

A New AACC International Approved Method to Measure Rheological Properties of a Dough Sample

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Flour millers and their customers can obtain key information on the performance characteristics of wheat flour when the rheological properties of a dough sample are measured. The Mixolab is a new device that enables users to measure the consistency of dough over time with a gradual increase in the applied temperature. The Mixolab measures both protein and starch characteristics of the flour and can also provide information about the dough development time,

protein breakdown, starch gelatinization, enzyme activity, and the gel strength.

This type of approach was first used by Van Stock, a miller from Rotterdam, who used an alcohol lamp to heat the batter. The approach was taken up by Buys using a machine called a Pétrinex. He replaced the batter used by Van Stock with dough made from flour and water. The Pétrinex was widely used in northern France and Belgium. Several authors such as A. Duranel (2) and G. Bussiere reported good repeatability with the Pétrinex, concluding that it could be used to conduct tests under repeatability conditions (reproducibility was not satisfactory). The current version of the apparatus, the Mixolab (Fig. 1), was introduced in 2004 by CHOPIN Technologies (Villeneuve la Garenne, France).

The Mixolab

The mixer has a 50-g flour capacity and is fitted with two blades (Figs. 2 and 3). The blades, which turn in opposite directions, are specially designed to ensure efficient dough development. The resistant torque exerted on the blades during dough mixing is measured by a sensor located on the axis of one of the blades. Heating resistors provide temperature control in the bowl, and water is added through an integrated water circuit. The test is completely managed by the computer and software, which also allows for calibration and data storage. Before testing begins, the instrument performs a self-calibration to ensure that the torque- and temperature-measuring systems are operating within the specified guidelines. Water is automatically fed via an integrated water circuit based on the instructions set by the user.

In order to provide improved comparisons between samples, the Mixolab works on a constant dough mass basis. This mass is 75 g by default (corresponding to about 45 g of flour). The instrument calculates the quantity of flour to be weighed depending on the moisture content of the sample and the water absorption

at which the test will be conducted. The quantity of water is automatically dispensed by the instrument.

Two years after the introduction of the Mixolab, the Physical Testing Methods Technical Committee of AACC International undertook the challenge of investigating the precision of the instrument and its suitability as an AACC Intl. Approved Method of Analysis. The collaborative study included 13 laboratories from various countries (all experienced users) (Table I) who performed the method on samples of flour and ground wheat.

The results of this study are presented here and provide the background and results for the approval of the new AACC Intl. Method 54-60.01.

Collaborative Study

For the study, each laboratory received 10 flour and 12 wheat samples consisting of both soft and hard wheats, representing a wide range of quality. Each laboratory was instructed to conduct duplicate analyses on each flour and wheat sample. In addition, two of the wheat samples were analyzed as blind duplicates (B03 and B07; B09 and B12). For the collaborative study, the “Chopin+” protocol was used.

Results of the Collaborative Study

Data analysis was performed in accordance with procedures as described in ISO Method 5725-2 (3). An important aspect of the Mixolab method that was also evaluated was whether the same method could be used on both ground wheat and flour samples with equivalent levels of precision and accuracy.

Milling is a critical factor that can affect Mixolab results, since water absorption depends on the particle size of the milled product. During the precollaborative trial, it was decided that 250 g of wheat would be ground in a Perten LM120 or LM3100 mill fitted with a 0.8-mm screen (Perten Instruments Inc., Springfield, IL, U.S.A.).

There are three preliminary steps that need to be performed prior to the Mixolab analysis (Table II). First, if the sample is

Table I. Participants in the Mixolab interlaboratory study

Agri-Energia	Spain
AWB Limited	Australia
CHOPIN Technologies	France
CIMMYT	Mexico
CRA Gembloux	Belgium
Enzipan	Colombia
Granotec	Argentina
Granotec	Mexico
NDSU Northern Crops Institute	U.S.A.
Rheotec	Belgium
Dr. Schär	Italy
ULICE/LCI	France
USDA GIPSA	U.S.A.

ground wheat, it must be ground in a laboratory mill fitted with a 0.8-mm screen. Second, the moisture of the sample must be determined. Finally, a preliminary hydration determination must be performed to ensure that the final Mixolab torque is within a specified range.

