

# METHODS FOR MEASURING REACTIVITY OF CHEMICAL LEAVENING SYSTEMS

## I. Dough Rate of Reaction<sup>1</sup>

JOHN R. PARKS<sup>2</sup>, AVROM R. HANDLEMAN<sup>3</sup>, J. C. BARNETT<sup>3</sup>,  
AND F. H. WRIGHT<sup>3</sup>

### ABSTRACT

The reaction rate of a chemical leavening system in a specified biscuit dough is measured manometrically during mixing by a standardized procedure. Operations of the apparatus are electronically programmed. Small pressure increases (resulting from evolution of leavening gas) are plotted as percent of total leavening against time, on a strip chart recorder.

Flour differences, flour age, and the presence or absence of milk and shortening influenced test results. Differences in geometry of the closed system, stirring rate, and test temperature also significantly affected the test values.

In the procedure employed with sodium acid pyrophosphate, the standard error of single determinations was 0.5; with anhydrous monocalcium phosphate the standard errors were 0.7 and 1.2 for 2- and 10-minute measurements respectively.

Although about 100 million pounds of leavening acids are consumed each year in self-rising flours, commercial and household baking powders, prepared mixes, and refrigerated bakery products, very little has been published concerning their mode of action. It is the purpose of this series of papers to describe methods for measuring reactivities of the important leavening chemicals and the effects of reactivity on quality of the leavened product.

Over-all quality of chemically leavened products may be affected by leavening during mixing, holding or handling prior to baking, and baking or frying. Knowledge of the reactivities of leavening systems is used in product development and in specifying performance characteristics of certain leavening acids.

The measurement of reactivity is important for control purposes where the leavening agent can vary in rate characteristics, especially when a particular reactivity range is required. Two such adjustable leavening acids are stabilized anhydrous monocalcium phosphate (AMCP) and sodium acid pyrophosphate (SAPP).

A satisfactory procedure for measuring reactivity must provide

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<sup>2</sup> Applied Mathematics Section, Research and Engineering Division.

<sup>3</sup> Respectively: Research Department, Technical Service, and Research Department, Inorganic Chemicals Division.

1) a reliable means for quantitatively measuring gas formation, and 2) a standardized method for combining the reactants and specified dough ingredients. Very close temperature control is mandatory for comparable results, and all methods involve maintaining the dough container at constant temperature.

Several different principles have been employed to follow the evolution of gas into and from test doughs or batters. Although these techniques were usually employed with yeast systems, most would be applicable to chemical leavening systems as well.

Pressure measurements employing mercury manometers (7,30) or liquid-filled Bourdon tube gages (24) attached to sealed dough containers have long been used. Solutions of brine and sodium hydroxide have been included, on occasion, to control humidity and to differentiate between retained and expelled gas respectively (16,35). Pressure-recording devices have been developed for manometers (35) and for use with electronic pressure transducers (29).

Constant-pressure gas production has been followed with different types of gas burets (11,31,32). Bailey (2) modified a volumetric device to estimate gas pressure within a fermenting dough. Weight-recording devices, which sense gas evolution by changes in the buoyancy of a system, have been employed (8,12,14). The relative effectiveness of these techniques has been evaluated by several workers (11,15,31).

Amount of liquid expelled from a closed rigid system has been used to indicate gas production (20,27,28). Modifications of this principle to permit measurement at constant pressure have been described (12,17,18,23,25).

Sequential carbon dioxide analyses of chemically leavened doughs<sup>4</sup> (21) have been employed to follow leavening reaction during baking. Simultaneous measurements of dough expansion and carbon dioxide analysis of the atmosphere above the dough were employed to follow retained and evolved gas during fermentation.

Special techniques have been developed to estimate (26) and to measure (19,22) the instantaneous rate of gas evolution. Tracer techniques have been used to study carbon dioxide diffusion rates (9). The possible use of C<sup>14</sup> sodium bicarbonate as a guide for following gas evolution from chemically leavened systems has been discussed in this laboratory, but technical difficulties in counting beta-radiation limit that approach at this time.

