

# Rheological Properties of Water-Extracted Air-Classified Spring Wheat Flours<sup>1</sup>

W. C. SHUEY, U. S. Department of Agriculture, and K. A. GILLES<sup>2</sup>, North Dakota State University, Fargo

## ABSTRACT

Four flours milled from two spring wheat varieties (Selkirk and Justin), and at two protein levels were air-classified into three fractions. The air-classified flours were extracted at 10:1 water-to-flour ratio (v./w.). The "initial water-solubles" (IWS) extracted at 10:1 ratio were further partitioned into two fractions with water by extracting the freeze-dried IWS at a ratio of 5.5:1 (v./w.). The residue of the second partitioning, designated as "precipitate" (P) fraction, contained constituents that reduced the mixing time of the extracted flour. The effect of P in reducing the mixing was similar regardless of origin of the flour, i.e., flours ground from different spring wheat varieties, or air-classified flour fractions from those flours. The quantity of material extracted, however, was dependent on the air-classified fraction and related to the physical and chemical characteristics of the flour.

Since the mid-1950s, air-classifiers have become important devices for separating flour according to particle size and density (1). The fine grinders and classifiers, such as described by Behrens (2), effectively separate flour into fractions with varying protein contents. Gracza (3) defined this change in protein content of the flour fractions as degree of protein shift. The studies of Peplinski et al. (4) demonstrated that the physical characteristics of wheat classes and varieties influence the protein shift.

Bloksma (5,6) concluded that rheological properties of a dough were important in determining the quality of the baked product, but understanding the relation between rheological properties and chemical and physical structure of dough to baking quality was inadequate. Gracza (7) concluded that there was considerable variation in the amount of mixing energy required among different air-classified flour fractions; but the optimum mixing energy generally increased with higher protein content. Kaminski and Halton (8) studied washed gluten from air-classified flour fractions with a 'research' extensometer, and showed that the fraction from the 35 to 63  $\mu$  particle-size fraction had the highest rate of stretching, while those fractions under 35  $\mu$  particle size had the lowest rate of stretching. However, they suggested that the differences might be due to state of oxidation of the fractions rather than strength.

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<sup>2</sup>Respectively: Research Food Technologist, North Central Region, Agricultural Research Service, U.S. Department of Agriculture; Professor and Vice President for Agriculture, North Dakota State University, Fargo, Dakota State University, Fargo 58102.

Fortmann et al. (9) studied some factors which influence the work required for mixing a dough and noted that adding gliadin to a dough decreased the power requirement. Ponte et al. (10), Gracza (7), and others have shown that air-classified fractions from the same parent flours, as well as the same air-classified fraction from different flours, vary in their mixing characteristics.

Jones and Dimler (11) observed that gluten separated from high- and low-protein air-classified flours gave essentially identical SGUE patterns. Mullen and Smith (12) obtained similar electrophoretic components for glutes from a short- and long-mixing flour. The differences observed were attributed to those noted between hard red spring and hard red winter wheats. Bietz and Rothfus (13) demonstrated amino acid sequence differences between gliadin and glutenin, although several peptides were common to digestion for both proteins. Williams and Butler (14) extracted flours ground from wheat species with the same and different chromosome numbers. They concluded that the Maes extraction procedure with differential solubility, in conjunction with disc electrophoresis, was better for identifying genotypes than differentiating baking and rheological properties between flours.

Přihoda and Bushuk (15) noted that 2% vital gluten added to a flour changes the absorption but not the farinogram. In the meantime, Lee and MacRitchie (16) found small variations in molecular weight of material derived from gluten would change the dough characteristics of a flour to which they were added. Smith and Mullen (17) concluded that the mixing response of a dough under different atmospheres with changes in pH values may be attributed to structural changes. Bloksma (18) stated, "...that there is no unequivocal relation between the total SH content and various rheological properties." Only a fraction of the SH and SS groups are rheologically effective and their number is much less than the chemically active ones.