During a typical Mixolab sample analysis, five distinct phases can be observed in the Mixolab curve. The phases and the parameters measured during each phase are summarized (Table III). Figure 4 shows a typical Mixolab output obtained using the “Chopin+” protocol where the green curve shows the torque recorded by the sensor in Nm, the red curve shows the mixer temperature in °C, the pink curve shows the dough temperature in °C, and the purple horizontal line shows the target consistency (1.1 Nm) that must be achieved during the hydration determination. The torque recorded during phase 1 must be equal to 1.10 ± 0.07 Nm (horizontal dotted lines) to be able to compare the tests.

Results

Statistical analysis was completed on all parameters for both wheat and flour samples. We estimated reproducibility and repeatability standard deviation for the mixing parameters, T1, stability, and water absorption (WA); for the torque parameters,

C2, C3, C4, and C5; and for the temperature parameters, D2, D3, D4, and D5. We observed that for some parameters, there is no proportionality between the standard deviations and the average value. Therefore, for these parameters, we can assume constant

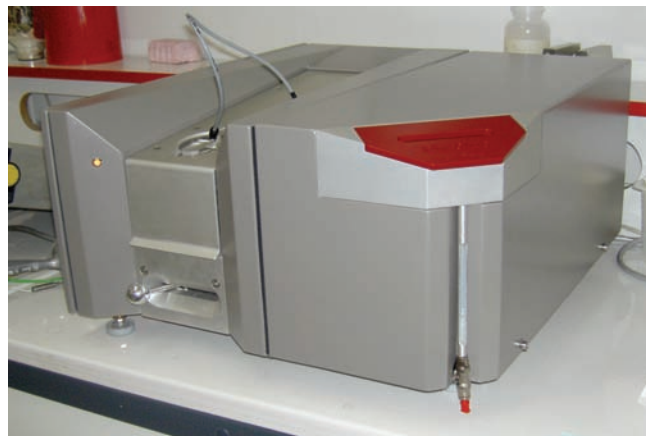


Fig. 1. The Mixolab, CHOPIN Technologies, Villeneuve la Garenne, France.

Table II. Mixolab preliminary steps

Preliminary Step	Parameters Measured	Comments
Wheat grinding	None	250 g of clean wheat is ground in a laboratory mill equipped with a 0.8-mm screen.
Moisture determination	Sample moisture: H ₂ O%	Measure sample moisture by following AACC Intl. Approved Method 44-15.02 (1).
Hydration determination	Water absorption: WA%	A preliminary test is conducted using the sample moisture content at fixed water absorption (i.e., 60%). During the first 5 min of the test, the operator should verify that the maximum torque is within 1.10 ± 0.07 Nm. If in range, allow test to continue. If not within range, stop test, clean the instrument, and conduct a subsequent hydration determination, taking into consideration the maximum torque obtained during the first test and adjusting the water absorption in order to meet the target range.

Table III. Mixolab curve phases and related parameters

Phase	Phase Name	Parameters Measured	Comments
Phase 1 Hold at 30°C	Initial mixing	Maximum consistency during Phase 1: C1 in Nm Time to reach C1: T1 in min Time during which torque is greater than a value of C1–11%: Stability in min Dough temperature at C1: D1 in °C (or °F)	The dough and the mixer are kept at 30°C for 8 min. The special Mixolab kneading process ensures dough formation and weakening during this phase. Standard dough behavior data during mixing is also measured.
Phase 2 30–60°C	Weakening proteins	Minimum consistency during Phase 2: C2 in Nm Dough temperature at C2: D2 in °C (or °F)	When the dough temperature increases, a reduction in torque is observed. This drop corresponds to the weakening of the proteins when the temperature is increased, thereby providing an indication of protein quality.
Phase 3 60–90°C	Gelatinization	Minimum consistency during Phase 3: C3 in Nm Dough temperature at C3: D3 in °C (or °F)	At a certain temperature, the starch granules burst. This gelatinization phase is measured on the dough, at absorption levels close to those used during commercial processing conditions. The value of C3 depends on the starch characteristics and amylase activity of the sample.
Phase 4 Hold at 90°C	Stability during baking	Minimum consistency during Phase 4: C4 in Nm Dough temperature at C4: D4 in °C (or °F)	The difference between C3 and C4 indicates the stability of the starch gel when heated. This drop may be more pronounced when amylase activity is high.
Phase 5 90–50°C	Retrogradation	Maximum consistency during phase 5: C5 in Nm Dough temperature at C5: D5 in °C (or °F)	The increase in torque between C4 and C5 provides an indication of the way the starch retrogrades when dough temperature is decreased. Ongoing studies have shown that the measurement of retrogradation can be correlated with the staling phenomena in bread.

