. DEPARTMENT OF AGRICULTURE / CONSUMER AND MARKETING SERVICE / MARKETING RESEARCH REPORT NO. 804

ESTIMATION and CONTROL of Experimental Error in the Falling Number Test for WHEAT and FLOUR

SUMMARY

An attempt was made to investigate all possible sources of error in the falling number test method using designed experiments. The results were analyzed statistically, and the following sources were found to be significant:

1. Temperature of boiling water bath
2. Temperature of water used in test
3. Volume of water used in test
4. Position of tube while mixing
5. Severity of mixing technique
6. The amount of sample milled
7. The amount of chlorine gas used for bleaching

Where it was possible, various measures were proposed to control the sources of error.

Ten sources proved to be not significant but are reported to inform the reader of the extent of this study. One of these was the addition of calcium compounds to flour to increase its nutritional value.

A recommended falling number procedure for testing samples is proposed based on the results of the factors studied.

ACKNOWLEDGEMENT

Appreciation is expressed to Ned S. Hurst, formerly of the Grain Division, Consumer and Marketing Service, for his assistance in conducting the numerous experiments in this study.

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PREFACE

The purpose of this study was to locate all significant sources of error in the falling number test and to establish controls for them. The number of samples used to investigate a source of error was minimized because of the amount of work required to examine all possible factors which might influence the falling number results. An effort was made to choose samples covering a broad range of falling number values for each experiment but at the same time to select them at random.

In some experiments the difference between the lowest and highest mean value approached the least difference for significance. If such experiments were repeated with a much larger number of samples, a significant difference might be shown for this particular source of error.

Since most of the experiments were carried out using three types of commercial flours--bread, all-purpose, and cake--the conclusions drawn are especially applicable to them. Work done with wheat was for the purpose of observing the effect of granularity resulting from the method of milling the wheat.

Some readers may not be well versed in statistics; therefore, the analyses of variance are not shown. Presentation of observations, mean values, and the Least Significant Difference should serve as adequate proof for the validity of the experiment. Readers desiring statistical analyses of the data can obtain them from the authors.

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* Factors which cause significant differences in falling number values.

ESTIMATION AND CONTROL OF EXPERIMENTAL ERROR IN THE FALLING NUMBER TEST FOR WHEAT FLOUR

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INTRODUCTION

The falling number test was developed by Hagberg (1, 2) as a simple, quick index for the alpha-amylase activity of grain. Falling number is the time, in seconds, required to stir and allow the viscometer-stirrer to fall a fixed distance through a hot aqueous flour suspension being liquefied by the enzyme. Perten (3) has shown the relationship between falling number, amylograph Brabender units, diastatic activity, and other measures of alpha-amylase activity.

Sprouting of wheat increases its alpha-amylase activity. When a flour-water suspension containing an excess of the enzyme is heated, the thick starch-gel first formed is quickly liquefied or thinned. Flour containing large quantities of alpha-amylase makes poor gravies and in breadmaking produces dough of poor quality and bread of low loaf volume.

On the other hand, an adequate amount of alpha-amylase is indispensable for breadmaking. In combination with beta-amylase, it converts colloidal starch to maltose which is used by yeast in dough fermentation. The residual sugar contributes to a desirable taste in the finished product.

It is therefore important to know the strength of the alpha-amylase in flour and to establish a range of values for its optimum behavior. Furthermore, it is desirable to have a quick reliable method for determining the alpha-amylase activity which has satisfactory interlaboratory agreement.

Although the falling number apparatus and its operation are simple, sources of interlaboratory error must be established before satisfactory agreement is possible. Operators unknowingly tend to introduce variables into an empirical test. The controls they use in their technique and maintenance of equipment invariably lead to interlaboratory differences in values which are not apparent to the operators.