An apparatus for measuring gas production in a chemically leav-

<sup>4</sup> Handleman, A. R., and Joslin, R. P. Determination of baking rate of reaction by means of the Chittick gasometer. Presented at the 41st annual meeting, New York City, May 1956.

ened batter or dough was described by Barackman (6). This device employed a metal bomb fitted with a stirring device, the shaft of which passed through a mercury seal to prevent leaks. Gas production was detected in a buret (10). Comparative data on several leavening acids were reported by Van Wazer and Arvan (34), who used a similar device. In these devices, water is added to dry dough or batter ingredients after the system is closed.

Standardized methods for measuring the reactivities of sodium acid pyrophosphate (SAPP) and anhydrous monocalcium phosphate (AMCP) during dough mixing are described in this paper. These methods were developed to control the uniformity of these products or their various grades. The methods are described in detail, along with sources of error and estimates of precision. Principles of apparatus design are outlined. Design details and working drawings can be obtained by writing to one of the authors<sup>5</sup>.

### Materials and Methods

The factors to be considered in measuring the dough rate of reaction (DRR) of a chemical leavening system include control of temperature, a uniform method of combining reactants, maintenance of a constant environment, and provision of means for detecting the extent of reaction as a function of time. The apparatus and procedure described below were designed to provide control over these factors and to minimize time required per measurement for the technician.

In essence, the test consists of 1) preparing a complete, dry dough mix; 2) adding a standard amount of water; 3) carrying out a standard mixing and holding cycle in a closed system; and 4) measuring gas production as a function of time. Whether the gas remains in the dough or leaves it is of little consequence in measuring the *reactivity* characteristics of the leavening acid.

*Apparatus for Dough Rate of Reaction.* The apparatus used senses very slight pressure changes electronically by means of a linear transducer. As an aid to reproducibility and simplicity of operation, the apparatus was programed to carry out all the steps of a determination automatically. Figure 1 is a photograph of the DRR machine, and Fig. 2 is a block diagram showing the essential components, which are 1) a reaction bomb fitted with a stirring mechanism; 2) a thermostatted bath (held at  $27 \pm 0.01^\circ\text{C}$ .) in which the bomb can be immersed; 3) a mechanism for adding a predetermined amount of water; 4) ballast tanks in the gas-receiving system to make changes in pressure small; 5) a linear pressure transducer connected to an electronic

<sup>5</sup> J. C. Barnett.

recorder capable of being adjusted for zero, millivolt span, and sensitivity; 6) a programming circuit to control the sequence of operations; and 7) a large air reservoir on the low-pressure side of the transducer

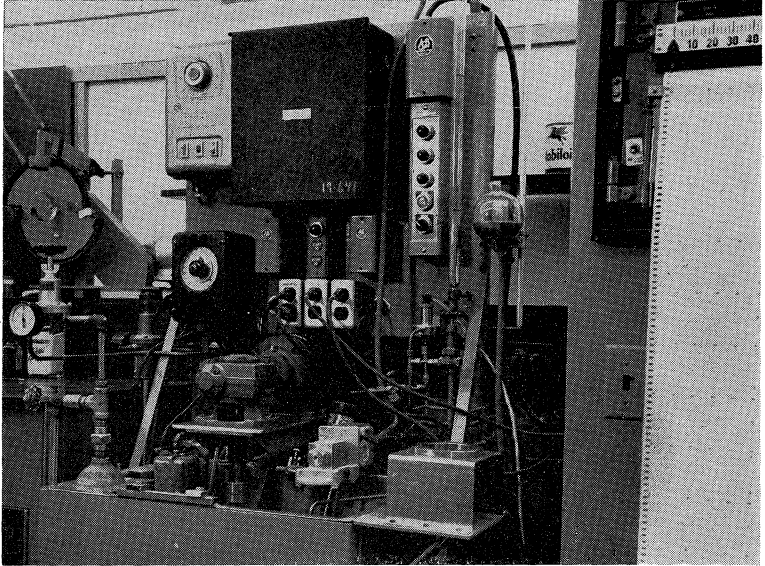


Fig. 1. Programed apparatus for measurement of dough reaction rate.

