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Results of an international ring test for the determination of water absorption capacity and physical properties of wheat dough using the Haubelt Flourgraph E 6 (ICC standard no. 179)

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RESEARCH ARTICLE

Abstract

The rheological assessment of the physical characteristics of wheat dough plays a crucial role in food industries. The introduction of ICC standard no. 179 Haubelt Flourgraph E 6 emphasises the importance of continuously developing methods to assess the physical properties of wheat dough. The aim of this multinational collaborative study is to measure the performance of this equipment for the validation of the draft standard method. The ring test for Flourgraph E 6 was organised and performed under the responsibility of Haubelt Laborgeräte GmbH. Ten laboratories participated in the ring, performing the test method on 5 flours of different rheological properties in addition to one sample investigated in duplicate (blind), results were collected by Haubelt Laborgeräte GmbH and the data forwarded to ICC's technical director for statistical evaluation of accuracy (trueness and precision) of measurement for the water absorption, dough development time and dough stability according to the requirements of ISO 5725 part 1, 2 and 6. The relationship between standard deviation of repeatability, reproducibility (s_r, s_R) and the mean values cannot be described sufficiently by a linear regression line. However the calculation of the mean repeatability and reproducibility as percentage of the mean value, respectively, helped to summarise and clarify the ring test results in a simple and brief way. The results of all three parameters (water absorption, dough development time, dough stability) had the standard deviation of reproducibility higher than the standard deviation of repeatability and in 10% of all cases $s_R = s_r$.

Keywords: rheology, wheat flour, dough mixing properties, dough quality, Haubelt Flourgraph E 6

1. Introduction

The International Association of Cereal Science and Technology (ICC), is an independent, globally recognised organisation with its original objective to develop internationally approved and accepted standard testing procedures for cereals and their by-products in the purpose of improving among other, food quality. Since wheat flour is an important raw material in the manufacture of several wheat-based foodstuffs such as bread, breakfast cereals, infant foods, snacks, and pasta, this is why the continuous introduction of innovative techniques and tools for the assessment of the rheological properties of dough are important for the development of the entire cereal's related sector.

Wheat dough is mainly formed of wheat flour, water, yeast, salt, and other ingredients. It is a transitory physical stage in the industrial process of transforming wheat, through flour, into bread. The rheological properties of dough which are conferred to it by its protein and gluten content (Schofield and Booth, 1983) especially during the kneading phase are important to wheat related industries because the distribution and swelling of flour particles during mixing favour the formation of a protein matrix that holds starch (Campos *et al.*, 1997) and affect the quality of the finished loaf of bread (Bloksma and Bushuk, 1988). Therefore, it is essential to gain a deep knowledge of dough behaviour and to ensure the accuracy of the measured rheological data.

Objective measurements of these properties were first developed by cereal scientists in the 1930's and one of the first special instruments that were designed for physical testing of wheat flour dough was the so-called Brabender® Farinograph® (Bloksma and Bushuk, 1988). The Brabender® Farinograph® which is the current reference in this field has a large mixing bowl (300 g flour), it measures and records the resistance of dough to mixing, it is used to evaluate absorption of flours and to determine stability and other characteristics of dough during mixing. The Haubelt Flourgraph E 6 (Haubelt Laborgeräte GmbH, Berlin, Germany) applies the same principles.

Shear flow is the predominant flow experienced during dough mixing and was investigated with the Haubelt Flourgraph E 6 which imitates the deformation experienced by the dough during kneading, and provides measurements of the physical properties of the dough during this crucial processing stage (water incorporation).

When using the Flourgraph E 6 mixer, the flour is first mixed with water using a combination of shear and extensional deformations that forms a dough that can be referred to as 'developed dough' (Campos *et al.*, 1996, 1997; Schluentz *et al.*, 2000) and afterwards is subjected to a stable mechanical stress by the mixer blades until it loses the viscoelastic properties that are strongly related to the gluten content and it's molecular structure. (Faubion and Hoseney, 1989; Janssen *et al.*, 1996).

The Flourgraph E 6 provides the measurement of the hydration capacity of the flour at a fixed arbitrary consistency (500 Haubelt units; HE) and a fixed temperature (30 °C) as well as specific dough rheological characteristics such as the dough development time and dough stability.