Hagberg (2) stated that the laboratory reproducibility of the falling number test is within + 5% of a mean value providing the conditions are controlled. Medcalf et al. (4) state that if the conditions are not controlled, the error will be significantly greater than 5%. The factors which they studied were: (1) blending of sprouted wheat, (2) method of milling the wheat, (3) temperature of water-bath, and (4) water-bath level.

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In the present study the authors used uniformly blended flour to test all factors except those relating to wheat. Thus, by eliminating the factor of granularity in wheat meal, the other factors under study were measured more accurately. The apparatus used was that made by Falling Number AB, Norrlandsgaten 16, Stockholm, Sweden. 3/ The method used was that of the International Association for Cereal Chemistry. The method is essentially the same as that provided with the instrument.

3/ The mention of firm names or trade products does not imply that they are endorsed or recommended by the U. S. Department of Agriculture over other firms or similar products not mentioned.

SOURCES OF ERROR

Water-bath level

Temperature of the vapor above the waterline of an unstoppered bath varied a few tenths of a degree over a period of two hours boiling, during which time the water level dropped two inches or more. Under such conditions the degree of immersion of the test tube would vary and this was considered as a possible significant source of error in the test.

Six samples of flour having falling numbers of 96, 127, 161, 175, 362, and 427 were tested in duplicate at the normal water-bath level, controlled by the overflow-outlet located one inch below the cover of the bath; another set was tested two inches below the cover. To maintain a constant bath level, an empty tube was inserted into the well between tests to prevent escape of steam. Results of this test, shown in Table 1, indicate that there is no significant difference between the means.

From this study it was concluded that the bath should be stoppered to insure results which do not vary significantly. It is recommended that the overflow level be adjusted so that when covered, the water level will be one inch from the top of the apparatus. This will insure that the length of the tube immersed in the boiling water will be constant for all laboratories.

Table 1.--Effect of water level in the bath on falling number

Overflow setting	Falling Number							
	Sample Number							
	1	2	3	4	5	6	Mean	
1" below cover	165	432	96	130	183	358	224.8	*
	157	423	95	124	167	367		
2" below cover	166	410	104	124	177	344	222.9	
	169	418	102	125	181	355		
L.S.D. (5%)-10.0								

* Means connected by same line are not significantly different.

Temperature of boiling water-bath

Four flour samples were tested at six controlled temperatures: 95.0, 96.0, 98.0, 100.0, 100.5, and 101.5° C. The prevailing boiling point of water in the author's laboratory for the experiment was 100.5° C. Adjustment to lower temperatures was made by adding isopropyl alcohol and to higher temperatures with glycerol. Falling number in relation to temperature is shown in Figure 1. The graphs for samples 2, 3, and 4 are almost linear; whereas, for sample 1 it is curvilinear. Apparently the higher the falling number the greater is the change in falling number per degree change in temperature.

Figure 1 shows that wide differences in temperature, which might be encountered among laboratories at different elevations, cause great differences in falling number. The data in Table 2 shows that variations exceeding 0.4° C. cause significant differences. It is highly probable that day-to-day fluctuations of as much as 0.5 inch in barometric pressure within a laboratory would affect the falling number values significantly if the tests were repeated.

Bath temperature control agents are being studied by the AACC subcommittee on falling number. If the collaborator range of mean values is narrowed and experimental error lowered through their use, it is likely that use of controls will become part of the official method.

Table 2.--Effect of bath temperature on falling number

	Falling Number					
Sample Number	1	2	3	4	Mean	
Temperature (°C.)						
99.0	200 193	152 145	119 119	117 110	144.4	
99.8	181 191	145 134	111 112	106 111	136.4	*
100.2	174 185	144 138	112 114	101 106	134.2	
101.0	175 159	133 130	103 109	99 102	126.2	
L.S.D. (5%) - 5.8						

* Means connected by same line are not signigicantly different.

It is recommended that the temperature of the boiling water-bath be recorded when the temperature of the boiling water-bath becomes constant. Insert a thermometer through a one-hole No. 5 rubber stopper and place it into the well which normally holds the falling number tube. The immersion tip of the thermometer must be below the bath level.