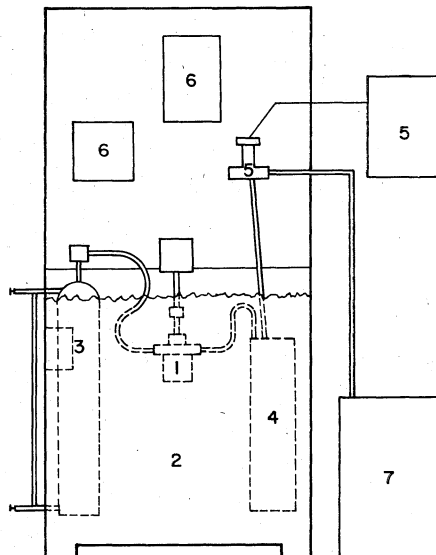


Fig. 2. Block diagram of programed apparatus for measurement of dough reaction rate. Numerals designate components described in test.

for isolating the system from the atmosphere during a test, to eliminate the effects of slight changes in atmospheric pressure. The pressure transducer-recorder system is calibrated to plot percent of total carbon dioxide evolved as a function of time.

The program is arranged 1) to provide a 5-minute delay for bomb and reactants to come to bath temperature; 2) to add the predetermined volume of water; 3) to close the system; 4) to stir for the standard time (3 minutes for SAPP and 10 for AMCP); 5) to keep the system closed until the cycle is complete (8 minutes for SAPP and 10 for AMCP); and then 6) to open the system and return controls to the start position. These operations could be carried out manually and gas evolution detected by other means so long as the equipment is calibrated. The electronic pressure-sensing system permits measurement of gas evolution with only slight changes in pressure on the dough (12 in. of water), but a mechanical pressure-measuring device might be employed, if larger pressure changes are shown not to bias results.

Figure 3 shows the bomb and stirring mechanism. For the work reported below, a stirring rate of 120 r.p.m. was used unless otherwise specified.

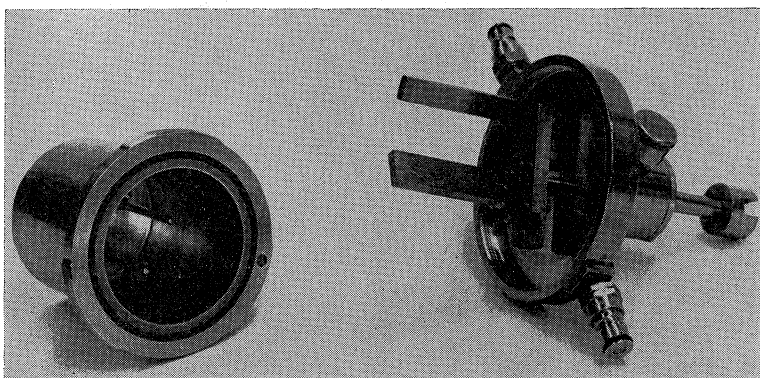


Fig. 3. Bomb and stirrer of apparatus for measurement of dough reaction rate.

*Dough Ingredients and Laboratory Apparatus.* Ingredient specifications and amounts used in doughs as employed for sodium acid pyrophosphate (SAPP) and coated anhydrous monocalcium phosphate (AMCP) are presented in Table I.

*Laboratory Apparatus.* Equipment required other than normal laboratory apparatus and the DRR machine itself is 1 table fork; 1 mixing bowl, 120–125-mm. diameter at top (an evaporating dish is

TABLE I  
COMPOSITION OF TEST MATRICES

INGREDIENT	SPECIFICATION	AMOUNT USED	
		Sodium Acid Pyrophosphate Matrix	Anhydrous Monocalcium Phosphate Matrix
		g	g
Flour	Highly chlorine-bleached (pH 4.8-5.0) low-protein angel food cake flour (12.5% moisture basis)	57.0	57.0
Nonfat dry milk	High-heat spray-dried	5.0	5.0
Shortening	Hydrogenated vegetable	6.0	0
Salt	Reagent grade sodium chloride	1.0	0
Soda	Granular sodium bicarbonate (about 5% retained on 100-mesh and about 5% through 270-mesh)	0.755	0.755
Leavening acid	Test	1.057	0.904
Water	Distilled, held at run temperature (27°C.)	40.0	40.0

satisfactory); two 16-oz. wide-mouth, screw-cap glass jars; flour sifter; 1-gal. wide-mouth jar.