This study, involving the water-soluble proteins derived with a 10:1 water-to-flour ratio from air-classified spring wheat flours, had the following objectives: 1) To determine whether or not there were qualitative differences in water-soluble fractions derived from various air-classified flour fractions, and 2) to determine whether or not there were qualitative differences in the water-soluble fractions isolated from air-classified flour fractions of two spring wheats with different mixing tolerances.

## MATERIALS AND METHODS

### Samples

Four hard red spring wheat samples from the 1963 crop were used in this study. Two were pure varieties of Selkirk and Justin each grown at two different locations, namely, Selkirk-C and Justin-C at Casselton, North Dakota; Selkirk-WP at Wolf Point, Montana; and Justin-W at Warren, Minnesota.

### Milling

The wheat samples were cleaned on an Emerson Dockage Tester (Hart Carter Co., Minneapolis, Minn.), and a modified Laboratory Forster Scourer, Model 6 (Forster Mfg. Co., Ada, Okla.). Twenty pounds of the clean, dry wheat samples was tempered to 15.5% moisture for 16 hr. prior to milling in a Miag Multomat Mill (Miag, Braunschweig, West Germany).

### Air Classification

The flour samples were ground in an Alpine Kolloplex Laboratory Mill, Model 160Z (Alpine American Corp., Natick, Mass.) at 14,000 r.p.m., and air-classified into three fractions in an Alpine Microplex, Model 132 MP. The air-classification flow diagram is shown in Fig. 1.

### Extraction Procedures

A Red Devil Paint Conditioner, Model 30 (Red Devil Tools, Union, N.J.), was adapted to hold a 2-liter jar (19). Flour aqueous suspensions of 1,800 ml. of distilled water and 180 g. of flour were shaken for 10 min. The suspensions were

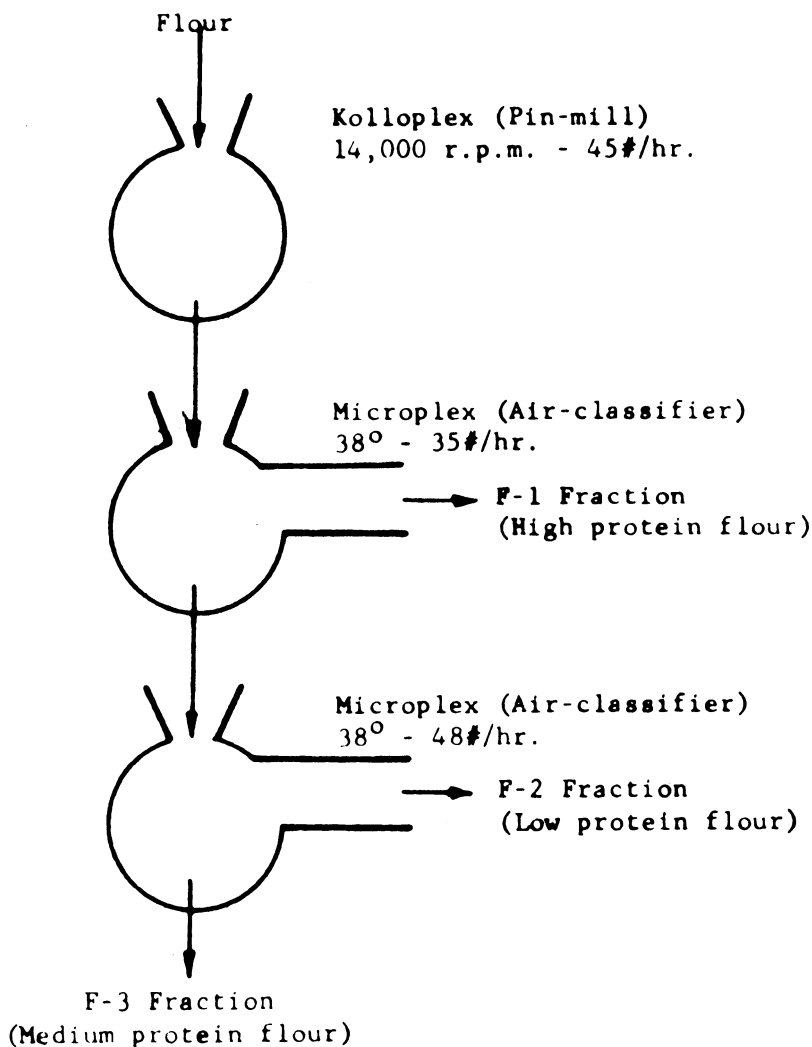


Fig. 1. Air-classification flow diagram.

