

NORTH CENTRAL REGIONAL PUBLICATION NC-140

# Quality Evaluation of Soft Winter Wheat

Progress Report of Work Under Regional Projects NC 30 and NCM 28

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Agricultural Experiment Stations of Illinois, Indiana,  
Michigan, Minnesota, Missouri, Nebraska  
North Dakota, Ohio and Wisconsin

in Cooperation with

Crops Research Division  
Agricultural Research Service  
United States Department of Agriculture

**THE OHIO AGRICULTURAL EXPERIMENT STATION**  
Wooster, Ohio

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## FOREWORD

This Research Bulletin is published under the auspices of Regional Project NCM-28, and includes work done under NC-30 which terminated January 30, 1960. The area of work is indicated in the title, "Market Quality and Utilization of Soft Winter Wheats." The publication has the approval of the Directors of the Agricultural Experiment Stations of Illinois, Indiana, Michigan, Missouri, Nebraska, North Dakota, Ohio and Wisconsin of the North Central Region. Most of the work was done in cooperation with the Crops Research Division Agricultural Research Service, United States Department of Agriculture. The primary objective of the project was, and is, an expansion of the eastern soft winter wheat varietal evaluation program of the Soft Wheat Quality Laboratory at Wooster, Ohio. Special emphasis has been placed on quality evaluation studies involving cake baking and more recently work involving the turbo-separation (air-classification) of eastern soft wheat flours, together with particle-size distribution studied on various types of soft wheat flours and their air-classified fractions.

Mr. D. H. Donelson, a chemist, was hired August 1, 1955, under funds made available to the Ohio Station under the NC-30 project for the 1956 fiscal year. With the added funds available for 1957, Mr. J. T. Wilson was hired effective September 1, 1956. These two men conducted cake quality studies at the Soft Wheat Quality Laboratory in cooperation with the C.R.D., ARS, U.S.D.A. Effective July 1, 1959, Mr. C. R. Sipes was hired as an additional member of the team under the North Central States Project. His principal duties have been concerned with turbo-classification and particle-size distribution studies on soft wheat flours and related products. The work of these men is summarized in this research bulletin. Studies of the action of chlorine on starch, conducted at Purdue University, under the direction of Dr. R. L. Whistler, with monies from the regional project, are also reported. Only highlights of the findings to date have been included.

The relatively short time that this project has been underway prohibits the drawing of many general conclusions. This publication is therefore more in the nature of a progress report of the cake baking studies and related investigations which have been conducted since the start of the cooperative program at the Wooster Laboratory in 1955.

Conclusions drawn from the research results presented seem logical in light of the available information, but it is to be expected that additional knowledge may change or modify some of the interpretations.

The purpose of this publication is to provide Ad-

ministrators, Cereal Researchers, Teaching and Extension personnel, and others with a brief review of some of the accomplishments of this project and an appraisal of its present status.

This research bulletin was prepared by C. A. Lamb and C. E. Bode in collaboration with the members of the Technical Committee and those active in the work under the project.

## INTRODUCTION

Bleached and unbleached soft wheat flours are used mainly for pastry purposes. Included in the list of baked products making use of soft wheats are various types of the following: biscuits, cakes, cookies, cones, crackers, doughnuts, pie crusts, pretzels, and other specialty products. The diversity of these food items and the various extractions of flours required for their production, plus the added confusion of the maturing or bleaching of certain of the flours prior to use, complicate the testing program for the quality evaluation of newly developed soft wheat selections. This development program is a vital step to insure adequate production of disease- and insect-resistant soft wheats for the future. Concern over the quality characteristics of the new varieties led to the establishment of the regional Soft Wheat Quality Laboratory at the Ohio Agricultural Experiment Station in 1936. Wheat breeders of the area cooperated with the newly established testing laboratory by submitting wheat samples for quality evaluation. It has been the practice to appraise the quality of the newer material in terms of the results obtained on a companion check sample of a known commercially acceptable variety grown in the same test field.

Tests used to evaluate varieties were not considered wholly satisfactory, and too little was known of the role of the flour in the various soft wheat products. Concurrent with the quality evaluation program, a part of the staff has devoted full time to fundamental re-research into certain aspects of soft wheat quality. A sugar-snap cookie has been used at the Laboratory as one of the principal baking methods in the quality evaluation of unbleached, straight-grade, experimentally milled soft wheat flours. To date, considerable progress has been made in determining the role of the various ingredients in affecting the quality of this product. Some of the fundamental research involved the mechanical separation of flours into four or more fractions. In the most commonly used procedure, four components were separated out: 1) gluten, 2) prime starch, 3) tailings starch, and 4) water solubles. Techniques were developed whereby this separation could be made, the flours reconstituted and cookies baked which were very similar in all respects to those

obtained using original flour. This technique permitted varying the proportion of the fractions, substitution of fractions from different varieties, treatment of single fractions, etc., and has proven to be a powerful tool for investigating differences between varieties.

In 1954 the North Central States Soft Wheat Technical Committee recommended to the Directors of the Experiment Stations of the region that additional basic research work on soft wheat quality be undertaken. As a result, the NC-30 project was activated. The first funds were made available for fiscal year 1956. The decision was made to concentrate the research effort on cake flour quality because this was one of the principal products for which soft wheat flour was used and one upon which more information was vitally needed. It was further decided to concentrate the initial effort at the Ohio Station, where the work could be done in the Soft Wheat Quality Laboratory, with the benefit of collaboration with the laboratory personnel and the opportunity to use the laboratory equipment, thus saving duplication of expensive apparatus.

## PROJECT ACTIVITIES

### Early Work

A cooperative project between the Department of Field Crops and Home Economics, Missouri Agricultural Experiment Station, and the Scott County Milling Company, Sikeston, Missouri, was started in 1945. The purpose of this project was twofold: a) to test and evaluate experimentally milled flours from the most promising wheats selected in the breeding program, and b) to study the utilization of soft wheat flour for culinary purposes.

With the establishment of the NC-30 project, Missouri 205 became a contributing project although no funds from NC-30 were allocated to it.

During the period 1955 through 1958, 34 varieties and experimental strains were tested for quality. Grain samples were supplied by the Department of Field Crops from the breeding plots, experimental milling was done in the laboratories of the Scott County Milling Company, Sikeston, and baking of experimental cakes and cookies was done in the laboratories of the Department of Home Economics. Routine tests made on the experimentally milled flours included mixogram area, viscosity, and the baking of experimental cakes and cookies. Results from these tests were used to evaluate the suitability of new strains developed in the breeding project for soft wheat milling and baking purposes. Experimental strains found to be equal or superior in quality to the

check varieties, Knox and Vermillion, were Missouri selections W6185, W6197, W6551, W6538, W6624, W6448, and W6696.

The testing of the experimental flours was discontinued after the 1958 season.

### Flour Fractionation

As indicated earlier, the decision had been made to limit the project activities at the Ohio Station to work on cakes and cake flours. Donelson began by attempting to apply to cakes the flour fractionation procedure developed by Yamazaki (12) in his cookie work. It was found that the technique was not satisfactory. The first problem then was to establish a method of flour fractionation that would be satisfactory for investigation of cake flours.

After numerous trials the large-scale batter process of Adams (2) was reduced to laboratory proportions and adopted as a standard procedure. The method involves collection of the gluten fraction on a vibrating silk screen after the formation of a flour and water batter. The starch filtrate, passing through the screen, is centrifuged and the water solubles, starch tailings, and prime starch separated by this means. After separation, all fractions are dried by lyophilization and stored for later use. In the course of work with reconstituted flour blends it was found best to rehydrate the combined fractions to the approximate moisture content of the original flour. Also, it was found desirable to add a part of the total water required in the finished cake and blend in a bread mixer until optimum gluten development was reached, before the addition of the remaining water and the other cake ingredients. The final mixing of the batter was done in a cake mixer equipped with a cake paddle. Similar techniques have been advocated by Yamazaki (12) and Sollars (7).

### The "Lean Formula" Cake

Kissell (6) had earlier developed a "lean formula" white layer cake test. This formula had been designed to place the maximum stress on the flour. This was accomplished by omission of such toughening ingredients as egg albumin and milk solids. On a laboratory scale, 150 g. of 50 percent extraction flour, bleached to pH 4.6-4.8, was mixed with sugar solution, emulsified shortening, baking powder, and water. Then 240 g. of batter was scaled in each of two 6-inch layer pans. The formula was very sensitive to liquid level, and optimum levels ranged from 103 percent to 121 percent depending on the flour used. There was satisfactory agreement in the ranking of flours by the lean formula and by the regular testing methods. This lean formula has been extensively used in the research program.