Only a National Bureau of Standards thermometer with a guaranteed accuracy of ± 0.3° C., or its equivalent, should be used. It should have easy-to-read divisions of 0.1° C. If necessary, a stem correction should be applied.

Mineral content of water used in test

Mineral-free distilled water and tap water with 7 and 35 p.p.m. minerals, respectively, expressed as calcium carbonate were used to determine falling number in duplicate on six samples of flour. The results are shown in Table 3. The mineral content of water within the limits tested is not a factor in the falling number determination hence no tolerance for mineral content of distilled water is suggested.

Table 3.--Effect of mineral content of water used in the test on falling number

Mineral content (p.p.m.) 4/	Falling number						
	Sample number						
	1	2	3	4	5	6	Mean *
0	171 166	339 347	168 186	135 136	199 214	102 104	188.9
7	173 167	359 332	182 189	143 142	224 225	103 104	195.2
35	191 185	361 349	163 175	137 134	225 211	95 97	193.6
L.S.D. (5%) - 9.6							

* Means connected by same line are not significantly different.

Temperature of water used in the test

Falling numbers were determined on six samples of flour using distilled water at temperatures of 70°, 78°, and 100° F. for preparing the suspensions. The mean falling numbers differed significantly, as shown in Table 4. This indicates that the temperature of the water used for preparing the suspensions should be controlled. Water from a still frequently is warm and might exceed 90° or even 100° F. It is recommended that the water used in the test have a temperature of 80°F., plus or minus 3°F.

4/ p.p.m. expressed as calcium carbonate.

Table 4.--Effect of temperature of water used in the test on the falling number

Water temperature (°F)	Falling number						
	Sample number						
	1	2	3	4	5	6	Mean
70	99 110	160 160	129 125	107 103	102 99	381 398	169.7 a*
78	109 111	159 179	134 138	106 107	107 109	387 390	164.4 b
100	100 97	158 155	128 126	99 96	97 99	383 388	160.5 c
L.S.D. (5%) - 3.2							

* Different alphabetical letters under falling number means indicate they are statistically different at the level of probability stated.

Volume of water in test

Five flour samples were tested with different quantities of water; namely, 24.5, 25.0, and 25.5 ml. The results in Table 5 show that an error of -0.5 ml. in measuring the 25.0 ml. of water can cause a significant error in the falling number value. On the other hand, no significant effect was observed where the error was +0.5 ml.

The effect of decreasing the water volume to 24.5 ml. is the same as increasing the percent flour in the mixture; namely, increasing the falling number. Increasing the volume to 25.5 ml. reduces the percentage of flour in the mixture and lowers the falling number (Figure 2).

For delivery of the correct volume of water it is recommended that in the order of convenience, either automatic pipets, 25-ml. graduated cylinders calibrated to deliver and graduated in 0.2 ml. intervals, or hand pipets be used.

Table 5.--Effect of quantity of water used in the test on falling number

Water volume (ml.)	Falling number: Sample number 1	2	3	4	5	Mean
24.5	166	102	127	375	99	171.3
	171	98	131	345	99	
25.0	154	100	126	344	96	168.4 *
	173	97	122	361	96	
25.5	164	103	124	345	95	166.9 *
	158	100	127	370	98	
L.S.D. (5%)-2.8						

* Means connected by the same line are not significantly different.

Size of flour particles

One hundred grams each of two samples of Buhler-milled flour were fractionated on a Rotap shaker using a nest of U. S. Standard No. 100, 170, 200, 230, and 270 woven-wire cloth sieves. The flour passing through each sieve had a particle size of less than 149, 88, 74, 62, and 53 microns, respectively. The results of falling number determinations and flour yield on each fraction are given in Table 6. Even though there were wide differences in particle size, the effect on falling number was not significant.