*Test Procedure.* Sift flour for a single day's tests four times and store in a 1-gal. screw-cap jar. Weigh dry ingredients other than shortening and place in 16-oz. bottle. Weigh flour and nonfat dry milk first and agitate sufficiently to partially blend before adding other materials. Blend dry ingredients by tumbling for 3 minutes, tapping the top and bottom of the jar on laboratory bench to free adhering material. In testing SAPP, place the blended dry ingredients in the evaporating dish and cut in the shortening with 50 downward strokes of the fork, using the edge rather than the flat. Clean the fork on the side of the bowl and repeat.

Place the resulting dry mixture in the bomb and attach to the DRR apparatus. Start the program already described. The DRR value of SAPP is defined as the 8-minute reading. For AMCP the 2- and 10-minute readings are taken.

The results with this programed machine were slightly higher than those obtained with a basically similar, manually operated device in which gas production was measured volumetrically with a Chittick gasometer (34). The results by the two machines were highly correlated; the differences in the absolute values are attributed to stirring rate and the dimensions of bomb and stirrer. AMCP results are corrected to correspond to results which would be obtained with the old machine in the data presented below, but SAPP results are presented as observed.

### Typical Results

Typical DRR curves obtained with monocalcium phosphate monohydrate ( $MCP \cdot H_2O$ ) and AMCP are shown in Fig. 4. The standard dough formula and procedure developed for AMCP were used in obtaining these curves. Values for 2- and 10-minute DRR, taken from these curves, are presented in the table below. The curve for

*Test Results with AMCP*

Sample No.	2-minute DRR	10-minute DRR
1	22.5	72.0
2	26.5	74.5
3	37.0	72.0

sodium bicarbonate alone shows the amount of reaction to be expected from the acidity of the dough constituents. The test procedure provides a means for selecting or specifying materials with desired characteristics such as AMCP's 1 and 2, which are typical of the extremes of good commercial products. Materials with limited delay, such as AMCP 3, can be rejected, thereby reducing product variation due to leavening characteristics. The test is of little value when applied to

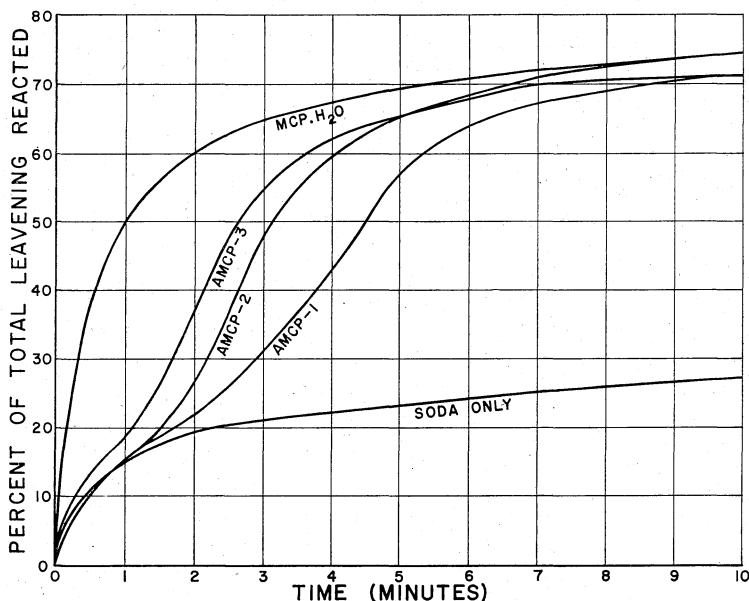


Fig. 4. Dough reaction rate curves obtained with anhydrous monocalcium phosphate (AMCP) and monocalcium phosphate monohydrate ( $MCP \cdot H_2O$ ).