poured into chrome-plated centrifuge cups and centrifuged for 30 min. at  $500 \times g$ . The supernatants were decanted and the residues were returned to the jar. For the second extraction, sufficient distilled water was added to equal the original volume of the suspensions and the process was repeated.

The supernatants and residues were freeze-dried. For each sample, the dried supernatants were combined and are referred to as the "initial water-solubles" (IWS); the dried residues are referred to as the "solids" (S). (See Fig. 2.) A portion of the dried IWS was extracted again twice using a ratio of 5.5:1 of distilled water to solids for both extractions. The appropriate amount of solids and water was placed in a 250-ml. flat-bottom centrifuge bottle and shaken for 30 min. on a Burrell shaker (Burrell Corp., Pittsburgh, Pa.), centrifuged at  $815 \times g$  for 45 min., and decanted. For the second extraction, water was added to equal the initial volume of the slurry and the process was repeated. The supernatants were combined and freeze-dried as well as the residue. The dried supernatants are referred to as the "water-solubles" (WS), and the residues referred to as the "precipitate" (P). (See Fig. 2.)

#### Starch-Damage Procedure

Starch damage was determined by the iodine absorption method of Medcalf and Gilles (20).

#### Farinograms

The farinograms were determined by the 80 g. constant dough weight method (21).

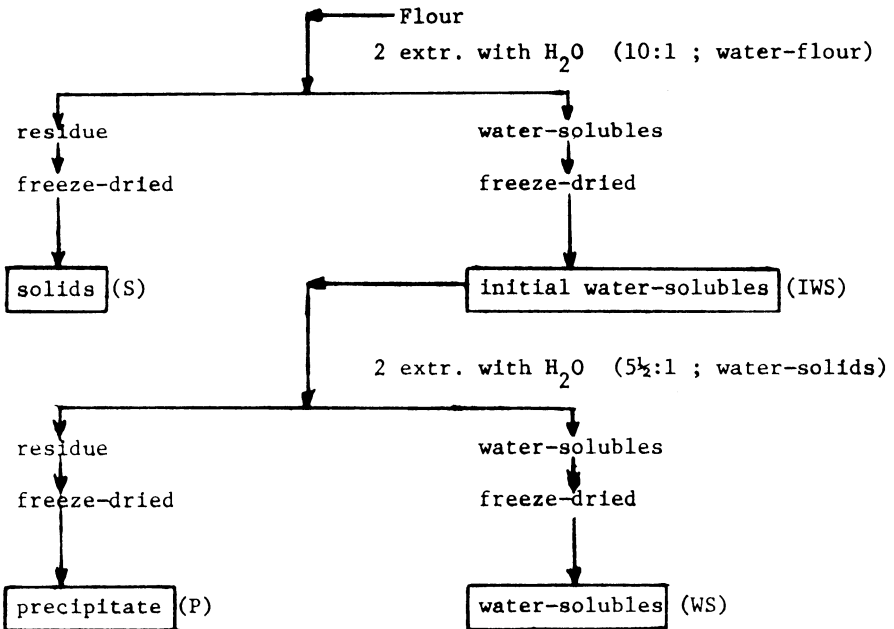


Fig. 2. Flour fractionation procedure.