Table 6.--Effect of flour-particle size on falling number

Flour Fraction (microns)	Percentage of original flour through sieve: Sample 1	Sample 2	Falling number: Sample 1	Sample 2	Mean
89 - 149	14	40	95	320	207.5 *
75 - 88	37	12	108	330	219.0 *
63 - 74	28	8	109	324	216.5 *
54 - 62	10	30	111	360	235.5 *
53	11	10	103	330	216.5 *
Original Flour	100	100	96	350	223.0 *
L.S.D. (5%)					

* Means connected by same line are not significantly different.

Within the limitations specified, flour granularity did not affect falling number significantly.

Any of the following mills may be used to mill the wheat, providing the meal is sieved through a U. S. No. 100 woven-wire sieve or its equivalent:

1. Brabender Quadrumat
2. Tag or Strand
3. Brabender Sedimat
4. Buhler
5. Labconco
6. Weber Pulverizer
7. Udy-Cyclone

Size of flour sample

The almost linear relationship between sample weight and falling number is clearly shown in Figure 2 for four flour samples. The degree of error in falling number due to an error in weighing 7.0 grams may be read from the curve. An error of 70 mg. (1 percent) in weighing a sample would cause an error of approximately 1 percent in falling number. However, it is not likely such an error would be made in weighing.

Moisture content of wheat

In Sweden, according to a communication from Perten, the moisture content of the wheat or flour is determined. Then a sample weight equivalent to 7.0 g. on a 15-percent moisture basis is selected from a table and weighed for the test.

In the United States most chemists might prefer to report their results on a constant 14-percent moisture basis. If so, they can easily prepare a table, as in Sweden, at the 14-percent moisture level and carry out the test in a similar manner.

Results on four samples, Table 7, show that falling number might be determined on an "as is" basis and then calculated to a 14-percent moisture basis. The wheat samples adjusted to approximately 9-, 11-, 13-, and 14-percent moisture content were equilibrated for 24 hours, milled on the Tag mill, and sieved. As the moisture content decreased, the falling number values became proportionately greater. This is in agreement with the results in Table 5 and Figure 2 which show that the falling number increased proportionately with the flour content of the suspension. However, more samples should be tested before recommending this procedure which eliminates weighing samples of different weight. The decision will be made by the AACC subcommittee on falling number.

Table 7.--Effect of moisture content of wheat on falling number

Sample 1			Sample 2		
	Falling number			Falling number	
Moisture (%)	"as is" moisture basis	14% moisture basis	Moisture (%)	"as is" moisture basis	14% moisture basis
9.0	354	335	8.7	166	156
10.6	370	356	11.3	159	154
12.6	345	339	13.1	148	146
14.0	340	340	14.5	151	152

Method of suspending flour in water

Position of flour in tube

It is recognized that the flour must be completely wetted to obtain precise results. Therefore, ease of wetting was investigated.

Six flour samples were tested in duplicate with the flour of each in the following two positions: (1) level, and (2) slanted at approximately 45° while water was added. The tubes were shaken upright (vertically) 30 times through a space of 8 inches in 10 seconds. No significant differences were observed between the mean values. See Table 8.

Table 8.--Effect of the flour position in the test tube on falling number

Position	Falling number							
	Sample number							
	1	2	3	4	5	6	Mean	
Level	162 155	423 415	101 102	129 123	162 169	358 360	221.6	| *
Slant (45°)	165 157	432 423	96 95	130 124	183 167	358 367	224.8	|
L.S.D. (5%) - 5.9								

* Means connected by same line are not significantly different.

Position of tube while mixing

Six replications of one flour sample were suspended in 25 ml. of distilled water by each of the following methods: (1) shaking tube upright 30 times through a distance of 8 inches in about 20 seconds, (2) shaking tube horizontally in the same manner, and (3) shaking tube at a 45° angle, also in the same manner.

