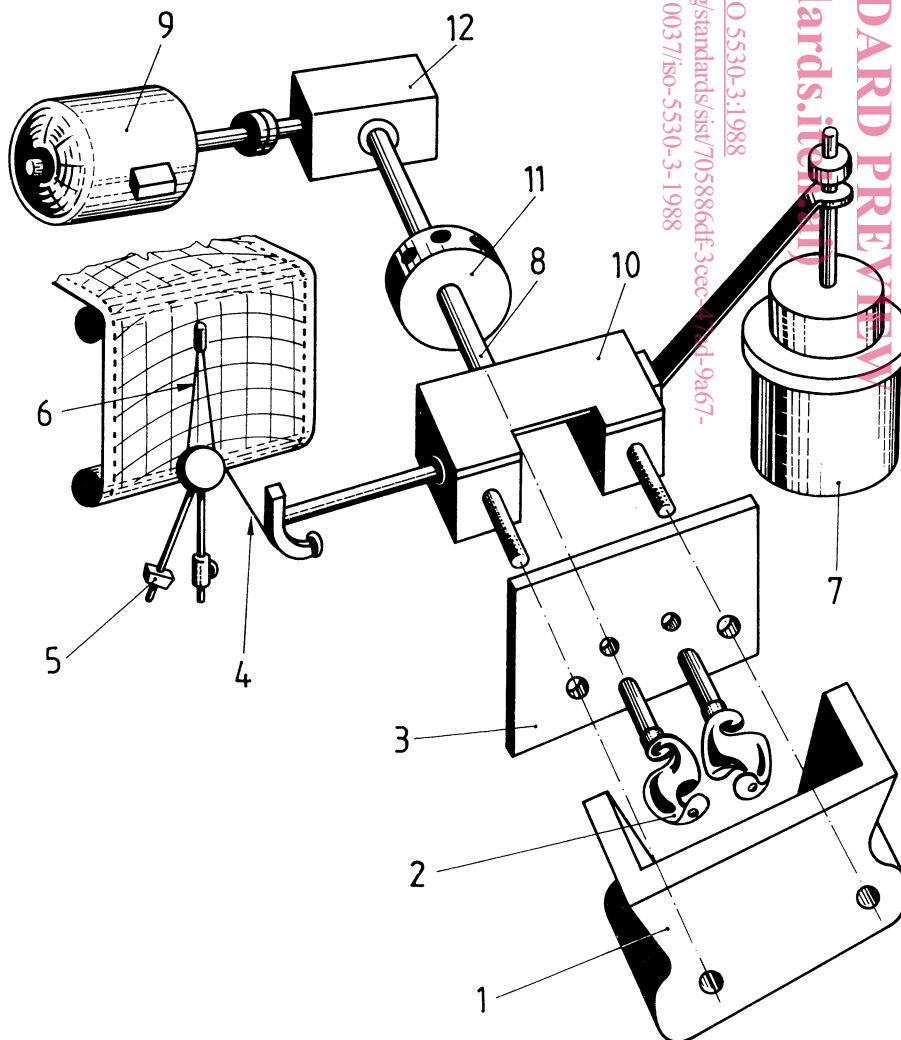
Annex A (informative)

Description of the valorigraph

A.1 General description

The valorigraph unit itself consists of a mixer, a means for recording dough consistency in the form of valorigrams, and a burette (A.2). Its components are illustrated diagrammatically in figure A.1.

The valorigraph unit is placed in a metal cabinet. The centre part of the cabinet contains the mixer, the burette, and a flask with the stock of water (clause 5). They are accessible by a plexiglass door in the front of the cabinet. The temperature of the air in the centre part of the cabinet is thermostatically controlled (A.3).



- | | |
|---|-----------------------|
| 1 Mixer, except back plate | 6 Arm of recorder pen |
| 2 Mixing blade | 7 Dash-pot damper |
| 3 Back plate of mixer | 8 Driving shaft |
| 4 Bronze band between arm of mixer and lever system of recorder | 9 Motor |
| 5 Lever system of recorder | 10 Reduction gear |
| | 11 Clutch |
| | 12 Worm gear |

Figure A.1 — Diagram of valorigraph unit

A.2 Valorigraph unit

A.2.1 The valorigraph unit consists of

- a) a mixer, the parts of which can be detached (A.2.2);
- b) an electrical motor, with reduction gear, driving the mixer (A.2.3);
- c) a lever system, acting as a dynamometer to measure the torque on the mixer (A.2.3);
- d) a dash-pot to damp the movements of the dynamometer (A.2.3);
- e) a recorder, the pen of which is actuated by the movements of the dynamometer (A.2.4);
- f) a burette to measure the volume of water added to the flour.

A.2.2 The mixer is two-bladed and is designed to mix doughs from 50 g of flour. It is made of four parts:

- a) the back plate, through which shafts project to attach the mixing blades;
- b) and c) two mixing blades;
- d) the remainder of the mixer, i.e. two sides, front and bottom in one piece.

Parts a) and d) are held together by means of two bolts and wing nuts. Parts b) to d) inclusive can be removed from the cabinet for cleaning.

The slower mixing blade is driven directly by the shaft from the gear; it rotates at a frequency of 64 min^{-1} . The faster mixing blade is geared, by cog-wheels, to rotate at a frequency that is 1,5 times that of the slower blade.

The mixer can be closed by a flat plastic plate, which is laid on top of the mixer. It is removed to add water and to scrape down the dough.

A.2.3 The mixer together with the reduction gear can pivot on the driving shaft. The resistance of the dough to being mixed causes a torque on the mixer which, if not balanced, would cause rotation of the mixer.

The mixer carries on its left-hand side an arm the end of which is connected by the lever system to the recorder pen. This causes a counter-torque on the mixer, which is proportional to the deflection of the recorder pen.

As a result, the deflection of the recorder pen is, if the two torques balance one another, proportional to the torque on the mixer, i.e. to the resistance of the dough to being mixed.

Movements of the mixer, lever system, and recorder pen are damped by a piston immersed in silicone oil; the piston is connected to the end of the arm on the right-hand side of the mixer. The extent of damping can be adjusted; more damping results in a narrower curve.

A.2.4 The paper for the recorder is supplied in the form of a roll. It is moved by an electric clock-type motor at a rate of 1,00 cm/min. Along its length it bears a printed scale in minutes. Across its width it bears a circular scale (radius 200 mm) with arbitrary units, running from 0 to 1000 valorigraph units.

A.2.5 The burette is operated by push-buttons, one for filling it, and another for delivering water to the mixer.

The choice between the two burettes is made on the basis of the expected water absorption.

A.3 Temperature control

For temperature control, the air in the centre part of the cabinet circulates through air ducts containing

- a) an electric preheating element with high power, controlled by a push-button;
- b) an electric heating element with low power, controlled by the thermoregulator c);
- c) a thermoregulator in the centre part of the cabinet, set at $30 \text{ }^\circ\text{C}$, that controls the heating element b);
- d) a fan, driven by an electric motor;
- e) metal tubing to cool the instrument by a flow of tap water.

The temperature of the oil in the dash-pot damper is maintained at $40 \text{ }^\circ\text{C}$ by an electric heating element and a thermoregulator that controls this heating element.

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