### Bleaching Flour Fractions

Commercial cake flours are all bleached with chlorine gas to a pH in the general range of 4.6 to 4.8. Flours from different sources or crops from different seasons may require greater or lesser amounts of treatment. A study was set up to determine which of the four flour fractions was affected by the bleaching. An unbleached commercial cake flour of 48 percent extraction was fractioned into water solubles, gluten, starch tailings, and prime starch. All but the water solubles were then rehydrated to approximately the moisture content of the parent flour. Portions of the fractions were treated at the rate of 0.2 ml. chlorine per gram. Using bleached and unbleached fractions, a series of reconstituted flours was made up as follows: all fractions unbleached, all fractions bleached, and one only of each of the four fractions bleached. These flours were then baked into cakes using the lean formula. Resulting cakes are shown in Figure 1. It is obvious that most of the improvement resulted from treatment of the prime starch; the cake baked from the reconstituted flour in which only the prime starch fraction was bleached was about equal to the one in which all fractions were bleached.

In a further investigation prime starch was treated with 3 increments of chlorine gas: (0.1, 0.2 and 0.3 ml.  $\text{Cl}_2/\text{g}$ . which resulted in pH levels of 5.50, 5.24 and 5.03, respectively). The cakes improved in volume and appearance with heavier chlorination of the starch as shown in Figure 2. It will be noted, however, that pH of the reconstituted flours did not fall below pH 5.0, while that of the control treated commercial flour was pH 4.7. This work agrees in part with that of Sollars (8) who concluded that chlorine affected the gluten and prime starch fractions of cake flours, resulting in improvement of baking quality.

### Bleaching Starch with Chlorine

The question logically arises as to the specific action which the chlorine has upon the starch. That particular problem has been under study in the Biochemistry Department of Purdue University, with financial support from this Regional Project.

The initial investigation has been completed, and a paper prepared for publication (9). An attempt was made to establish the mode of attack of chlorine gas on dry or semi-dry wheat starch. There is evidence that the attack is accelerated by the presence of certain ions such as iron, cobalt, or nickel, and by the presence of bromine or iodine. This suggests catalytic influences. Furthermore, it appears that the specific catalytic effect of light should be investigated.

Unde. aqueous conditions of oxidation, Whistler and

Schweiger (10) showed that the predominant oxidative attack of hypochlorite on starch is at carbons C2 and C3 of D-glucose residues. This attack presumably creates a carbonyl group which, in the neutral pH range, enolizes and allows further rapid oxidation to occur at the double bond with consequent cleavage between carbon atoms C2 and C3.

In contrast, chlorine gas oxidizes semi-dry wheat starch rather slowly. The oxidation is temperature dependent, and is markedly affected by the moisture content of the starch; the higher the moisture, the more rapid the oxidation. At the 15.3 percent moisture level, hydrolysis products suggest that the principal attack is on carbon atom C1. Very little oxidation of carbon atom C6 occurred during a seven day period, and there was practically no oxidative attack on carbon atoms C2 and C3. The extensive production of D-gluconic acid at high moistures might suggest that the low pH levels induced hydrolysis of the glycosidic linkages, freeing reducing groups which would rapidly oxidize to glyconic end units. Approximately 42 percent of the chlorine consumed in the oxidation is required for production of the isolated D-gluconic acid.

Since oxidation is extensive at low moisture and even under dry conditions, it is suggested that a second non-hydrolytic depolymerization occurs, possibly induced by a free radical mechanism. If this is so, then it might be expected that the two types of depolymerization would occur simultaneously at intermediate moisture levels. Further studies are in progress.

It is apparent that during chlorine gas bleaching of wheat flour some depolymerization of the starch probably occurs with the formation of D-gluconic acid end units, which in turn may influence the properties of dough or cakes produced therefrom.

### Proportion of Flour Fractions

Another question that arose in the Ohio work was the importance of the level of protein in a flour to its cake baking potential. This led to an attempt to set up a series of flours produced by varying the proportions of the different fractions. The same commercially milled 48 percent extraction, unbleached flour was used as in the study on bleaching. Cake volume was the prime criterion used as a measure of cake quality, though other measurements were also taken, including an overall "cake score".

Design of the experiment presented a problem. The difficulty arose because it is not possible to vary the components independently since they always total 100 percent. After much study and consultation,

CAKES BAKED WITH RECONSTITUTED FLOUR  
BLEACHED FRACTION SERIES

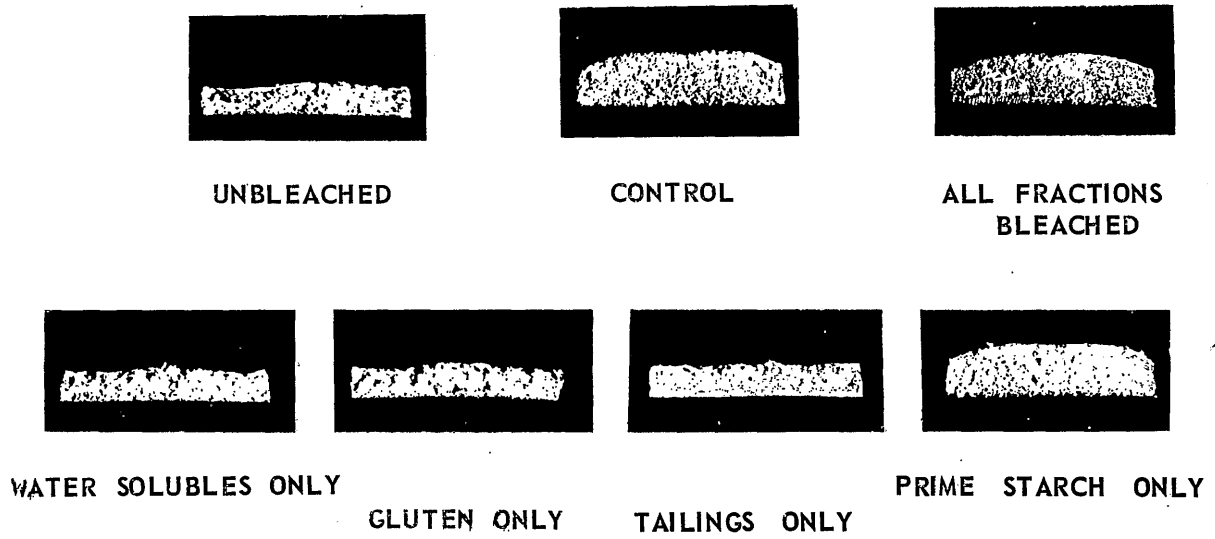


Fig. 1

CAKES BAKED WITH RECONSTITUTED FLOUR  
BLEACHED PRIME STARCH SERIES





				
				CONTROL
FLOUR pH	5.50	5.24	5.03	4.70
VOLUME	486	531	565	555

Fig. 2



W. D. Hanson, formerly of Biometrical Services, ARS, suggested a multiple regression study, and a design of the Box-Wilson multiple response surface type was chosen. Details of the design and the analysis have been given by Donelson and Wilson (3). The essential feature was the choice of three ratios, involving the four variables, such that each ratio could be varied independently. The ratios finally chosen were:

$$X_1 = \text{Water solubles}/(\text{gluten} + \text{tailings} + \text{starch})$$

$$X_2 = \text{Gluten}/(\text{tailings} + \text{starch})$$

$$X_3 = \text{Tailings}/\text{starch}$$

Each ratio could be varied independently, but specification of all three uniquely determined the amount of all four components. The relation between cake volume and flour composition could be approximated by fitting a second order polynomial in three variables; i.e., the familiar Taylor series expansion with all terms beyond the second order considered as a negligible remainder,  $R(x_i)$ :

$$y = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{11}x_1^2 + b_{22}x_2^2 +$$

$$b_{33}x_3^2 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + R(x_i)$$

In this equation, the cross product terms afforded an estimate of interaction, the remainder term showed the adequacy of the second order approximation, and the statistical analysis indicated the degree and location of significance of the various factors.

The design included 22 treatments, and each was replicated. Ranges of flour component percentages were: water solubles, 1 percent to 7 percent; gluten, 5 to 17 percent; starch tailings, 3 to 20 percent; prime starch, 65 to 82 percent. The center point of the Box-Wilson design was the original flour composition: water solubles 4.1 percent, gluten 11.4 percent, starch tailings 12.0 percent, prime starch 72.5 percent. This composition is typical of soft red winter patent flours. The range of each ratio was divided into five equally spaced intervals.

General results are presented in Table 1. The analysis of variance is shown in Table 2.