The determination of these parameters with acceptable repeatability (r) and reproducibility (R) is important especially for the 'water absorption' capacity, because the dynamic rheological properties of wheat flour dough are strongly influenced by water content.

The elastic and viscous properties decrease with increasing water content (Hibberd and Wallace, 1966; Letang *et al.*, 1999; Masi *et al.*, 1998; Navickis *et al.*, 1982). This method is applicable to bread wheat *(Triticum aestivum* L.*)* flour.

The aim of this study, performed by 10 laboratories on 6 samples of wheat flour under the responsibility of Haubelt Laborgeräte GmbH and the supervision of ICC's technical director, is to establish the accuracy of the results of the rheological parameters gathered through Haubelt Flourgraph E 6: water absorption capacity, dough development time and dough stability.

Statistical results have been achieved according to ISO 5725-1, 5725-2 and 5725-6 (ISO, 1994), respectively.

2. Materials and methods

Materials

The wheat flour material consisted of six samples with water absorption capacities varying from what is arbitrary agreed upon as 'low' to 'high' with intermediate values, to represent as much as possible the whole range encountered in practical application to bread wheat flour. Six flour samples in total have been investigated each individual sample has been measured in duplicate. Flours no. 3 and 6 are the same (duplicate). Of these 5 samples, 2 flours are of type 550 from Germany and 3 flours from a Haubelt partner in Turkey.

Ten laboratories participated in the ring test, 5 thereof being from Turkey, 2 from Germany and 1 from Austria, Bulgaria and Cyprus, respectively (Jbeily *et al.*, 2014). Thus the requirements of ISO, ICC and IUPAC for an international ring test are fulfilled.

Dough mixing and determination of viscoelastic properties

Sampling, sample preparation and chemical properties of flour were determined according to:

- ICC standard no. 130 (ICC, 1980);
- ICC standard no. 110/1 (ICC, 1976);
- ISO 3696 (ISO, 1987).

The dough was made with the Haubelt Flourgraph E 6 (Figure 1) according to ICC draft standard method no. 179 (ICC, 2012). The Flourgraph E 6 makes programmable electronic measurements using Haubelt Flourgraph E 6 PC software (Haubelt Laborgeräte GmbH) and the titration curves are easy to follow on a touch-screen monitor (Figure 2) located directly near the mixer device that has a capacity of 100 g of flour and a stable temperature of 30 °C. Kneading arms rotation is 63 rpm.

The following parameters were determined in a Haubelt Flourgraph E 6:

- Water absorption-percentage of water required to yield dough consistency of 500 HE.

Figure 1. Haubelt Flourgraph E 6.

- Development time of the dough is defined as the time between the start of the measurement (addition of water) and the point of the torque curve (middle curve) just before weakening begins (Figure 3).
- Stability is defined by the time difference between the first and second intersecting point of the upper trace of the torque curve with the line of consistency (i.e. 500 HE). When the consistency (middle of the diagram in the maximum) does not meet 500 HE, the actual consistency is used for determining the intersection points instead of the 500 HE consistency line.

Statistical analysis

Statistical results have been achieved according to ISO 5725-1, 5725-2 and 5725-6, respectively (ISO, 1994) and the statistical analysis of the data was prepared using SPSS predictive analytics software for windows version 14.0 (SPSS Inc., Chicago, IL, USA) in order to determine the repeatability, reproducibility and standard deviations within and between the laboratories.

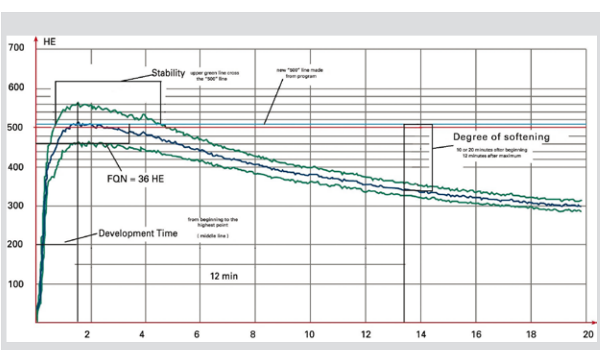

Figure 3. Flourgraph E 6 result graph (HE = Haubelt units).