repeatability and reproducibility. We observed constant repeatability and reproducibility for all of the parameters studied with the exception of parameters T1 and stability, which show proportionality between the average value and the standard deviation.

Analysis of Precision Data

The statistical data for all parameters are shown in Tables IV–VI, and an overview of the results for the mixing phase parameters is shown in Table IV. The repeatability data for WA were obtained on the basis of blind duplicates. The repeatability and reproducibility estimates for stability and T1 are proportional to the average value. For example, for an average stability value of 7 min, $s(r) = 0.645$ min and $s(R) = 1.14$ min.

The precision data for the torque parameters are shown in Table V. For all torque measurements, the repeatability and reproducibility estimates (and therefore uncertainty) are constant.

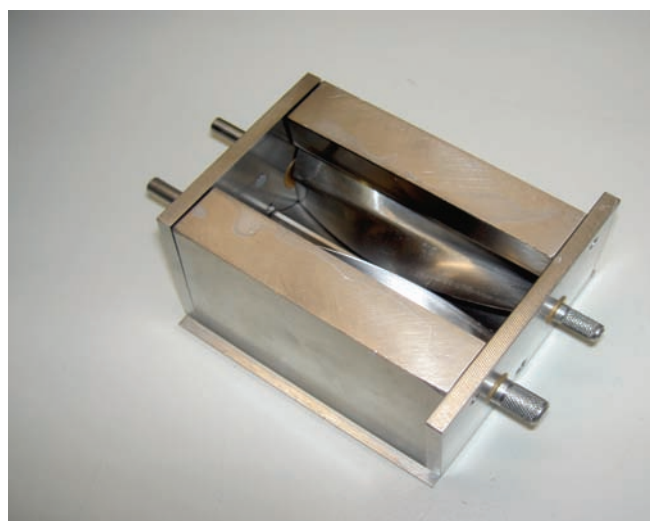


Fig. 2. A detailed image of the Mixolab.

These parameters are most important because they correspond to the Mixolab measurement. The precision data for all dough temperature parameters are shown in Table VI. For all of the dough temperatures measured, the repeatability and reproducibility estimates (and therefore uncertainty) are constant.

Blind Duplicate Study

Each laboratory received two identical wheat samples, which were coded differently so that the laboratory repeated the analysis on the same sample without prior knowledge. Of the 13 laboratories that participated in the study, two were excluded from the data analyses. One was due to the laboratory not following the specified method; the other was excluded by means of the Cochran and Grubbs tests for outliers as described in ISO Method 5725-2 (3). The average and statistical results obtained for 11 laboratories are shown in Tables VII, VIII, and IX.



Fig. 3. The Mixolab dismantled.

Table IV. Overview of precision data for mixing phase parameters

	Mean	s(r)	s(R)
Water absorption (WA)	51.6–63.4 (%)	0.378%	0.90%
Stability	4.45–11.20 (min)	$= (-0.0902 * \text{stability} + 1.2762)$	$= (-0.1513 * \text{stability} + 2.2014)$
T1	1–7.30 (min)	$= (0.0814 * T1 + 0.1252)$	$= (0.01761 * T1 + 0.1147)$

Table V. Overview of precision data for torque parameters

	Mean (Nm)	s(r) (Nm)	s(R) (Nm)
C2	0.37–0.63	0.01	0.03
C3	1.59–2.27	0.02	0.08
C4	0.95–2.12	0.03	0.09
C5	1.46–3.73	0.08	0.19

Table VI. Overview of statistical data for dough temperature parameters

	Mean (°C)	s(r) (°C)	s(R) (°C)
D2	52.2–57.7	0.65	1.59
D3	75.2–86.2	0.78	1.69
D4	83.5–88.7	0.77	1.72
D5	58.1–60.6	0.74	1.65