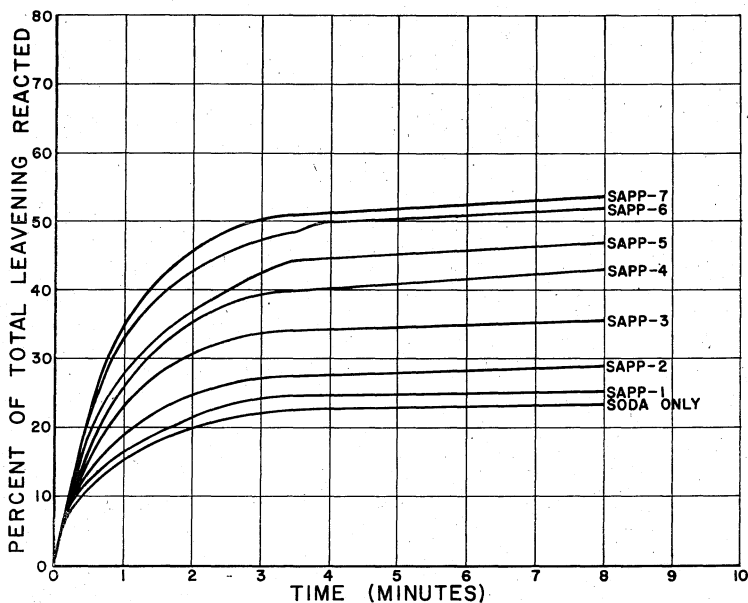


Fig. 5. Dough reaction rate curves obtained with sodium acid pyrophosphates (SAPP's) of varying reactivity.

MCP·H<sub>2</sub>O, because this material has little or no delay as shown by the curve, which is characteristic for this material and shows little variation from sample to sample.

A number of typical DRR curves obtained with SAPP's of a wide range of reactivities are illustrated in Fig. 5. DRR values (8-minute reading) for these materials and for the test in which leavening acid was omitted are listed in the table below. From these data, it is appar-

*Test Results with SAPP's*

Sample No.	DRR Value (8-minute reading)
1	(Soda only) 25.0
2	28.5
3	35.5
4	42.5
5	46.5
6	52.0
7	54.0

ent that SAPP's can be prepared so as to give a wide range of dough reactivities. Since SAPP's of several different reactivities are commercially available, it is possible to specify a material with reactivity most suitable for a specific application on the basis of its DRR value.



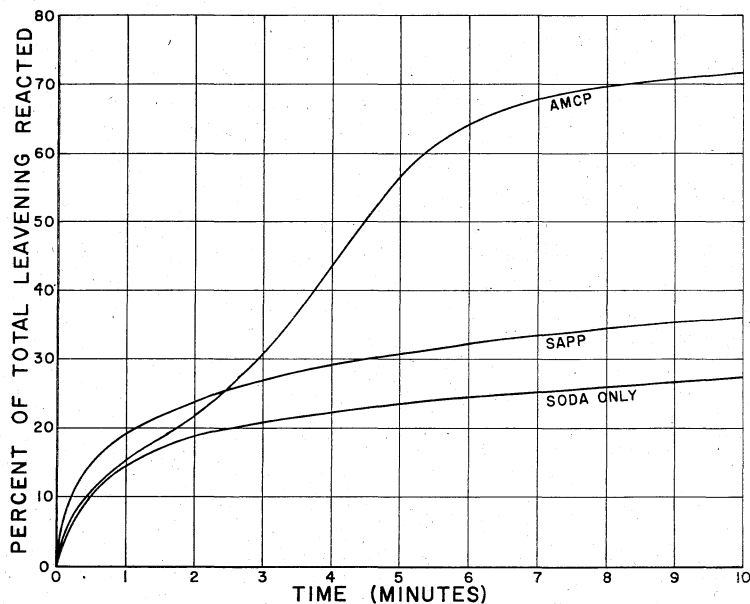


Fig. 6. Comparison of delayed leavening characteristics of sodium acid pyrophosphate (SAPP) and anhydrous monocalcium phosphate (AMCP) determined by the AMCP method.