Figure 2. Haubelt Laborgeräte program.

3. Results and discussions

Flours 1 to 6 selected for this study have different mixing parameters, regardless of their chemical composition, they were analysed in duplicate by the 10 participating laboratories, and the results for the three main parameters (water absorption, development time, stability) were statistically evaluated.

Dough water absorption

The water absorption or hydration capacity is the amount of water absorbed to achieve standard consistency of 500 BU (or in this case HE) (Lazaridou *et al.*, 2007). Water plays a vital role in hydrating the protein fibrils and starting the interactions between the proteins cross-links with the disulphide bonds, there exist an optimum water level in developing a cohesive and viscoelastic dough with optimum gluten strength (Abang Zaidel *et al.*, 2008; Faubion and Hoseney, 1989). The studied equipment expresses the consistency as HE. The mean value for the water absorption for the six samples varied from 53.63 to 69.33 ml/100 g flour after elimination of one outlier (laboratory 3 for sample 1) when determined with the extreme studentized deviate test (Cochran and Grubbs test). The raw data for water absorption and the statistical data are detailed in Table 1 and 2 and the standard deviations within each of the participating laboratories as well as between them are represented in Figure 4.

Dough development time

The development time is the necessary time needed for the mixture to reach the maximum consistency (Bordei, 2007). It is dependent on the formation time of gluten structure in the dough. Gluten is obtained naturally during dough mixing from the interactions between polymers

Table 1. Raw data of dough water absorption (ml/100 g flour) determined with the Flourgraph E 6.

Laboratory number	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5		Sample 6[a]	
Measurement	1	2	1	2	1	2	1	2	1	2	1	2
1	60.6	60.6	61.0	60.9	58.5	58.9	68.9	68.9	52.9	53.2	59.1	58.9
2	61.2	61.4	61.5	61.2	59.6	59.7	69.2	69.6	53.6	53.9	59.7	59.6
3	59.2	61.3	61.3	61.2	59.3	59.4	69.5	69.6	53.6	53.7	59.6	59.6
4	61.8	61.6	61.4	61.1	59.9	60.0	69.7	69.8	53.5	53.7	59.7	59.7
5	61.6	61.8	61.2	61.5	59.5	59.7	69.6	69.7	53.2	53.6	59.4	59.5
6	61.0	61.0	61.2	61.1	59.7	59.6	69.8	69.6	53.4	53.6	59.9	59.7
7	61.6	61.5	61.4	61.5	59.5	59.5	69.2	69.2	54.0	54.1	59.4	59.5
8	61.4	61.4	61.4	61.0	59.4	59.1	69.2	69.1	53.8	53.6	58.9	59.1
9	61.8	61.6	61.4	61.3	59.2	59.1	69.1	69.0	53.7	53.6	59.2	59.3
10	61.6	61.6	61.0	61.3	59.2	59.3	68.9	69.0	53.9	53.9	59.4	59.3
Mean value	61.39		61.25		59.41		69.33		53.63		59.43	

[a] Sample 6 is identical to sample 3, i.e. is a blind sample.

Table 2. Precision data for the results of water absorption (ml/100 g flour) determined with Flourgraph E 6.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6[a]
L	9	10	10	10	10	10
Mean	61.39	61.25	59.41	69.33	53.63	59.43
s_r	0.10	0.17	0.13	0.11	0.16	0.09
RSD_r	0.2%	0.3%	0.2%	0.2%	0.3%	0.2%
r	0.27	0.47	0.37	0.32	0.44	0.26
s_R	0.28	0.18	0.27	0.25	0.24	0.21
RSD_R	0.5%	0.3%	0.5%	0.4%	0.4%	0.4%
R	0.78	0.50	0.76	0.69	0.66	0.59

[a] Sample 6 is identical to sample 3, i.e. is a blind sample.
L = number of laboratories considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within laboratories; r = repeatability (r = $2.8s_r$); s_R = standard deviation between the different laboratories; RSD_R = relative SD between laboratories; R = reproducibility (R = $2.8s_R$).