TABLE I. MILLING DATA

Sample	Ext. <sup>a</sup> %	Patent <sup>b</sup> %	Yield <sup>c</sup> %	Flour Protein <sup>d</sup> %	Ash <sup>d</sup> %
Selkirk-C	66.4	91.9	72.2	12.7	0.434
Selkirk-WP	62.6	89.0	70.4	15.1	0.357
Justin-C	61.6	88.7	69.5	13.9	0.361
Justin-W	59.5	87.9	67.7	16.5	0.361

<sup>a</sup>Percent wheat converted to patent flour on total products basis.

<sup>b</sup>Percent of total flour produced which was patent flour.

<sup>c</sup>Percent of wheat converted to flour (patent plus clear flours) on total products basis.

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

### Mixograms

The mixogram was determined with 30 g. of flour and 20 ml. of water. The sensitivity spring setting was set at 10. All mixograms were run with constant weight of flour and volume of water. Absorptions reported were adjusted according to the height of the mixogram.

### Baking Procedure

A straight-dough baking procedure was used. The formula was:

100 g. flour (14% m.b.)	3 g. NFDM
2 g. salt	3 g. yeast
5 g. sugar	2 g. (melted Crisco)
0.2 g. malted barley flour	

Enough water was added for proper consistency and the dough mixed in a 100-g. National Manufacturing (Lincoln, Nebr.) special mixer. The doughs were fermented at 86°F. (30°C.) for 3 hr. with first punch at 105 min. and second punch at 45 min. Thirty minutes after the last punch, the dough was sheeted and panned, proofed for 55 min. at 86°F., and baked for 20 min. at 430°F. (221°C.). Volume of the bread was determined by rapeseed displacement.

## RESULTS AND DISCUSSION

The data are given in Table I for the flour samples studied. There was approximately a 3% spread in protein content for each variety between locations.

Each flour sample was classified into three fractions (Fig. 1): the fines (F-1) containing the highest protein content; the medium fines or middle cut (F-2) containing the lowest protein content; and the coarse fraction (F-3) containing a protein content approximately the same as the initial flour. The analytical and physical data for the air-classified flours are given in Table II.

The data obtained on the air-classified flour fractions reveal a wide range in the results among fractions for a given sample. There were, however, similarities in the results for each sample series. The particle size increased from F-1 to F-2 to F-3, while the starch damage and ash decreased.

TABLE II. PHYSICAL AND ANALYTICAL DATA FOR AIR-CLASSIFIED FLOURS

Sample	Fraction	Ext. %	Ash <sup>a</sup> %	Protein <sup>a</sup> %	Particle Size		S.D. <sup>a</sup> %	Protein Shift <sup>b</sup> %
					Fisher $\mu$	M-S-A $\mu$		
Selkirk-C	F-1	21.3	.611	20.4	4.4	12.5	39.3	...
Selkirk-C	F-2	15.2	.409	8.7	11.5	21.8	24.8	...
Selkirk-C	F-3	63.5	.366	12.4	19.2	35.0	10.8	...
Selkirk-C	Cum. <sup>c</sup>	100.0	.426	13.3	...	...	...	8.9
Selkirk-C	Orig. <sup>d</sup>	...	.434	12.7	...	...	...	...
Selkirk-WP	F-1	17.1	.615	18.2	4.3	12.5	34.1	...
Selkirk-WP	F-2	15.9	.314	10.4	12.4	24.3	22.4	...
Selkirk-WP	F-3	67.0	.267	15.7	27.0	49.0	7.2	...
Selkirk-WP	Cum. <sup>c</sup>	100.0	.335	15.2	...	...	...	4.5
Selkirk-WP	Orig. <sup>d</sup>	...	.357	15.1	...	...	...	...
Justin-C	F-1	19.9	.572	19.9	4.8	11.5	40.6	...
Justin-C	F-2	15.1	.318	9.1	10.0	21.5	25.7	...
Justin-C	F-3	65.0	.284	13.1	19.5	38.0	10.7	...
Justin-C	Cum. <sup>c</sup>	100.0	.348	13.8	...	...	...	7.3
Justin-C	Orig. <sup>d</sup>	...	.361	13.9	...	...	...	...
Justin-W	F-1	19.9	.581	22.3	3.9	11.0	36.1	...
Justin-W	F-2	14.1	.331	9.9	9.5	20.8	24.9	...
Justin-W	F-3	66.0	.301	15.7	20.0	33.5	12.1	...
Justin-W	Cum. <sup>c</sup>	100.0	.361	16.3	...	...	...	6.5
Justin-W	Orig. <sup>d</sup>	...	.361	16.5	...	...	...	...