**TABLE 1.—Treatment Combinations with Corresponding Flour Compositions, Cake Volumes and Scores.**

Variable Coded Value			Flour Composition <sup>a</sup>				Cake Volume Average	Cake Score Average <sup>b</sup>
X <sub>1</sub>	X <sub>2</sub>	X <sub>3</sub>	Water Solubles	Gluten	Starch Tailings	Prime Starch		
			%	%	%	%	cc	
0	0	0	4.1	11.4	12.0	72.5	596	5.0
0	0	0	4.1	11.4	12.0	72.5	587	6.5
-1	-1	-1	2.6	8.5	8.3	80.6	499	5.3
-1	-1	+1	2.6	8.5	16.6	72.3	595	7.5
-1	+1	-1	2.6	14.5	7.7	75.2	566	5.8
-1	+1	+1	2.6	14.5	15.5	67.4	585	7.0
+1	-1	-1	5.6	8.2	8.1	78.1	583	3.8
+1	-1	+1	5.6	8.2	16.1	70.1	596	5.3
+1	+1	-1	5.6	14.1	7.5	72.8	583	5.8
+1	+1	+1	5.6	14.1	15.0	65.3	565	6.5
0	0	-2	4.1	11.4	3.3	81.2	544	4.5
0	0	+2	4.1	11.4	19.1	65.4	574	7.5
0	-2	0	4.1	5.0	12.9	78.0	546	5.5
0	+2	0	4.1	17.0	11.2	67.7	562	6.3
-2	0	0	1.0	11.8	12.4	74.8	631	5.5
+2	0	0	7.1	11.1	11.7	70.1	575	6.5
0	0	-1	4.1	11.4	7.9	76.6	561	5.5
0	0	+1	4.1	11.4	15.7	68.8	607	7.0
0	-1	0	4.1	8.3	12.5	75.1	604	4.8
0	+1	0	4.1	14.3	11.6	70.0	613	6.5
-1	0	0	2.6	11.6	12.2	73.6	629	5.8
+1	0	0	5.6	11.2	11.8	71.4	594	5.5

<sup>a</sup>14% moisture basis.

<sup>b</sup>Higher score preferable.

**TABLE 2.—Analysis of Variance for Cake Volume  
Data of Reconstituted Cake Flours.**

Source of Variance	Degrees of Freedom	Mean Squares
Due to regression	9	
Linear		
X <sub>1</sub>	1	455
X <sub>2</sub>	1	499
X <sub>3</sub>	1	5,208**
Quadratic		
X <sub>1</sub> <sup>2</sup>	1	23
X <sub>2</sub> <sup>2</sup>	1	6,982**
X <sub>3</sub> <sup>2</sup>	1	7,404**
Interaction		
X <sub>1</sub> by X <sub>2</sub>	1	1,958**
X <sub>1</sub> by X <sub>3</sub>	1	3,511**
X <sub>2</sub> by X <sub>3</sub>	1	2,889**
Deviations from regression	11	893**
Error	23	151

\*\*Significant at the 1% probability level

A three-dimensional model was constructed showing changes in cake volume with changes in the variables X<sub>2</sub> and X<sub>3</sub>. Variable X<sub>1</sub> was maintained constant as its median value. This model is shown in Figure 3.

Conclusions drawn from this comprehensive study included the following: 1) An increase in the concentration of the water solubles fraction tended to decrease cake volume, although not greatly. 2) Increase in concentration of starch tailings had a marked effect in increasing volume and improving internal appearance. 3) Small changes of concentration of gluten or prime starch above or below the normal amounts had little effect on volume, but much greater or smaller than normal concentrations of either resulted in smaller cakes. 4) Because of interaction, responses of cakes to changes in the amount of a fraction or fractions varied significantly at different concentrations of the fractions.

The relative proportion or balance of flour components appears to condition the contribution to cake structure of each component and have a significant effect upon the quality of the cake.

#### Flour Fractions from Different Sources

Another approach to the significance of the different flour fractions was to investigate the effect on cake quality of fractions from different sources. For this study two flours were chosen: a good quality soft red winter unbleached cake flour commercially milled to 48 percent extraction, and a poor quality

cake flour from Pawnee wheat, experimentally milled on Allis-Chalmers<sup>1</sup> laboratory equipment to 50 percent extraction. The Pawnee flour was then passed through the Raymond swing-hammer mill twice to obtain a particle size comparable with the high quality commercial sample. The protein content of the commercial flour was 7.6 percent and that of the Pawnee 10.1 percent. The commercial flour was the same one used in the fraction-proportion study.

The experimental design was selected with the aid of C. R. Weaver, the Ohio Station statistician. Essentially it consisted in baking a series of cakes in which the flour fractions were incorporated at the level found in each of the two original flours. In addition, a complete series of fraction interchanges was made up at the same two fraction ratios. All reconstituted flours were bleached, and layer cakes baked, using the lean formula. Results have been published by Donelson and Wilson (4).

The experiment was considered as a 2<sup>5</sup> factorial using as factor pairs the two sources for each of the four flour fractions, and the two levels of interchange of fractions. The total of 32 treatments was baked, each in three replications, each replicate the average of two cakes per batter. The experiment was set up in 12 blocks, each containing eight treatments, and all the cakes in any one block were baked in a single day. Day to day variation, however, was found upon statistical analysis to be unimportant. Results are presented in Tables 3 and 4. Appearance and volume of cakes from the 32 bakes is shown in Figures 4, 5, and 6. Analysis of variance is presented in Table 5.

The contributions to cake volume of the gluten, water solubles, and starch tailings fractions from the good flour designated as Commercial were significantly greater than those of the corresponding fractions of the poor (Pawnee) flour. The effects on gross cake structure of these fractions were likewise superior for the good flour. On the other hand, the prime starch from the Pawnee flour was significantly superior to that of the good flour.

Gluten had the greatest effect on cake volume and structure. Interactions of gluten x composition (relative proportion of flour fractions) and water solubles x starch tailings x composition were highly significant. This indicated considerable dependence of the responses on the concentration of these flour components.

It was suggested that the expression of the quality factors might be dependent on two things: An inherent

<sup>1</sup>Mention in this publication of a trade product, equipment, or a commercial company does not imply its endorsement by the U.S. Department of Agriculture or cooperating agencies over similar products or companies not named.

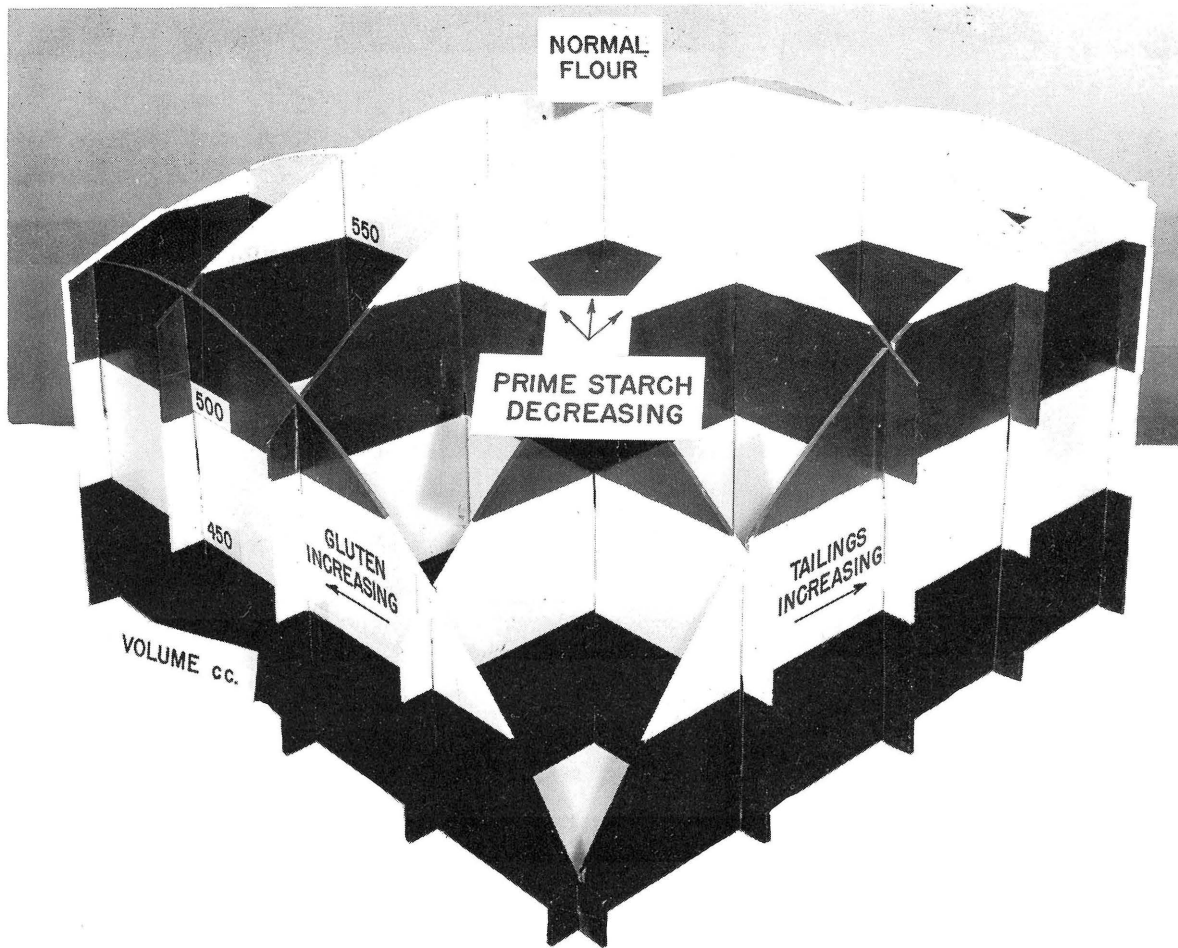


Fig. 3.—Three-dimensional model showing changes in cake volume with changes in variables  $X_2$  (indicated as gluten increasing) and  $X_3$  (indicated as tailings increasing). Variable  $X_1$  is constant at the median value of its range. Variables  $X_2$  and  $X_3$  extend from -2 to +2 (coded values).