The results are shown in Table 9. Whereas no significant difference was observed between replicates in any position, there was a significant difference between the horizontal mean compared to the means for the upright and 45°-angle positions. No significant difference was observed between the upright mean and the 45°-angle mean.

The difference in the mean values might be due to the alpha-amylase not going into solution as completely when the tube is shaken horizontally compared to vertically. Release of less enzyme would explain the higher mean falling number value for the horizontal shaking position as shown in Table 9.

Table 9.--Effect of tube position while shaking on falling number

Position of tube	Falling number							
	Replications							
	1	2	3	4	5	6	Mean	
Horizontal	108	106	106	102	107	107	106.0	*
45° Angle	104	103	106	98	104	104	103.2	⎤
Upright	99	103	102	101	99	106	101.7	⎦
L.S.D. (5%) - 2.7								

* Means connected by same line are not significantly different.

Severity of mixing technique

It was observed that some operators testing the same samples under almost identical conditions obtained different values. The following methods to suspend the flour were used by three experienced operators: (1) shaking the mixture 30 times with the tube upright through a distance of 8 inches in 20 seconds, (2) shaking the upright tube 20 times through 8 inches in 15 seconds, and (3) shaking the upright tube 10 times through 8 inches in 10 seconds.

The results in Table 10 show no significant differences due to the number of strokes but a highly significant difference due to operators.

If the mixture is shaken forcefully with jerks to suspend the flour as was done by Operator 3 (Table 10) the results will be too high. A forceful, jerky motion probably compacts the flour and retards alpha-amylase extraction. Shaking with a smooth energetic stroke--the method used by Operators 1 and 2--is recommended.

Since there was no significant difference between the means of 10, 20, or 30 strokes, it is recommended that 10 be used.

Table 10.--Effect of severity of hand-shaking the flour-water mixture on falling number

Operator	1			2			3		
No. of strokes	10	20	30	10	20	30	10	20	30
Sample 1	104 107	103 105	102 104	105 108	104 103	103 104	110 114	116 110	114 112
Sample 2	106 105	110 102	109 109	111 110	105 106	106 106	110 108	112 104	114 112
Sample 3	128 128	129 133	134 131	133 137	131 132	127 128	142 143	136 140	139 135
Sample 4	103 101	102 93	98 99	106 103	107 101	101 102	107 110	106 104	103 101
Sample 5	101 104	104 102	104 98	103 105	103 107	105 107	110 112	109 110	108 110
Mean for operators		108.6			110.3*			115.4	

L.S.D. (1%) - 2.3

Mean for strokes (all operators) 112.0 111.0 110.9*

L.S.D. (5%) - 1.7

*Means underscored by same line are not significantly different.

Number of viscometer-stirrer strokes

The test requires 110 strokes in 55 seconds where a stroke is a complete down-and-up movement of the viscometer-stirrer through the mixture. The effect of variation from this number was studied. Falling numbers were determined in duplicate on six flour samples using 88, 110, and 132 strokes at a uniform rate in 55 seconds.

If 88 strokes are used, the mean falling number will differ significantly from those for 110 and 132 strokes, as shown in Table 11. Whereas 110 strokes is probably optimum, less vigorous stirring probably fails to produce a uniform mixture and proper liquefaction. The use of more than 110 strokes is unnecessary.

Table 11.--Relationship between the number of viscometer strokes and falling number

Number of strokes	Falling number						
	Sample number						
	1	2	3	4	5	6	Mean
88	176 178	475 477	111 109	134 133	188 191	363 372	242.2
110	165 157	432 423	96 95	130 124	183 167	358 367	224.8 *
132	156 152	427 417	90 98	123 122	163 163	347 351	217.4 *
L.S.D. (5%) - 12.1							

* Means connected by same line are not significantly different.

Tests on uniformity of apparatus

The most likely source of error due to differences in the make-up of the falling number apparatus would be in the size and weight of the viscometer-stirrer. Other factors would be the heating capacity of the hot plate and thickness or diameter of the falling number tubes.