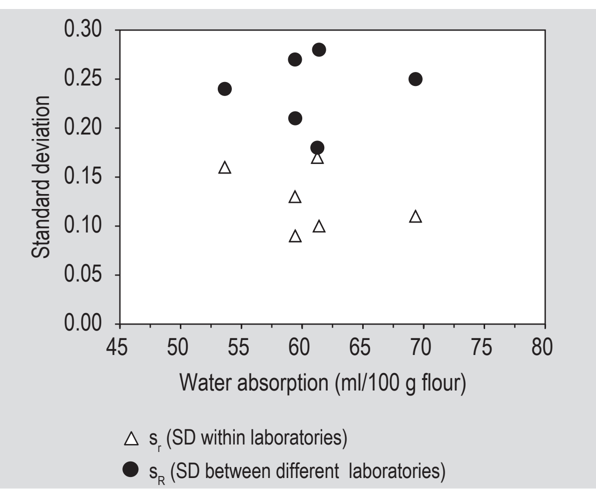

Figure 4. Standard deviation of repeatability and reproducibility for water absorption.

cross-links forming disulphide bonds which contribute to an increased dough strength. However, when dough is mixed longer pass its optimum development, the cross-links will break, thus the importance of quantifying the optimum mixing time.

The six flour samples also demonstrated various aggregation capacities and mixing properties. The dough development time varied from short mixing time (1.7 min) to long mixing time (13.2 min), the raw data for dough development time (min) and the statistical data are detailed in Table 3 and 4, and the standard deviations within each of the participating laboratories as well as between them are represented in Figure 5.

Table 4. Precision data for the results of dough development time (min) determined with the Flourgraph E 6.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6[a]
L	10	10	10	10	10	10
Mean	13.2	4.6	2.2	4.8	1.7	2.2
s_r	0.9	2.2	0.1	0.4	0.3	0.2
RSD_r	6.6%	48.8%	6.7%	7.4%	16.6%	7.9%
r	2.5	6.2	0.4	1.0	0.8	0.5
s_R	0.9	3.2	0.2	0.4	0.4	0.2
RSD_R	6.6%	69%	6.9%	8.9%	21.3%	8.5%
R	2.5	8.8	0.4	1.2	1.0	0.5

[a] Sample 6 is identical to sample 3, i.e. is a blind sample.
L = number of laboratories considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within laboratories; r = repeatability ($r = 2.8s_r$); s_R = standard deviation between the different laboratories; RSD_R = relative SD between laboratories; R = reproducibility ($R = 2.8s_R$).

Table 3. Raw data of dough development time (min) determined with the Flourgraph E 6.

Laboratory number	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5		Sample 6[a]	
Measurement	1	2	1	2	1	2	1	2	1	2	1	2
2	12.9	13.2	1.7	1.8	2.1	2.0	4.7	4.9	2.7	2.2	1.9	2.0
3	14.5	12.1	1.4	1.5	1.9	2.2	4.2	4.9	2.5	2.4	2.4	2.0
4	13.0	12.7	1.8	1.6	2.3	2.3	4.6	5.4	1.7	1.6	2.4	2.2
5	13.2	13.2	1.5	7.8	2.4	2.3	4.8	4.7	2.1	1.4	2.1	2.3
6	14.8	12.3	1.6	1.8	2.1	2.4	5.4	5.8	1.3	2.2	2.1	2.3
7	12.9	13.0	8.8	9.3	2.0	2.2	4.7	4.2	1.6	1.4	2.4	2.1
8	13.9	14.1	1.4	9.1	2.2	2.1	4.6	4.3	1.5	1.6	2.2	2.0
9	12.8	14.2	9.0	9.1	2.3	2.2	4.6	3.7	1.3	1.5	2.1	2.0
10	13.9	13.5	1.5	1.2	2.3	2.0	4.6	4.6	1.5	1.5	2.1	2.0
Mean value	13.2		4.6		2.2		4.8		1.7		2.2	

[a] Sample 6 is identical to sample 3, i.e. is a blind sample.