In Fig. 6, the AMCP standard procedure is used to illustrate the basic difference between the modes of action of AMCP and SAPP. It is apparent that the delay in AMCP is quite temporary, whereas that of SAPP is of considerably longer duration. Both react to near completion in the oven (see footnote 4). Selection again depends on product requirements.

#### Precision of Dough Reaction Rate Test

The SAPP standard procedure yielded results with a standard error  $\pm 0.5$  for single determinations. This value was obtained from 25 determinations with one sample and no change in ingredient supply. The tests were made by five different operators on five days during a 3-week period. Other measurements made before and after this test agree well with this value. Since SAPP's are commercially available in a DRR range from 24 to 43, this test is capable of distinguishing several classes within the range.

Results obtained with the AMCP procedure had standard errors of  $\pm 0.7$  for the 2-minute value and  $\pm 1.2$  for the 10-minute value. These values were obtained from 39 determinations with no change

in the ingredient supply. The values agree with previous and later determinations. Since AMCP is not available in a series of grades, the test results are used to indicate product quality and uniformity. The lower limit of the 2-minute value is that obtained when sodium bicarbonate is reacted without an acid leavener present. AMCP should not have a 2-minute value more than a few percent above that obtained for soda alone.

### Sources of Variation in Test Results

The important variables have been examined for their effects on test results. Although temperature data are not presented, the DRR increases about 10% per degree Centigrade in the practical temperature range. Further, the large ballast tanks used in the above-described apparatus to maintain nearly atmospheric pressure make this apparatus very sensitive to changes in bath temperature during a run. Variations in bath temperature of 0.1°C. produce variations in observed DRR of 0.9. For this reason, a large bath with a very sensitive temperature control is used. Other important effects are treated individually below.

*Flour.* The flour used in this test is specified as highly chlorine-bleached angel food cake flour, because it is thought that such flour shows a relatively small change in acidity on storage. Nevertheless, differences between flours and changes during storage affect the DRR test. Table II compares the 2-minute and 10-minute DRR values obtained with three different batches of flour. Results obtained

TABLE II  
EFFECT OF FLOUR ON TEST RESULTS

FLOUR SAMPLE <sup>a</sup>	DRR VALUES OBTAINED WITH SODIUM BICARBONATE BUT NO ACID LEAVENER BY AMCP PROCEDURE	
	2-Minute Value	10-Minute Value
A	27.4	35.5
B	24.5	32.2
C	23.2	31.6

<sup>a</sup> Average of 4, 15, and 28 determinations, respectively.

with flours B and C do not differ significantly, but flour A differs from flour C by about 4 DRR units and should be discarded. When changing flours, it is necessary to select a new supply which gives results agreeing with those from the old; otherwise, reactivities measured with the different flour supplies will not be comparable.

As flour ages, its effect on the observed DRR changes. In Fig. 7, observed DRR values are plotted against flour age for two flours.

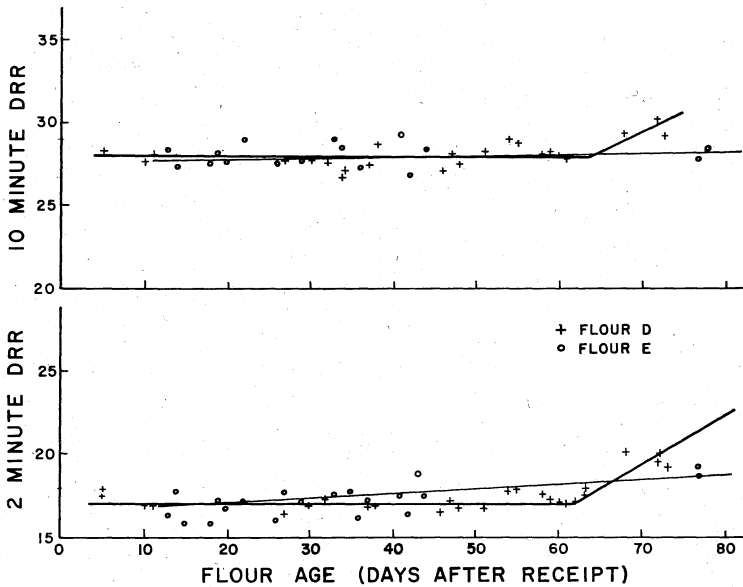


Fig. 7. Effect of flour age on test results with sodium bicarbonate only, for two samples of dough reaction rate (DRR) flour.