**TABLE 3.—Flour Fraction Interchanges, Liquid Levels and Baking Results for Cakes Using Commercial Composition.**

Fraction Composition (percent) <sup>a</sup>						
Water Solubles 3.6	Gluten 11.2	Tailings 9.8	Prime Starch 75.4	Average Cake Volume	Average Cake Score	Liquid <sup>b</sup> Level
				cc.		%
C	C	C	C	604	6.5	103
P	C	C	C	576	5.8	103
C	P	C	C	500	3.5	97
C	C	P	C	596	7.0	103
C	C	C	P	613	6.2	103
P	P	C	C	449	3.0	103
P	C	P	C	516	4.3	109
P	C	C	P	583	5.3	103
C	P	P	C	458	3.0	103
C	P	C	P	496	3.8	103
C	C	P	P	580	5.2	103
P	P	P	C	442	3.0	97
P	C	P	P	541	4.2	103
C	P	P	P	489	3.5	103
P	P	C	P	493	3.3	97
P	P	P	P	438	3.0	103

<sup>a</sup>C = fractions from commercial flour; P = fractions from Pawnee flour.

<sup>b</sup>Based on flour weight; flour at 14% M.B.

**TABLE 4.—Flour Fraction Interchanges, Liquid Levels and Baking Results for Cakes Using Pawnee Composition.**

Fraction Composition (percent) <sup>a</sup>						
Water Solubles 3.7	Gluten 17.6	Tailings 12.3	Prime Starch 66.4	Average Cake Volume	Average Cake Score	Liquid <sup>b</sup> Level
				cc.		%
P	P	P	P	483	3.7	115
C	P	P	P	484	3.5	109
P	C	P	P	557	6.7	115
P	P	C	P	511	3.7	115
P	P	P	C	478	4.2	115
C	C	P	P	522	5.2	121
C	P	C	P	523	5.0	115
C	P	P	C	481	3.7	121
P	C	C	P	539	6.8	115
P	C	P	C	519	5.3	115
P	P	C	C	470	3.7	115
C	C	C	P	556	6.8	115
C	P	C	C	493	4.0	121
P	C	C	C	502	5.3	121
C	C	P	C	500	4.5	121
C	C	C	C	538	6.2	121

<sup>a</sup>C = fractions from commercial flour; P = fractions from Pawnee flour.

<sup>b</sup>Based on flour weight; flour at 14% M.B.

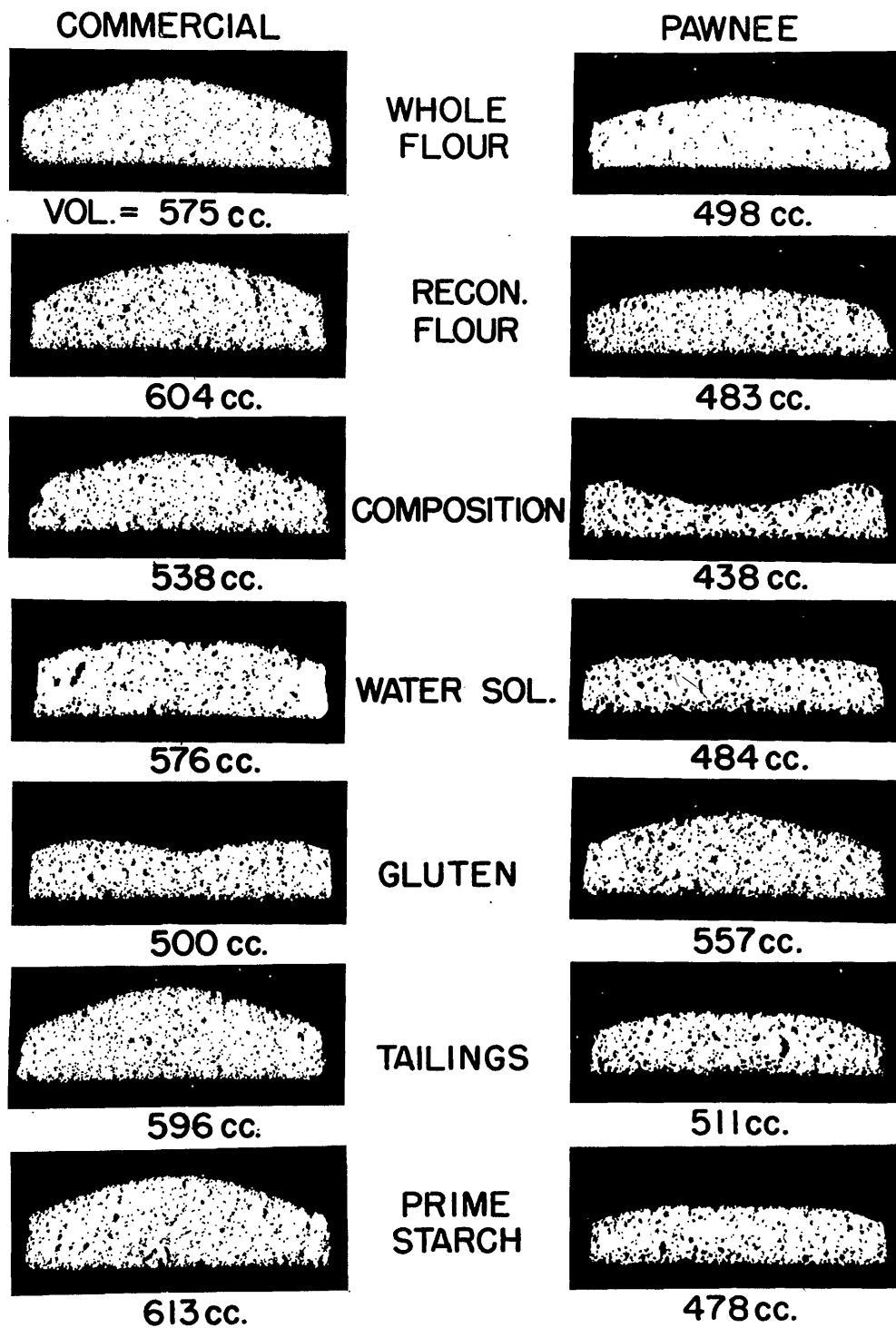


Fig. 4.—Effect of single factor interchanges on cake volume and structure. Left column all commercial components except those interchanged and right column all Pawnee components except those interchanged. Rows 1 and 2 comparison of cakes baked from whole and reconstituted flours. Rows 3 through 7 cakes resulting from single factor interchanges as noted.