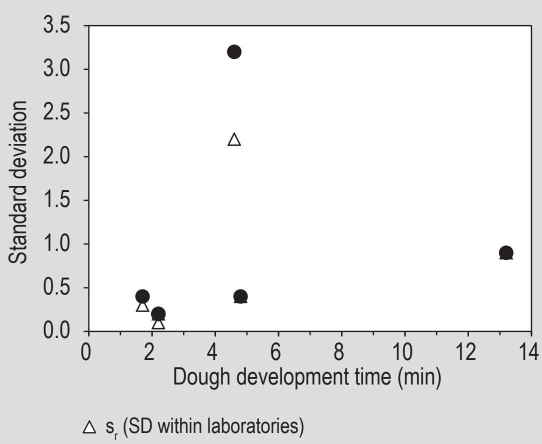

Figure 5. Standard deviation of repeatability and reproducibility for dough development time.

Dough stability

The stability which is also known as 'mixing tolerance' is the time interval between the moment when the top edge of the curve crosses the line corresponding to the maximum consistency of the dough and the moment when it exceeds it (Bordei, 2007). The mixing tolerance of dough is closely related to the gluten quantity and quality, a strong gluten network leads to increased mixing tolerance.

The flour samples showed different mixing tolerances varying from 'low' (3.1 min) to 'high' (12.5 min) after elimination of two outliers (laboratory 2 and 6 for sample 1) when determined with the 'extreme studentized deviate' test (Cochran and Grubbs test).

The raw data for dough stability (min) and the statistical data are detailed Table 5 and 6, and the standard deviations within each of the participating laboratories as well as between them are represented in Figure 6.

4. Discussion

From the results of all three parameters (water absorption, dough development time, dough stability) shown in Figure 4, 5 and 6 the following can be deduced:

Generally the standard deviation between different laboratories is higher than within the individual laboratories (in 10% of all cases $s_R = s_r$).

The relationship between SD (s_r, s_R) and the mean values cannot be described sufficiently by a linear regression line.

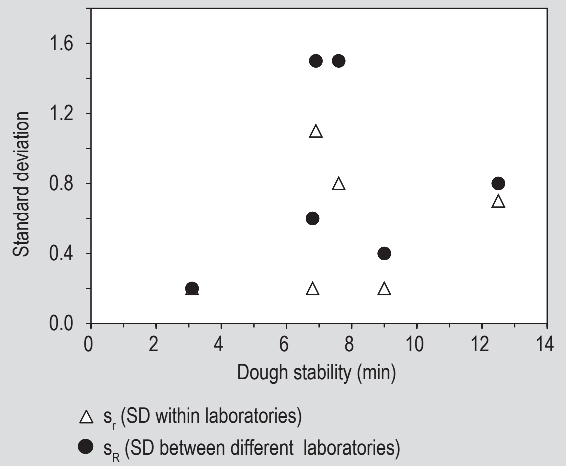

Figure 6. Standard deviation of repeatability and reproducibility for dough stability.

Table 5. Raw data of dough stability (min) results determined with the Flourgraph E 6.

Laboratory number	Sample 1		Sample 2		Sample 3		Sample 4		Sample 5		Sample 6[a]	
Measurement	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**	**1**	**2**
2	17.0	9.0	12.0	12.0	6.0	5.0	6.8	6.6	3.1	3.0	8.5	8.5
3	10.0	9.0	12.0	11.0	6.4	8.4	6.4	6.3	3.3	3.2	9.7	7.9
4	8.5	8.5	13.0	13.0	7.4	8.0	6.8	6.5	3.2	3.2	9.5	7.9
5	9.5	9.6	11.5	12.0	9.3	8.6	6.2	6.0	3.3	3.5	8.8	9.5
6	0	0	13.0	12.5	7.5	8.4	5.9	6.7	3.5	3.5	9.2	8.8
7	8.5	8.5	12.0	12.5	5.7	4.5	6.9	7.3	3.0	2.9	7.5	7.3
8	8.5	9.0	12.5	13.0	6.9	7.5	7.9	7.6	3.0	2.8	7.3	5.5
9	9.0	9.0	12.5	12.5	5.8	3.8	7.8	7.8	2.7	3.0	6.2	4.5
10	9.0	9.0	15.0	12.5	4.5	5.9	7.4	7.3	2.7	2.8	3.8	3.6
Mean value	9.0		12.5		6.9		6.8		3.1		7.6	

[a] Sample 6 is identical to sample 3, i.e. is a blind sample.

Table 6. Precision data for the results of dough stability (min) determined with the Flourgraph E 6.