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

<sup>b</sup>Determined from the distribution curve at 50% finer than.

<sup>c</sup>Cum. - Calculated value obtained by accumulating results proportional to the individual fractions.

<sup>d</sup>Orig. - Original flour before air classification.

Comparison of the cumulative results (Table II) with the original flour sample data shows reasonable recovery after air classification.

The farinogram data given in Table III show a wide range in the mixing characteristics of the air-classified fractions. Representative farinograms are shown in Fig. 3 for the control and air-classified flour fractions.

Flour fractions from one series were baked to characterize their baking performance. The results given in Table IV show the F-1 fraction gave a large open loaf, characteristic of high gluten flour; the F-2 fraction gave a small compact loaf, similar to a low-protein wheat; and the F-3 fraction a typical loaf of a hard red spring wheat flour for the particular protein content.

The results obtained from the air-classified flour fractions demonstrated divergent characteristics of the fractions. The range and magnitude of the analytical, physical, and rheological characteristics indicated the samples to be excellent starting materials for this study.

The three air-classified fractions (F-1, F-2, and F-3) from each of the four flours were extracted with distilled water according to the extraction procedure outlined in Fig. 2. The extraction data (Table V) indicated that, on an average, 96.5% of the starting material was recovered. The F-1 fraction consistently had the lowest

TABLE III. FARINOGRAM DATA FOR AIR-CLASSIFIED FLOURS

Sample		Abs. <sup>a</sup>	AR	PK	STAB.	MTI	TMD
Selkirk-C	F-1	75.8	6.0	15.5	... <sup>b</sup>	5	5
Selkirk-C	F-2	59.3	1.0	1.5	1.0	100	125
Selkirk-C	F-3	56.9	1.5	10.5	22.5	10	20
Selkirk-C	Orig. <sup>c</sup>	59.6	2.0	9.0	18.0	20	30
Selkirk-WP	F-1	78.2	4.0	8.0	13.5	15	40
Selkirk-WP	F-2	59.5	1.0	2.0	4.5	55	90
Selkirk-WP	F-3	61.7	4.0	7.5	16.0	20	30
Selkirk-WP	Orig. <sup>c</sup>	62.1	3.0	5.0	9.0	35	45
Justin-C	F-1	79.1	5.0	9.5	15.0	15	25
Justin-C	F-2	57.7	1.0	1.5	1.0	90	120
Justin-C	F-3	61.4	6.5	14.5	... <sup>b</sup>	5	10
Justin-C	Orig. <sup>c</sup>	63.3	6.5	19.0	... <sup>b</sup>	5	0
Justin-W	F-1	81.0	7.0	13.0	15.0	10	25
Justin-W	F-2	58.5	0.7	1.0	1.5	85	115
Justin-W	F-3	61.2	5.0	10.5	20.0	10	10
Justin-W	Orig. <sup>c</sup>	64.2	5.0	8.5	16.0	20	20

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

<sup>b</sup>Stability longer than 30 min.

<sup>c</sup>Orig. - Original flour before air classification.