While aging caused increased apparent DRR values with both flours, the rate of change for flour D increased abruptly after 60 days, whereas that for flour E continued to increase gradually. The effects of flour variations can be minimized by maintaining a record of DRR obtained with sodium bicarbonate alone and comparing daily results with the average. When the values observed in repeat runs are out of line, it is time to change flour.

*Nonfat Dry Milk.* Milk exerts a pronounced effect on the behavior of leavening agents. Table III shows that the DRR values for sodium bicarbonate alone are higher in the presence of milk, whereas those for SAPP are much lower in its presence. This difference is explained by the fact that alkaline earth ions supplied by the milk are required for the SAPP delay to be manifested (33). With AMCP, milk is also required for the typical 2-minute delay, as shown in Table IV. In this case, however, milk has an opposite effect on the 10-minute value. The relationship is not yet understood.

Because of their critical effect on observed DRR value, milk solids must be included in test doughs, since the majority of leavened products contain milk.

Variations in the milk effect have been noted with different batches of aged milk solids. For this reason the supply of milk solids should

be stored at 0° to -5°F. New supplies of milk should be checked against the old for test results with the same leavening acid. Only milk giving results which agree with those obtained with the standard supply should be used in the test dough.

*Salt.* Table IV presents data obtained, with and without salt, for sodium bicarbonate alone and for a complete AMCP leavening system. Salt did not have any significant effect on the results. Earlier DRR work was carried out with the same dough ingredients for testing both SAPP and AMCP. Salt has been dropped from the AMCP procedure for simplification. It is included in the SAPP procedure, because SAPP data comparable to that in Table IV are not yet available, but its value is questionable.

TABLE III  
EFFECT OF NONFAT DRY MILK ON TEST RESULTS

SPRAY-DRIED NONFAT DRY MILK	DRR VALUES OBTAINED WITH SODIUM BICARBONATE BY THE AMCP PROCEDURE	
	2-Minute Value	10-Minute Value
Present <sup>a</sup>	24.5	32.2
Absent <sup>b</sup>	18.5	23.1
With AMCP present		
Present	27.5	75.5
Absent	47.5	70.5
DRR Values Obtained with Sodium Bicarbonate and SAPP by the SAPP Procedure (8-Minute Values)		
	SAPP-x	SAPP-y
Present	29.2	28.4
Absent	47.1	43.6

<sup>a</sup> Average of 15 determinations.

<sup>b</sup> Average of 4 determinations.

TABLE IV  
EFFECT OF SALT ON RESULTS WITH ANHYDROUS MONOCALCIUM PHOSPHATE

REAGENT GRADE SALT	DRR VALUES OBTAINED WITH SODIUM BICARBONATE	
	2-Minute Value	10-Minute Value
No acid leavener		
Present <sup>a</sup>	23.9	31.5
Absent <sup>b</sup>	24.5	32.2
AMCP as acid leavener		
Present <sup>c</sup>	33.9	83.5
Absent <sup>d</sup>	32.2	81.4

<sup>a</sup> Average of 7 determinations.

<sup>b</sup> Average of 15 determinations.

<sup>c</sup> Average of 4 determinations.

<sup>d</sup> Average of 2 determinations.

*Shortening.* The data in Table V suggest a slight decrease in reactivity when shortening is present in the test dough. This effect is attributed to an interference with wetting rather than an influence on the characteristics of the leavening acid being tested. It is thought that omitting shortening has no effect on the relative reactivities observed, and it has been dropped from the AMCP procedure to avoid a possible source of human error and the work of incorporating it. It is still included in the SAPP procedure, however, because data comparable to that obtained with AMCP are not available.

*Sodium Bicarbonate.* Sodium bicarbonate is available in relatively pure form, and differences between grades are usually related to particle size. By specifying a particle size range, variations due to soda will be avoided.