Falling number was determined in duplicate on seven flour samples using two falling number instruments. One was an earlier model without an automatic timer cut-off and signal device. The results of these tests are shown in Table 12. There was no significant difference between the mean results that might have been caused by variations in instrumentation, at least for these two instruments.

Table 12.--Falling numbers determined with two instruments

	Falling number							
	Sample number							
	1	2	3	4	5	6	7	Mean
Old model	161	134	106	102	355	324	145	
	164	138	106	103	380	325	140	192.0 *
New model	173	130	104	98	360	333	140	
	172	130	110	99	371	330	138	191.6 *
L.S.D. (5%) - 5.6								

* Means connected are not significantly different.

In a study conducted in a midwest laboratory 5/, four machines were tested by three operators. The results are shown in Table 13. No significant differences were observed in the falling numbers determined with the different instruments or between operators.

Table 13.--Falling number determined with four instruments by three operators 6/

	Operator			
	1	2	3	
Instrument	Falling number			Mean (Instrument)
1	188	188	200	192.0 *
2	190	194	185	189.7 *
3	187	190	189	188.7 *
4	170	180	190	180.0 *
Mean (operator)	183.8*	188.0*	191.0*	
L.S.D. (5%) - 10.9				L.S.D. (5%) - 12.5

* Not significantly different.

5/ Doty's laboratory workshop on falling number, July 22, 1966, in Kansas City.
6/ Doty's laboratory workshop, July 22, 1966, in Kansas City.

Influence of type of mill used for milling on falling number

The subcommittee on falling number 7/ of the AACC in addition to flour has tested one sample of wheat, milled on four different mills; namely, Tag, Labconco, Quadruplex, and Weber. The ground meal was sieved on a U. S. Standard No. 100 woven-wire-cloth sieve to obtain flour for falling number tests. The mean falling number of the resulting flour was 193.8 with a coefficient of variation of 7.9, about the same as would be found in a sample of flour. It was concluded that different mills would not constitute a significant source of error providing the flour passed through a standard sieve.

In another test, four samples of wheat were ground on the Labconco Mill using the No. 8, 16, and 18 settings to achieve a wide spread in granularity of the meal. The falling numbers on the unsieved meal as compared with that of Tag-milled flour are shown in Table 14. Differences in granularity, obvious to the eye, did not produce significantly different falling numbers. However, a comparison of individual observations of sample 2 for different settings shows some rather wide deviations. Such differences, we believe, are due mostly to granularity. Within-sample variation should be minimized by obtaining the finest grind possible. A particle-size distribution with a Rotap shaker and a nest of sieves should be:

greater than 500 microns......... 0 - 10 percent

less than 500 microns but
greater than 210 microns......... 25 - 40 percent

less than 210 microns............ 75 - 50 percent

Table 14.--Effect of granularity of wheat meal prepared on the Labconco Mill on falling number

Sample number	Falling number			
	Labconco Mill			Tag Mill (flour)
	Setting number 8/			Setting
	8	16	18	.023"
1	274 281	264 265	295 295	295 310
2	235 201	171 151	180 177	231 220
3	254 245	260 245	250 226	242 246
4	227 254	262 253	273 270	268 271
Mean	246.4	233.9	245.8	260.4*
L.S.D. (5%) - 29.6				

* Means underscored by same line not significantly different.

7/ Report of the AACC Falling Number Subcommittee, September 8, 1966.

8/ Granularity increases almost proportionately with setting number.

Note: Because of its high bran content wheat meal contains proportionately less starch than flour samples. Tag results are probably higher because of its greater starch content which, no doubt, increases falling number.