Table VII. Blind duplicate analysis for torque parameters

	Sample	B3/B7	B9/B12
C2	N Labs	(13/11)	(13/11)
	Mean	0.48	0.59
	s(r)	0.0200	0.0194
	s(R)	0.0334	0.0342
C3	N Labs	(13/11)	(13/11)
	Mean	1.77	2.12
	s(r)	0.0383	0.0372
	s(R)	0.0797	0.0796
C4	N Labs	(13/11)	(13/11)
	Mean	1.28	1.97
	s(r)	0.0738	0.0563
	s(R)	0.0907	0.1376
C5	N Labs	(13/11)	(13/11)
	Mean	1.93	2.95
	s(r)	0.1482	0.1082
	s(R)	0.1964	0.1731

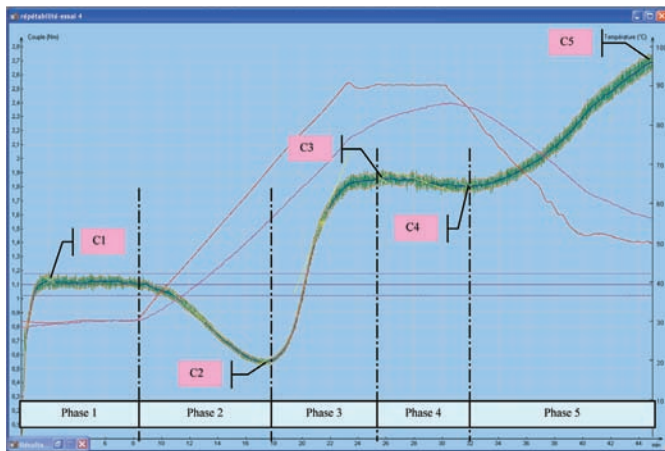


Fig. 4. Mixolab test results—The curve.

Table VIII. Blind duplicate analysis for temperature parameters

	Sample	B3/B7	B9/B12
D2	N Labs	(13/11)	(13/11)
	Mean	56.82	56.22
	s(r)	0.7138	0.8420
	s(R)	1.4844	1.6050
D3	N Labs	(13/11)	(13/11)
	Mean	77.67	79.13
	s(r)	0.6767	0.9390
	s(R)	1.8601	1.8817
D4	N Labs	(13/11)	(13/11)
	Mean	85.36	86.54
	s(r)	1.0399	0.6545
	s(R)	2.0511	1.5835
D5	N Labs	(13/11)	(13/11)
	Mean	59.06	59.65
	s(r)	0.7814	0.8666
	s(R)	2.5710	2.6842

Table IX. Blind duplicate analysis for WA, T1, and stability

	Sample	B3/B7	B9/B12
Water analysis (WA)	N Labs	(13/11)	(13/11)
	Mean	61.39	60.18
	s(r)	0.7648	0.6581
	s(R)	0.9541	0.8675
T1	N Labs	(13/11)	(13/11)
	Mean	5.23	7.15
	s(r)	0.0508	0.9457
	s(R)	0.7911	1.3342
Stability	N Labs	(13/11)	(13/11)
	Mean	10.32	11.290
	s(r)	0.2357	0.2365
	s(R)	0.3551	0.3378

Conclusions

The Mixolab is an instrument that allows users to thoroughly analyze wheat and flour samples. It has been used in numerous research laboratories worldwide and is gaining recognition as a quality control tool in flour mills and quality labs.

In this study, two matrices (flour and ground wheat) were evaluated. It is notable that similar results were observed for the precision data obtained with flour and with ground wheat. Therefore, it is possible to simplify testing by providing operators with the same standard for the two matrices.

The results of this study were accepted by the AACC Intl. Physical Testing Methods Technical Committee, and the method is under review by the AACC Intl. Approved Methods Technical Committee. If approved, it will be published as AACC International Approved Method 54-60.01 (see Editor's Note below).

Acknowledgments

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Editor's Note

As the May-June issue of *Cereal Foods World* went to press, the AACC International Approved Methods Technical Committee was reviewing this method for inclusion in the online *AACC International Approved Methods of Analysis, 11 Edition*. If approved, the method is expected to post online soon.

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