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6[a]
L	8	10	10	10	10	10
Mean	9.0	12.5	6.9	6.8	3.1	7.6
s_r	0.2	0.7	1.1	0.3	0.2	0.8
RSD_r	2.1%	5.8%	16.3%	4.1%	4.8%	11%
r	0.5	2.0	3.1	0.8	0.4	2.3
s_R	0.4	0.8	1.5	0.6	0.2	1.5
RSD_R	4.2%	6.2%	22.1%	8.4%	7.6%	19.9%
R	1.1	2.2	4.3	1.6	0.7	4.2

[a] Sample 6 is identical to sample 3, i.e. is a blind sample.

L = number of laboratories considered (according to Cochran and Grubbs test); s_r = standard deviation within the laboratory; RSD_r = relative standard deviation within laboratories; r = repeatability ($r = 2.8s_r$); s_R = standard deviation between the different laboratories; RSD_R = relative SD between laboratories; R = reproducibility ($R = 2.8s_R$).

(However, there are indications for dough development time and dough stability, respectively, that for a larger number of samples there might exist a linear dependency). Calculation of the mean repeatability and reproducibility as percentage of the mean value, respectively, helped to summarise the ring test results in a simple and brief way. (As there are significant differences for dough development time between the laboratories for sample 2 this sample has been excluded from calculation of the value for dough development time in Table 7).

However two minor peculiarities were raised in the process of accomplishing this method validation study and particularly during the data collection and statistical evaluation; these points are detailed as follows:

1. In order to address the point raised in connection with the standard deviation of repeatability and reproducibility being equal in 10% of the cases in this study, a revision or even re-validation might be required when a larger diversity of users may give a more realistic picture than the currently very limited prevalence of application of the method. For this reason a note was added at the end of ICC standard no. 179 (ICC, 2012) citing: 'In order to support the reliability of this ICC standard, a usual revision of the standard and its underlying data and figures will be performed after an adequate minimum time of the standard being in practical use (2 years), according to ICC rules and guidelines for standardization'. This is mainly due to the peculiarity of reproducibility figures, which need to be confirmed in another ring trial when the method is more widespread and thus more realistic figures can be obtained.
2. With sample 2 there were difficulties with the measurement of the dough development time as dough consistency increased again for a short time after the primary increase and subsequent decreases (Figure 3), and the Instruction manual did not give information on how to deal with this occurrence. For this same sample two values are given by participants with extreme differences in dough development time (Table 3).

This double peak is not uncommon in this kind of rheological testing, it has been addressed by similar ICC methods (ICC standard no. 115/1; ICC, 1992) where guidelines are given on how to handle such cases, this recommendation should be included in the user's manual of the Flourgraph E 6.

5. Conclusions

The Haubelt Flourgraph E 6 repeatability and reproducibility values for the three parameters (Table 7.) are quite satisfactory when compared to similar other instruments that works on empirical methods using the same 'shear action' principle. These numbers are comparable to those of the Brabender® Farinograph®, which is the current reference. Table 8 compares the repeatability and reproducibility figures obtained in this ring test, with those specified by ICC standard no. 115/1 (ICC, 1992) concerning the Brabender® Farinograph®.

Moreover, these similarities in precision have been also asserted by a recent comparative research study, in which the results obtained also showed that the values for the Haubelt Flourgraph E 6 are comparable with Brabender® Farinograph® values and units. Hydration capacity values, calculated for a standard consistency of 500 HE, obtained on these two devices are in close correlation; R^2=0.8441 for white flour and R^2=0.9995 for brown flour (Iancu *et al.*, 2010).

Table 7. Precision of the investigations by means of the Haubelt Flourgraph E 6.

	Repeatability (% of mean values)	Reproducibility (% of mean values)
Water absorption (ml/100 g flour)	0.58	1.09
Dough development time (min)	21	23
Dough stability (min)	20	30

Table 8. Comparative data for repeatability and reproducibility of Flourgraph E 6 with the Farinograph.

	Repeatability (% of mean values)		Reproducibility (% of mean values)	
	Flourgraph E 6	Farinograph	Flourgraph E 6	Farinograph
Water absorption (ml/100 g flour)	0.58	0.52	1.09	1.60
Development time (min)	21	16	30	48

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