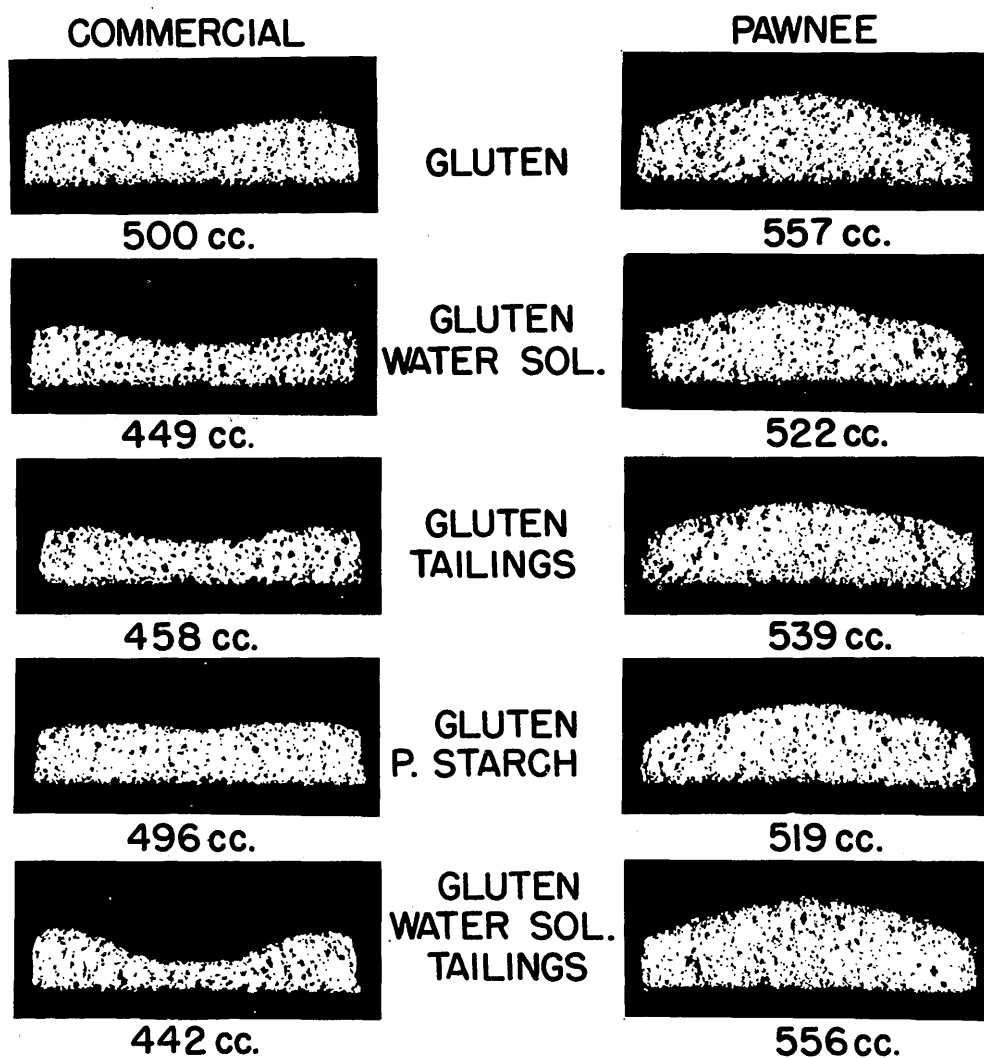


Fig. 5.—Effect of gluten and interaction of other flour components with gluten upon cake volume and structure. Left column, all commercial fractions except for interchanges, and all at the commercial proportions. Right column, all Pawnee fractions except for interchanges, and all in Pawnee proportions.

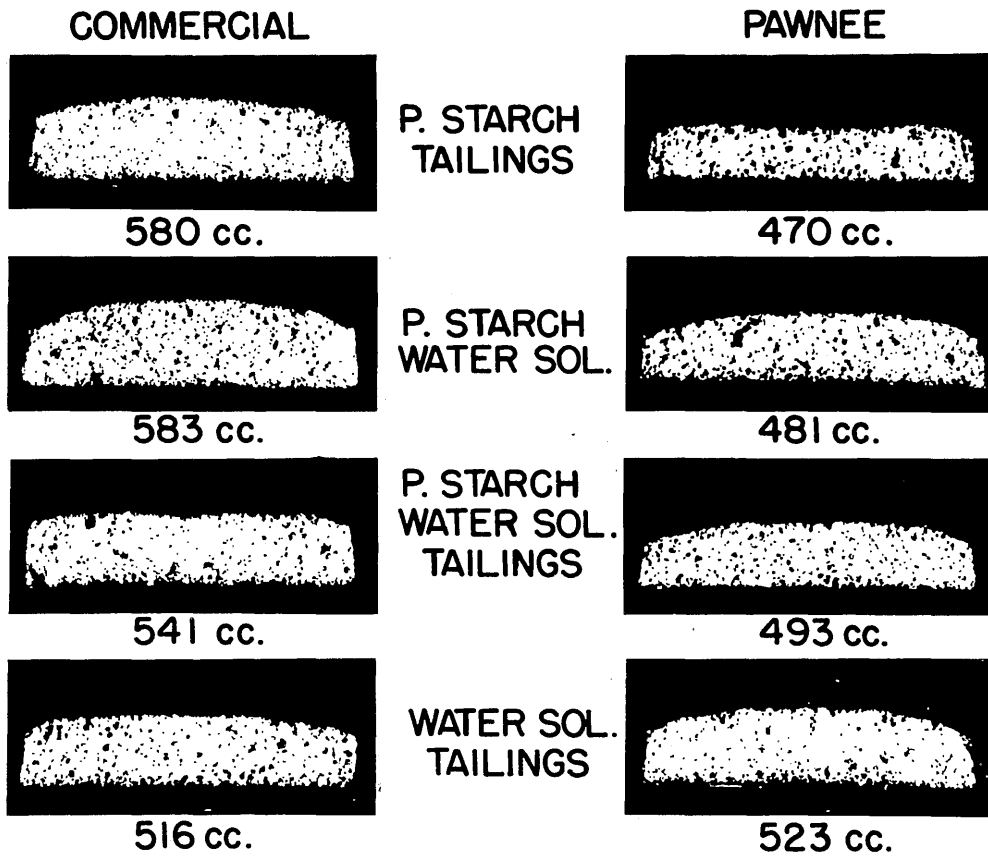


Fig. 6.—Effect of prime starch, water solubles, and tailings interchanges upon cake volume and structure. Left column, all commercial fractions except interchanges, and all at commercial proportions. Right column, all Pawnee fractions except for interchanges, and all at Pawnee proportions.

**TABLE 5.—Analysis of Variance of Cake Volume Data.**

Source of Variation	Degrees of Freedom	Mean Squares
Replication	2	
Blocks	9	
Main Effects		
Water solubles	1	10,626**
Gluten	1	124,704**
Tailings	1	12,331**
Prime starch	1	7,704**
Composition	1	4,428**
Interactions		
Water solubles x composition	1	2,513*
Gluten x composition	1	26,800**
Tailings x composition	1	1,980*
W.S. x tailings x composition	1	4,187**
Combined non-significant interactions	22	358
Error	53	377

\*Significant at the 5% probability level

\*\*Significant at the 1% probability level

quality aspect, and concentration. The first of these would be dependent on the physical and chemical constitution of each fraction, and the latter related to the percentage composition of the flour.

#### Batter Temperatures During Baking

Another aspect of the cake problem which has been investigated is the collecting of temperature data on cake batters during the baking process. A rack of six spaced thermocouples was placed in the batter in various positions, and connected to a strip chart recorder. Continuous records were kept throughout the baking process.

It was found that the cake batter bakes by conduction radially from the outside towards the center, and simultaneously from the bottom upward. Until the batter reached about 75°C., the rate of increase in temperature was almost the same at all thermocouple locations. Between 75° and 90° the rate of change increased markedly. If it may be assumed that the rate of heat transfer into a particular interior portion of the baking batter decreases because of lower conductivity of the set batter exterior to it, then the more rapid temperature rise may indicate that in the temperature range between 75° and 90° an exothermic reaction occurs. Temperatures rise also because of the decreased heat transfer through the set batter exterior to the zone of thermal activity. The rate of temperature rise is more marked in batters made up on only flour and water and also increases with higher liquid levels.

Brabender Viscograms obtained with cake batters

may be divided into four characteristic stages: 1) A decrease in viscosity during the warming range from 25°-55°C. 2) an increase in batter viscosity between 55° and 75°, apparently due mainly to increased volume resulting from the leavening action. 3) an induction stage 75°-85° during which rate of water evaporation increased, significant starch gelatinization occurred, with perhaps concurrent protein denaturation. It is suggested that there is an intense competition between the hydrophilic batter components for free water during this stage. 4) the setting stage, with rapid thickening, occurred above 85°.

#### Liquid Level and Cake Quality

Since layer cakes are sensitive to liquid content, a systematic study (11) was made of the effect of water level on cake structure. A number of commercially milled and improved cake flours, and laboratory milled flours treated with chlorine gas to pH 4.6-4.8, were employed in baking by a standard white layer formula and the lean formula. For each flour seven equally spaced liquid levels, at 6 percent intervals, were selected to bracket the optimum level, which was estimated from preliminary bakes.

Three types of data were collected: 1) cake volume, 2) layer structure score, and 3) layer contour score. To permit quantitative treatment of the data, numerical values were assigned to the structure and contour scores. Standard curvilinear regression methods were applied, and second order regression equations fitted.