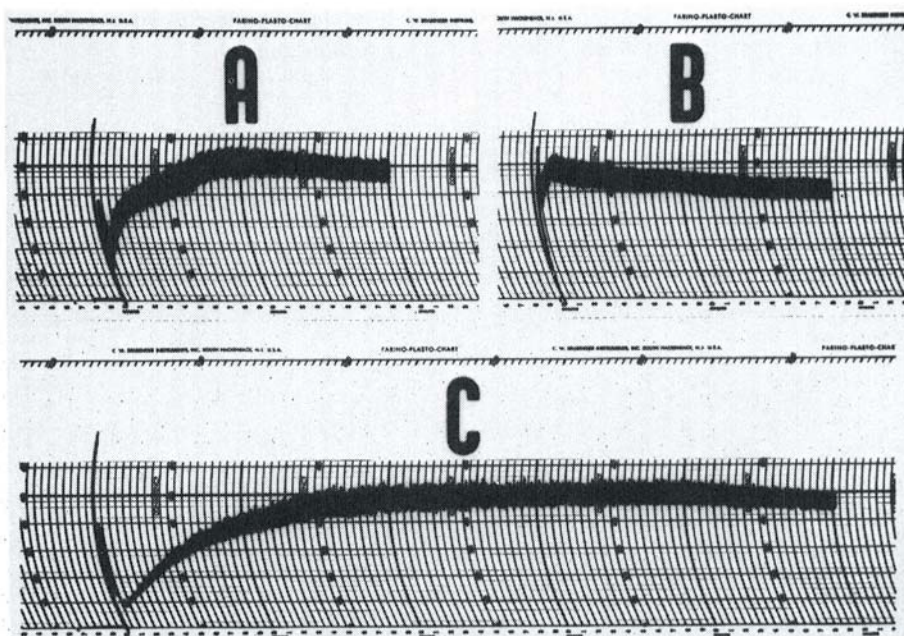


Fig. 3. Farinograms of: A = fine particle size, fraction F-1; B = medium particle size, fraction F-2; and C = coarse particle size, fraction F-3.

TABLE IV. BAKING RESULTS FOR AIR-CLASSIFIED FLOURS

Fraction	Abs. <sup>a</sup> %	Mixing Time min.	Dough Character <sup>b</sup>	Loaf Volume cc.
F-1	93.8	6.00	S	880
F-2	62.8	3.75	W dd	680
F-3	64.4	4.75	S-M	810
Std. <sup>c</sup>	63.6	3.75	S-M	855

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

<sup>b</sup>S = Strong; W dd = Weak, Dead; S-M = Strong to Mellow.

<sup>c</sup>Std. - Milling and baking 1963 crop hard red spring wheat.

recovery (94.4% average) and the F-3 the highest (98.7% average), while the F-2 was intermediate.

The amount of WS extracted was highest for the F-1 fraction and lowest for the F-3, with the F-2 fraction again intermediate.

Although there is a similarity between recovery data and water-solubles data, it does not seem reasonable that the two observations should be related. The studies of Deschreider (22) would explain, in part, the differences in amount of water-solubles extracted; with increased ion concentration, there was an increase in the amount of protein extracted. Comparison of the data in Table II and Table V shows that with increased flour ash, there was an increase in the amount of WS extracted. Since the ratio of water to flour was constant, the salt concentration of the extracting solution would change according to the flour ash.

TABLE V. WATER-SOLUBLES EXTRACTION DATA FOR AIR-CLASSIFIED FLOURS

Sample	Fraction	Solids %	Initial Water- Solubles %	Water- Solubles %	Precipitate %	Total Recovery %
Selkirk-C	F-1	90.6	9.4	7.1	2.3	95.1
Selkirk-C	F-2	92.7	7.3	5.1	2.2	97.4
Selkirk-C	F-3	92.0	8.0	3.5	4.5	99.9
Selkirk-WP	F-1	89.9	10.1	7.8	2.3	93.5
Selkirk-WP	F-2	93.2	6.8	5.4	1.4	95.6
Selkirk-WP	F-3	91.8	8.2	3.8	4.4	97.9
Justin-C	F-1	90.3	9.7	7.5	2.2	95.2
Justin-C	F-2	93.0	7.0	5.0	2.0	97.8
Justin-C	F-3	92.7	7.3	3.3	4.0	98.9
Justin-W	F-1	91.0	9.0	7.2	1.8	93.7
Justin-W	F-2	92.9	7.1	5.0	2.1	95.1
Justin-W	F-3	91.7	8.3	3.6	4.7	98.0
Averages	F-1	90.4	9.6	7.4	2.2	94.4
Averages	F-2	92.9	7.1	5.1	2.0	96.5
Averages	F-3	92.0	8.0	3.6	4.4	98.7