*Machine Differences.* Two bombs and two stirrers, originally identical, were used in obtaining the data in Table VI. Stirrer A had become slightly bent and could not be perfectly restored to its former configuration. The data show no difference between the bombs, but there was a distinct difference between results obtained with the two stirrers. The difference points up the importance of geometry, if comparable results between laboratories are desired. A correlation between results obtained with the different stirrers can be established, but

TABLE V  
EFFECT OF SHORTENING ON TEST RESULTS

HYDROGENATED VEGETABLE OIL (Shortening)	DRR VALUES OBTAINED WITH SODIUM BICARBONATE	
	2-Minute Value	10-Minute Value
	No acid leavener	
Present <sup>a</sup>	23.5	30.5
Absent <sup>b</sup>	23.9	31.5
	AMCP as acid leavener	
Present <sup>c</sup>	32.1	80.7
Absent <sup>c</sup>	33.9	83.5

<sup>a</sup> Average of 7 determinations.

<sup>b</sup> Average of 6 determinations.

<sup>c</sup> Average of 4 determinations.

TABLE VI  
EFFECT OF DIFFERENT BOMBS AND STIRRERS ON TEST RESULTS

STIRRER	DRR VALUES OBTAINED WITH SODIUM BICARBONATE BUT NO ACID LEAVENER OR MILK BY AMCP PROCEDURE			
	BOMB A		BOMB B	
	2-Minute Value	10-Minute Value	2-Minute Value	10-Minute Value
A	20.7	24.5	20.0	24.2
B	18.4	23.4	18.5	22.6

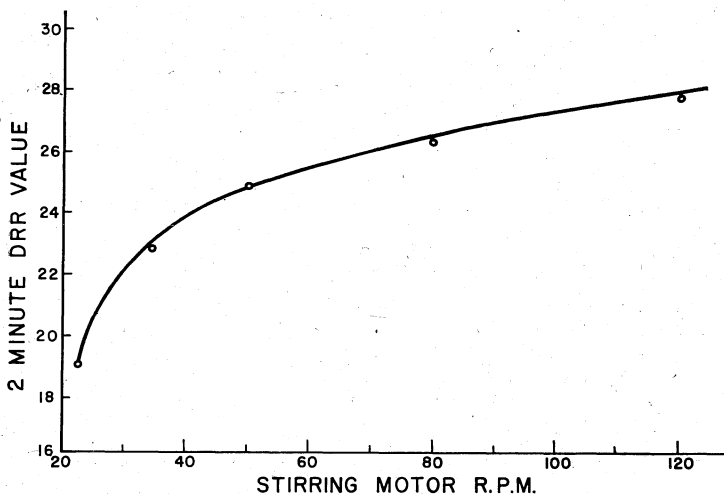


Fig. 8. Effect of stirring rate on 2-minute dough reaction rate observed with anhydrous monocalcium phosphate.

to obtain values which agree, they, and presumably the bombs, must be identical. The gas production sensor can employ nearly any of the principles described in the literature review so long as it is properly calibrated.

*Stirring Rate.* Figure 8 shows the effect of increased stirring rate on observed DRR. As rate increases, the effect of variations diminishes. The arbitrarily chosen rate of 120 r.p.m. was selected for this equipment, because lower rates gave larger experimental errors and higher rates offered no advantages.

### Conclusions

DRR procedures have been used as leavening performance yardsticks for at least 25 years, but their usefulness has been curtailed by lack of standardization of methods among laboratories. It was possible, in a given laboratory at a given time, to compare the reactivities of leavening acids, but results obtained at a later date or in another laboratory were not comparable.

By standardizing test ingredients and test procedure and by correlating DRR equipment with a given reference apparatus, it is possible to obtain comparable results in different laboratories. To establish the relationship between two DRR machines, measurements should be made on several leavening acids of different reactivities with the two machines.

The importance of more exact definition of leavening reactivity

characteristics is being heightened by the development of new products with more stringent leavening requirements and less tolerance for variation. The DRR test described in this paper has proved satisfactory for this purpose in routine commercial practice.

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