It was found that in all cases regression accounted for well over 90 percent of the variance, and therefore the quadratic form was considered adequate to represent the data. From the equations, the liquid levels for maximum volume and optimum structure could be obtained.

It was clearly demonstrated that all three measurements on the cakes changed regularly as functions of liquid. With increasing amounts of water, the top contour changed progressively from deeply sunken to rounded to peaked. At the same time, crumb structure changed from dry and coarse with thick-walled, open, irregular cells and a broad distribution of cell sizes, to a tight, uniform distribution of small thin-walled cells, and then to a compact very moist texture. Layer volume increased rapidly with liquid level to maximum, then decreased with excess liquid.

Figure 7 shows cakes baked using the full formula for one of the flours at the seven moisture levels and Figure 8 shows cakes from the same flour using the lean formula. It should be noted that the sunken cakes did not fall; they never rose. Peaked cakes resulted when the layer rose to a large volume in the oven, followed by settling of the material toward the pe-



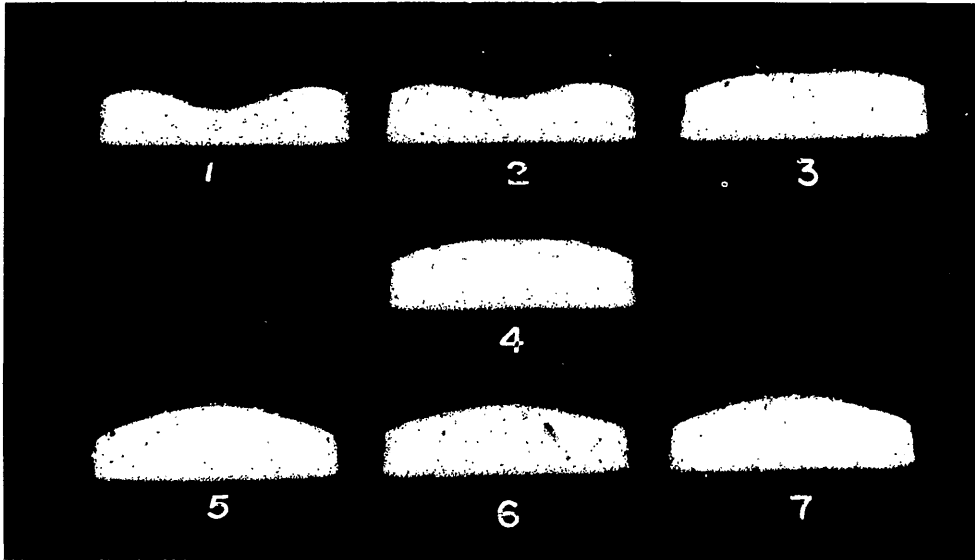


Fig. 7.—Full-formula cakes. Increasing numbers indicate higher absorption levels, the optimum being cake No. 4.

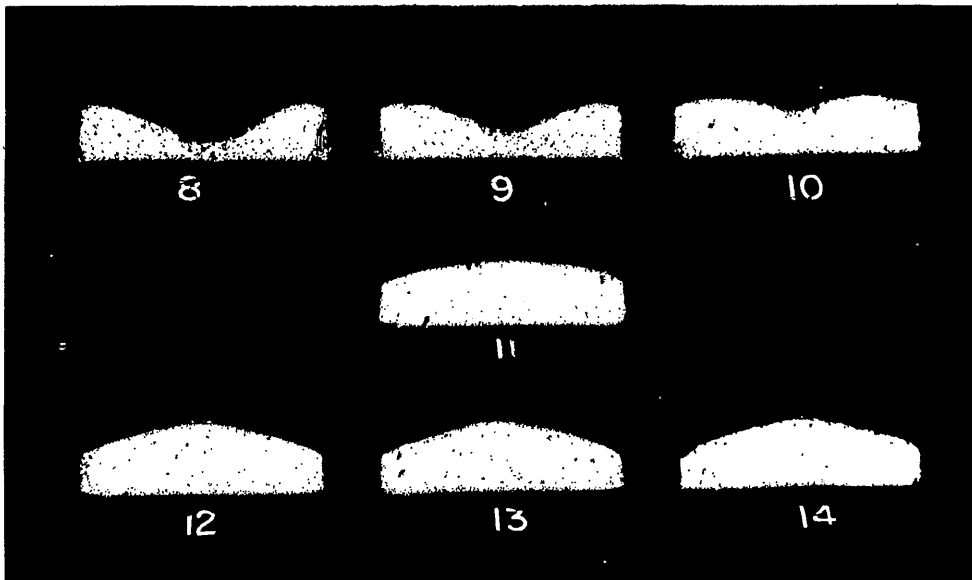


Fig. 8.—Lean-formula cake. Increasing numbers indicate higher absorption levels, the optimum being cake No. 11.

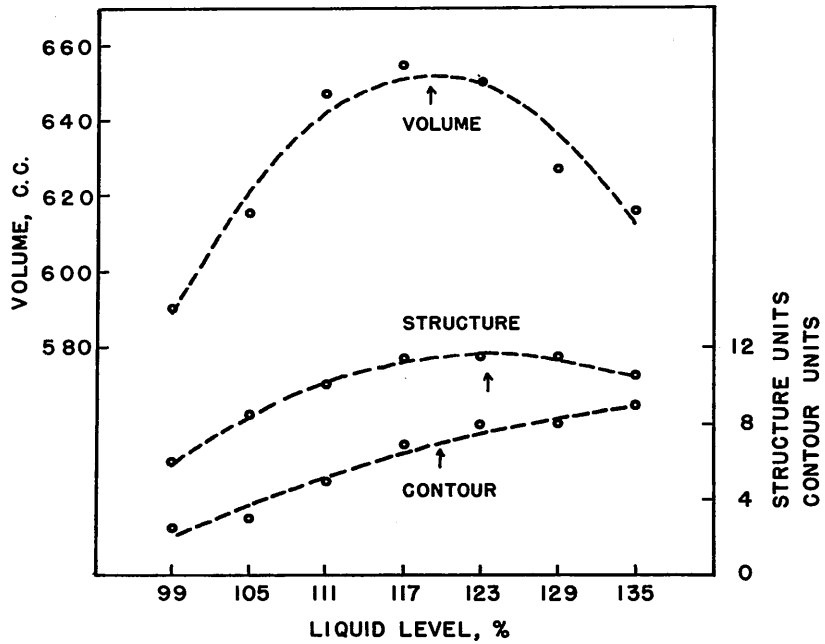


Fig. 9.—Graphs of volume, structure and contour score of white layer cakes baked with a full formula at different liquid levels. The curves are best fitting second order polynomials for the data. Arrow beneath each curve is the estimated liquid level for optimum response.

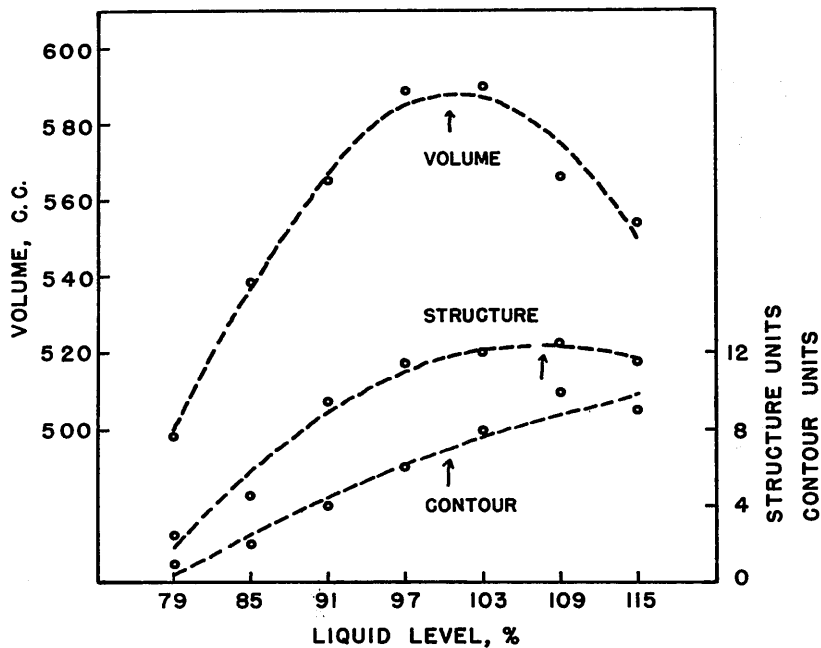


Fig. 10.—Graphs of volume, structure and contour score of white layer cakes baked with a lean formula at different liquid levels. The curves are best fitting second order polynomials for the data. Arrow beneath each curve is the estimated liquid level for optimum response.

riphery of the pan. None of the cakes "fell" in the ordinary sense of the term.