TABLE VI. PROTEIN CONTENTS OF AIR-CLASSIFIED AND WATER-EXTRACTED FRACTIONS<sup>a</sup>

Sample	Fraction	Original Flour %	Solids %	Initial Water-Solubles %	Water-Solubles %	Precipitate %
Selkirk-C	F-1	18.7	17.3	28.2	15.0	70.3
Selkirk-C	F-2	8.7	6.8	29.2	13.2	69.4
Selkirk-C	F-3	12.4	9.1	50.0	17.9	73.7
Selkirk-WP	F-1	18.2	17.6	20.0	14.1	41.6
Selkirk-WP	F-2	10.4	8.9	26.0	14.3	70.0
Selkirk-WP	F-3	15.7	12.1	47.7	20.0	71.9
Justin-C	F-1	19.9	18.7	26.5	16.5	64.0
Justin-C	F-2	9.1	7.1	32.1	16.7	70.5
Justin-C	F-3	13.1	9.7	49.7	21.7	74.1
Justin-W	F-1	22.3	21.2	29.2	19.3	69.8
Justin-W	F-2	9.9	7.7	36.0	18.9	74.5
Justin-W	F-3	15.7	11.6	54.0	26.5	74.5
Averages	F-1	19.8	18.7	26.0	16.2	61.4
Averages	F-2	9.5	7.6	30.8	15.8	71.1
Averages	F-3	14.2	10.6	50.4	21.5	73.6

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

Two other factors which could affect the amount of WS extracted were the extent of starch damage and the enzymatic activity of the fractions. The degree of starch damage in the air-classified fractions, previously cited in Table II, shows an increase for the fractions which tend to increase the yield of WS. Gracza's (3) data reveal that the finer fractions have the higher diastatic activity. This higher diastatic activity coincided with the higher yield of water-solubles. The complementary effect of these two factors (ash and starch damage) cannot be disregarded.

The protein content data for the fractions are given in Table VI. Comparison of Tables V and VI substantiates the explanations given for the different amounts of WS obtained for the various fractions. A correlation coefficient of +0.88 was obtained between flour protein and percent IWS extracted.

The data show higher protein contents in all of the precipitate fractions and low protein contents in the WS fractions. The average protein content of the F-1 and F-2 WS fractions is essentially the same. However, the protein contents of the IWS show the F-2 fractions to have higher protein contents than the F-1 fractions, although the original F-2 flours had much lower values. Therefore, either more nonproteinaceous constituents must be extracted in the F-1 WS fractions than in the F-2 fractions to produce a higher yield of WS, or the two factors, ash and starch damage, increase the solubility of certain proteins in F-1 but not F-2. It is possible that both occur, but the latter explanation appeared to be more reasonable because of the effect of the F-1 WS on mixing discussed later.

Mixograms were obtained on all of the original flour fractions, solids, solids plus IWS, solids plus WS, and solids plus P. The amounts of solids, IWS, WS, and P in the reconstituted series were proportional to their content in the original flour fractions. The exemplary mixogram data are given in Table VII.