Graphs of the volume, structure score and contour score for a representative commercial cake flour in the full formula are shown in Figure 9 and similar graphs for the same flour in the lean formula in Figure 10. The fitted curves for volume were parabolic downward and optimum liquid level occurred at slope zero. Structure score yielded similar curves. Contour score, however, was in general monotone, increased without a maximum, and was not markedly curved. The general shape of the layers and the internal structure were similar at the optimum liquid level of the two recipes, but changes in liquid had a more marked effect with the lean formula. Full formulation required 16 to 18 percent higher liquid level for optimum volume because of the inclusion of milk solids and egg protein. For the same reason, this formula gave consistently larger cake volume. There were differences between flours in sensitivity to liquid level. For each flour optimum liquid levels for volume and contour appeared to be nearly identical within each experiment, but the liquid level for best structure score lay at a somewhat higher value, so that the best structure appeared in slightly peaked cakes.

The standard full formula, with its supplementary ingredients, minimized individual flour differences and produced greater product uniformity. The lean formula therefore appeared superior as a means of detecting real differences between flours.

#### Viscograph Studies of Starch Pasting

A thermoanalytic method of studying starch pasting was developed for use with the Brabender Viscograph. Analysis of viscograms obtained with potato, corn, and wheat starches and wheat flour in citrate buffer indicated that there was regularity and precision to the viscosity-concentration relationship and that a temperature dependent term was involved. An empirical equation was developed, relating viscosity to starch or flour concentration and slurry temperature. Reaction rate equations were worked out on the assumption of a first order rate process. Preliminary analysis yielded apparent activation energies for wheat starch in the order of 90 K. Cal. per mole. However, it was estimated that the free energy of activation was in the range of normal chemical reaction, and the very large entropy term indicated an extreme change from a highly ordered structure to a randomized configuration.

In another set of experiments, the effect of chlorine treatment of prime starches prepared from hard and soft wheat flours was followed by viscograms. As chlorine dosage increased, it was noted that: 1) The initial viscosity rise occurred at a lower temperature,

2) there was an accelerated rate of viscosity change with temperature, 3) a higher maximum viscosity was attained.

A series of tests made with these starches five weeks after the original chlorination showed a further increase in intermediate viscosity but much lower viscogram peaks, and peaks were reached at several degrees lower temperatures than with freshly treated samples. The pH had decreased only 0.1 to 0.3 units during the aging period, but apparently considerable physical change had occurred. Possibly chlorine "tenderizes" the starch by reducing the energy of certain of the intramolecular bonds, thus effecting a shift of lability to lower temperatures. Bonds not before accessible may be brought to lower energy levels, permitting a greater degree of granule swelling.

#### Flour Lipids

It appeared possible that flour lipids might have an influence on cake flour quality, despite the fact that only relatively small amounts are present. An unbleached, commercially milled cake flour was studied. A series was prepared including: 1) a control, 2) a flour wetted with water-saturated N-butanol, 3) a flour extracted with the butanol, the fats recovered and added back to the defatted sample, and 4) a defatted flour. Part of each sample was then treated with chlorine gas, and cakes baked by the Kissell lean formula (6) using the bleached and unbleached materials. Cake volumes are given in Table 6.

The normal improvement from bleaching was evident except in the defatted flour. There was no marked effect from merely wetting the flour with the solvent (N-butanol). When lipids were removed and replaced, baking performance was not greatly affected. However, a drastic change was evident when the flour was defatted. A portion of the effect may have been due

TABLE 6.—Volumes of Lean Formula Cakes Showing the Effect of Lipid Removal from Cake Flour.

Treatment	Cake Volume	
	Flour Unbleached	Flour Bleached
Control	cc. 438	cc. 584
Exposed to solvent	428	566
Lipids removed and replaced	479	559
Defatted	336	314

to presence of the solvent and to the various manipulations; but allowing for this, removal of the fatty components from the flour appeared to account for much of the radical decrease in volume. The subject needs further study.

### Air-Classification of Flours

Air-classification of flours is a relatively new development of the milling industry. It consists essentially of an added operation. Flours produced by roller mills are subject to a particle-size separation, and the resulting fractions combined in the proportions necessary to meet the baker's specifications.

In 1959, the Soft Wheat Quality Laboratory purchased a Type T-11 Laboratory Model Air Turbo-Separator built by the Pillsbury Company. A wide range of separations can be made through adjustment of five variables: 1) Velocity of the rotor unit, 3500 or 5850 r.p.m. 2) disposition of the classifier blades, forward or backward, 3) selection of the number of classifier decks, from one to six, 4) adjustment of the louver curtain angle at 10° or 35°, and 5) rate of feed, adjustable from 25 to 100 pounds of flour per hour.

For the preliminary work, a four-stage operation was used. At each stage the flour was separated into two fractions, fine and coarse. The coarse fraction was then reclassified at the next stage setting. Upon completion of the schedule, the flour had been separated into five fractions, progressively coarser in size. A commercially milled, unbleached straight grade soft wheat flour was classified using this procedure. The results shown in Table 7 are typical for soft wheats.

When wheat is milled, the endosperm is separated more or less completely from the bran and germ and the particles are reduced in size. The endosperm consists of starch granules of varying size, embedded in a more or less continuous matrix of proteinaceous material. Some endosperm cell wall material is also present. In general, the finest particles of the flour are largely protein material, the next larger particles are free starch granules, and the coarser granules of chunky endosperm are masses of starch grains still embedded in the protein matrix. The separation used gave a low yield of a very high protein fraction, a much higher yield of the next three separates, one moderately higher in protein, the other two much lower than that of the original flour, and the final fraction not too different from the parent material in protein content.

Moisture figures show that there was some drying of the flour during processing, and the finer the parti-

**TABLE 7.—Analytical and Baking Data on Classified Separates of a Straight Grade Soft Wheat Flour.**

Flour	Yield	Moisture	Protein <sup>a</sup>	Ash <sup>a</sup>	Cake Volume
	%	%	%	%	cc.
Parent	100.0	13.0	8.8	.37	564
1st fine	5.1	10.6	25.7	.43	491
2nd fine	21.6	10.7	14.3	.39	590
3rd fine	22.4	11.8	4.8	.33	619
4th fine	21.8	11.8	4.3	.34	583
4th coarse	24.4	11.9	9.0	.43	522

<sup>a</sup>14 percent M. B.

cles the greater the loss of water. The range of ash was not very great. Baking results with the Kissell formula, Table 7, indicate that the first fine and fourth coarse cuts produced cakes inferior to the original material, but the other three fractions baked superior cakes.

The distribution of the protein of the original flour among the fractions is of particular interest, and it seemed desirable to devise some means of expressing the changes mathematically. The change in protein content with the removal of the finer particles has been designated as the protein shift, expressed as:

$$\frac{(\% \text{ prot. of fraction minus } \% \text{ prot. of parent flour}) (\% \text{ yield of fraction})}{(\% \text{ prot. of parent flour})}$$

The formula is particularly useful in comparing the behavior of different flours. Figure 11 illustrates the shift graphically. The area of the larger rectangle in the top diagram represents the amount of protein of the straight grade flour. The hatched area parallel to the abscissa represents the protein removed from the parent flour upon classification and transferred to the appropriate fines fraction. Thus, the hatched area along the ordinate represents the additional amount of protein in the fine fraction, or the protein shift. The protein content of the parent flour was 8.8 percent in this example. Thus, the protein shift was calculated to be 13.5 percent. A similar graph for the results at the second cut setting is given in the lower diagram.

For research purposes it was deemed highly desirable to learn something about the size and size distribution of particles in the various air-classified fractions. A Coulter Counter was acquired with NC-30 funds for this purpose. A great deal of preliminary study was necessary to determine the most advantageous settings to be used, to check replicability, and to ascertain whether the particle size as determined agreed with results by other methods, such as sedimentation rates, or direct microscopic counts.

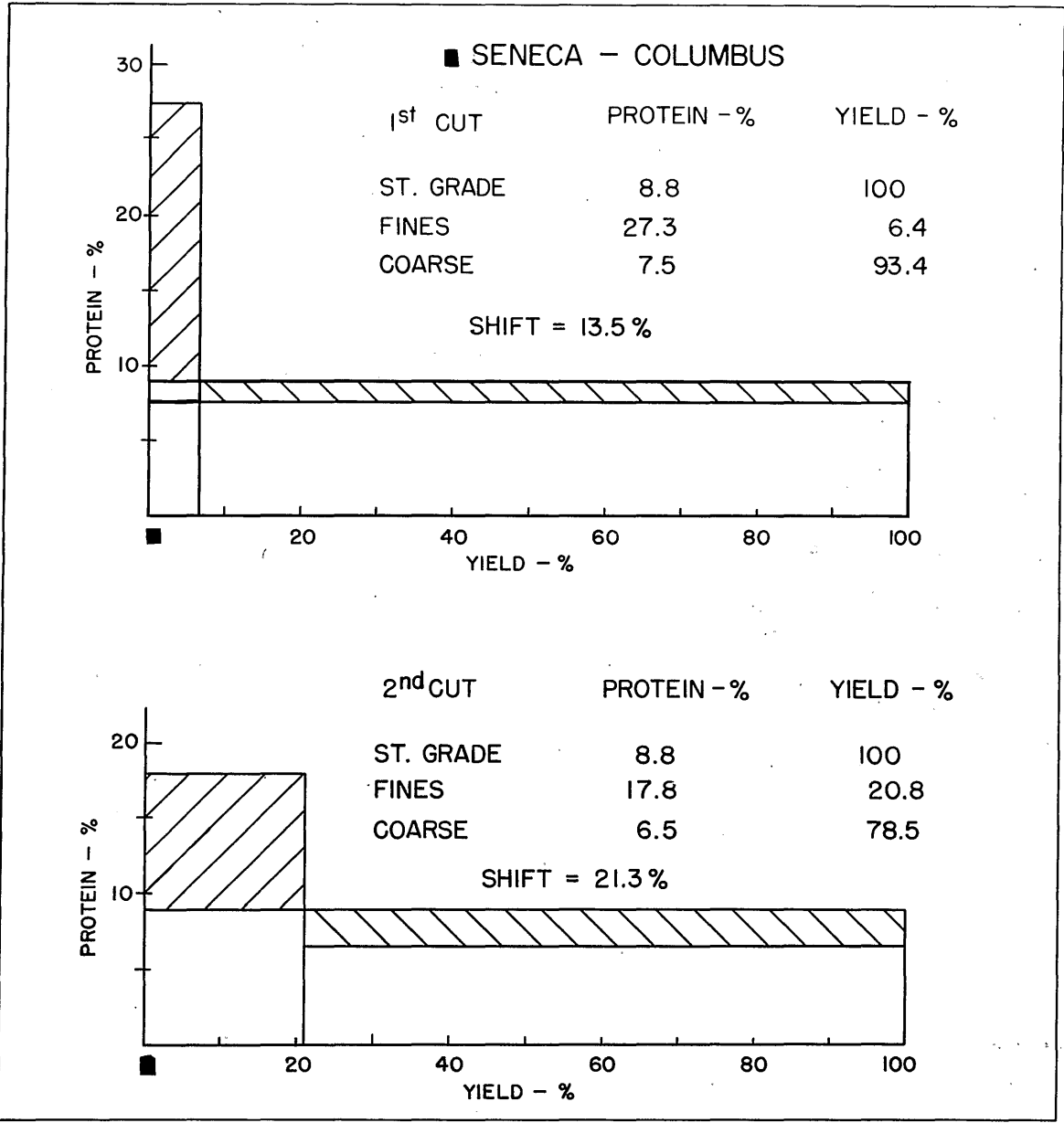


Fig. 11.—Histograms showing protein shift of 1st and 2nd cut air-classified Seneca flour.

The Coulter Counter is claimed to give a valid estimate of particle volume, independent of shape. Because starch granules are not true spheres, measurement of one diameter cannot give the true volume. However, Equivalent Sphere Diameters (ESD) may be estimated. It was found that particle-size distributions of the air-classified wheat starch fraction fitted logarithmic normal distributions extremely well. In this distribution, the frequency  $f$  with which a particle of diameter  $d$  occurs is

$$f(d) = \frac{\sum n}{\sqrt{2\pi} \ln \sigma_g} \exp \left[ -\frac{(\ln d - \ln M)^2}{2 \ln^2 \sigma_g} \right]$$

where  $M$  is the geometric mean diameter and  $\sigma_g$  is the standard deviation. In effect, transforming the particle size to the logarithmic form reduces the data to the ordinary normal distribution.

It developed that there was a bimodal distribution of particle size in prime starch, with the overlap point at about 8-10 microns. Both sets of starch data followed log-normal distributions. It is interesting to speculate on how such distributions arise, but there is no clear evidence to support any specific hypothesis at present.

While several papers have been published (1) (5), more work is needed on the effect of wheat variety, location, and season on the characteristics of samples insofar as their air-classification is concerned. One

study has been completed on a number of soft and one semihard wheat, Purkof. Samples were grown at Aberdeen, Idaho, Columbus, Ohio, and East Lansing, Michigan. All were from the 1959 crop. Each sample was Buhler milled, given one pass through the Raymond mill, and then blended. Each of the samples was then air-classified at the second ( $12\mu$ ) setting that separated them into fine and coarse fractions. Table 8 presents these data.

The soft wheats consistently had a higher yield of the fine fraction and higher protein in the fine fraction. The typical soft wheat varieties Fairfield and Thorne showed the greatest protein shift. Particle size measurements of the parent straight grade flours showed a smaller particle size for the soft wheats. However, in the fine fractions, Thorne and Purkof had similar particle sizes; in the coarse fractions, Thorne had smaller average particle size.

#### Work in Progress

In Ohio, a number of problems are being attacked under NCM-28, and work is expanding rapidly in the air-classification studies. At Purdue, the chlorine-starch investigations are continuing. At the Missouri Station, studies are underway on the nature of wheat proteins. Publications reporting results will appear from time to time.

TABLE 8.—Yield and Protein Shift of Classified Flours.

Variety	Location	Protein Straight Grade %	Yield %	Fines		Coarse		
				Protein %	Protein Shift %	Loss %	Yield %	Protein %
Purkof	Aberdeen	8.3	14.8	12.7	7.8	0.7	84.6	7.6
Fairfield	Aberdeen	8.2	18.9	15.7	17.3	0.6	80.5	6.1
Thorne	Aberdeen	8.7	19.0	17.0	18.1	0.5	80.5	6.8
Blackhawk	Aberdeen	9.9	20.4	16.3	13.2	1.2	78.4	8.2
American Banner	Aberdeen	8.6	19.4	15.3	15.1	0.3	80.2	6.8
Purkof	Columbus	8.9	15.8	12.8	6.9	0.6	83.6	7.9
Fairfield	Columbus	7.6	20.3	14.7	19.0	1.3	78.4	5.6
Thorne	Columbus	8.4	20.6	16.1	18.9	0.7	78.7	6.2
Blackhawk	Columbus	9.8	19.2	17.0	14.1	1.1	79.7	7.9
Purkof	E. Lansing	7.9	18.4	12.5	10.7	0.7	80.9	6.9
Fairfield	E. Lansing	7.1	20.6	14.4	21.2	0.8	78.7	5.2
Thorne	E. Lansing	7.5	20.0	14.5	18.7	1.5	78.5	5.4
Blackhawk	E. Lansing	9.2	20.9	16.1	15.7	0.8	78.3	7.2

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## PROPOSED PUBLICATIONS

### OHIO

- Bode, C. E., Sipes, C. R., and Heizer, H. K. Air-Classification and Particle Size Distribution of Experimentally Milled Soft Wheat Flours (Submitted To *Cereal Chem.*)
- Donelson, D. H., Wilson, J. T., and Sipes, C. R. Effect of Variety and Air-Classification on Cake Flour Quality.
- \_\_\_\_\_, \_\_\_\_\_, and \_\_\_\_\_. Distribution of Chlorine in Bleached Cake Flours.
- Wilson, J. T., and Donelson, D. H. Studies on the Dynamics of Cake Baking. I. The Effect of Liquid on Layer Cake Structure (Submitted to *Cereal Chem.*)
- \_\_\_\_\_, and \_\_\_\_\_. II. Time-Temperature Relations in Cake Baking.
- \_\_\_\_\_, and \_\_\_\_\_. III. Consistency Changes in Heated Cake Batters.
- \_\_\_\_\_, and \_\_\_\_\_. Thermoanalytic Study of Starch Pasting.
- \_\_\_\_\_, and \_\_\_\_\_. Interaction of Chlorine and Liquid in Formation of Layer Cake Structure.
- Wilson, J. T., Donelson, D. H., and Sipes, C. R. Air-Classification of Reduced Chunky Endosperm.
- Wilson, J. T., and Sipes, C. R. Particle-Size Distribution of Wheat Starches.
- \_\_\_\_\_, and \_\_\_\_\_. Specific Surface of Air-Classified Flour Fractions and the Ratio of Flour Surface to Starch Granule Surface.
- \_\_\_\_\_, and \_\_\_\_\_. Comparison of Flour Particle-Size Distribution Measured by Electrolytic Resistivity and Microscopy.

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