TABLE VII. MIXOGRAM DATA ON RECONSTITUTED SAMPLES

Sample <sup>a</sup>	Fraction	Origin							
		Selkirk-C		Selkirk-WP		Justin-C		Justin-W	
		Abs. <sup>b</sup> %	Peak min.	Abs. <sup>b</sup> %	Peak min.	Abs. <sup>b</sup> %	Peak min.	Abs. <sup>b</sup> %	Peak min.
Original	F-1	85.7	8.9	86.2	6.3	90.8	9.1	96.6	7.9
S	F-1	84.1	21.3	83.6	15.9	85.0	21.4	87.2	10.6
S + IWS	F-1	85.5	10.9	83.9	7.6	87.2	9.0	86.6	7.8
S + WS	F-1	85.0	13.0	85.0	11.1	85.8	13.7	89.4	10.8
S + P	F-1	85.8	11.2	85.0	8.7	86.3	12.7	88.2	8.1
Original	F-2	62.4	4.6	65.2	3.5	63.4	4.9	62.5	4.4
S	F-2	64.2	30+	63.1	30+	62.3	30+	59.3	30+
S + IWS	F-2	64.2	10.8	62.9	5.4	62.5	8.8	62.6	5.2
S + WS	F-2	63.9	30+	63.6	30+	63.6	30+	63.0	30+
S + P	F-2	66.4	12.8	62.4	9.8	63.9	30+	62.1	11.5
Original	F-3	64.4	5.4	72.6	5.2	70.2	5.7	73.7	6.1
S	F-3	55.0	30+	61.7	30+	55.6	30+	60.5	30+
S + IWS	F-3	63.8	6.6	60.8	3.7	63.6	6.7	66.3	5.8
S + WS	F-3	58.3	30+	61.9	30+	58.7	30+	62.4	16.6
S + P	F-3	63.6	6.9	61.7	4.4	62.2	9.1	65.5	7.6

<sup>a</sup>S = Solids after 10:1 extraction with water to flour; IWS = initial water-solubles extracted at 10:1; WS = water-solubles obtained from dried IWS extracted at 5.5:1 with water to dried IWS; P = insoluble precipitate of extracted dried IWS.

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

TABLE VIII. MIXOGRAM DATA ON SPECIAL BLENDS

Sample <sup>a</sup>	Fraction					
	F-1		F-2		F-3	
	Abs. <sup>b</sup> %	Peak min.	Abs. <sup>b</sup> %	Peak min.	Abs. <sup>b</sup> %	Peak min.
Solids	86.3	14.4	58.8	30+	56.9	30+
Solids + WS	88.2	13.2	61.8	30+	59.7	30+
Solids + P	87.2	10.3	62.6	12.6	64.9	7.6
Solids + crude gliadin	85.8	11.1	66.7	8.9	64.9	11.0

<sup>a</sup>WS = Water-solubles; P = precipitate.

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

Samples which did not break down after 30 min. of mixing were arbitrarily stopped. The peak time was recorded as 30+ min. for these samples. One sample was tested for longer than 30 min. and after 1 hr. mixing, it showed no signs of dough breakdown.

The data show that removal of the IWS increased the mixing time as determined by the mixogram peak, whereas the addition of the IWS to the solids decreased the mixing time for the majority of the samples, and made them appear comparable to the original flour. Although the WS had no appreciable effect on mixing, the P had a similar but less effect on mixing than that of the IWS when added to the solids, except for the Justin-C F-2 flour.

The data in Table VIII again show that the WS had little effect on mixing time, whereas the P reduced the mixing time. The gliadin, like the P, reduced the mixing time. This verifies the findings of Mattern and Sandstedt (23), Smith and Mullen (24), and others. Since somewhat different proteins were extracted from the F-1 fractions compared to the F-2 fractions, this would account for the F-1 WS fractions shortening the mixing time, even though it was not as appreciable as the IWS or P fractions.

Lower molecular weight proteins of gluten (gliadin) contribute to the extensibility and nonelastic characteristics of a dough. They may be extracted with water, but the amount and composition of the proteins depend on either the number of extractions or the ratio used. These gliadin or "gliadin-like" proteins were partitioned into the P fraction, which accounted for the effect on mixing time. The effect of extending the mixing time was not unique to a wheat variety or classified fraction, but was caused by the P similarly extracted from each. Since the F-1 fractions contained higher amounts of ash, it was shown that some "gliadin-like" proteins were extracted by the effect of the F-1 WS on the mixing time.

Although the amount of WS extracted from a given air-classified flour fraction was similar regardless of the source of the initial flour of comparable grade, the quantity extracted depended on the air-classified fraction. The differences observed in the amount of WS extracted from the air-classified fractions and their protein contents were attributed to the initial physical and biochemical properties of the flours rather than the wheats from which the flours were ground.

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