The following are some mills capable of producing meal with satisfactory particle-size distribution:

1. Kamas-Slago (Swedish mill)
2. Weber Pulverizer (.024 inch sieve)
3. Udy-cyclone (.024 inch sieve)
4. Labconco (No. 8 setting)

The Kamas-Slago (a Swedish mill), the Labconco mill adjusted to the No. 8 setting, and the Weber Pulverizing mill with the .024 inch sieve were used to grind four wheat samples. Results of the falling number determinations made on the unsieved meal are shown in Table 15. There was no significant difference in falling number of the meal as produced by the three mills.

Table 15.--Comparison of falling number of the wheat meal produced by three mills

Sample number	Falling number obtained when using: Labconco Mill	Weber Pulverizing Mill	Kamas-Slago Mill
1	283 290	332 331	302 302
2	207 212	221 213	229 233
3	292 289	286 289	279 272
4	279 279	291 288	273 275
Mean	267	282	271 *
L.S.D. (5%) - 24.5			

* Means underscored are not significantly different.

The effect of the size of sample milled on falling number

The Labconco Mill at the No. 8 setting was used to grind 50-, 100-, and 200-gram portions of four wheat samples.

Falling numbers were determined in duplicate on each sample. The results show that a significant difference exists between the 50- and 100-gram samples. It is suggested that at least a 100-gram sample be used when the Labconco mill is employed.

The Udy-cyclone Mill employing a .024 and .041 inch sieve was used to grind 25-gram samples, it is apparent that, although there is no significant difference between the mean values, there is greater difference between duplicates where the .041 inch sieve was used. Both mean values agree with the mean values of the Labconco mill for the 100- and 200-gram samples. If the Udy-cyclone or Weber Pulverizer mill is used, 25-gram samples are sufficient.

Table 16.--Effect of size of sample milled on falling number

Sample number	Falling number obtained when using:				
	Labconco Mill (No. 8 setting)			Udy-Cyclone	
	50 g.	200 g.	100 g.	.024" 25 g.	.041" 25 g.
1	298 272	323 314	338 318	336 340	284 294
2	119 120	116 119	97 111	140 147	120 99
3	222 219	228 230	210 209	212 215	253 265
4	118 127	115 110	146 145	144 149	162 142
Mean	186.9	194.4	196.8*	210.4	202.4*
L.S.D. (5%) - 9.2					

* Means underscored by same line are not significantly different.

CALCIUM-ENRICHED FLOUR

The U. S. Government is procuring calcium-enriched flour for several of its donation programs, and is considering establishing falling number requirements for this type of flour as well as for noncalcium-enriched flour. The question arose as to whether enrichment of flour with calcium salts would affect the falling number.

Calcium carbonate and calcium dihydrogen phosphate were added to subsamples of flour so that they contained 500, 600, 1200, and 2400 mg. of calcium per pound. The falling numbers of these as compared with non-fortified flour are shown in Table 17.

Table 17.--Effect of calcium addition to flour on falling number

	Falling number					
Calcium added (mg. Ca/lb.)	500	600	1200	2400	Control	Mean
Additive						
Calcium carbonate	169 172	160 169	169 155	172 170	156 162	165.4
Calcium phosphate	170 167	165 157	156 164	156 159	155 169	161.8
Mean	169.5	162.8	161.0	164.2	160.5*	
L.S.D. (5%) - 11.9						
L.S.D. (5%) -						7.5

* Means underscored by same line are not significantly different.

FLOUR BLEACHING WITH CHLORINE

A cake flour and an all-purpose flour were bleached with chlorine at a milling company 9/. The flours were tested for falling number and pH value prior to and 24 hours after bleaching. The results are as follows:

Chlorine Treatment	Cake Flour		All-Purpose Flour	
	F.N.	pH	F.N.	pH
Before	400	6.10	409	5.80
After	452	5.50	508	4.75

9/ Wilkins Rogers Milling Company, Washington, D. C.

It is apparent that chlorine treatment caused a significant increase in falling number, and, as expected, a decrease in pH value.

In another experiment, four Buhler laboratory-milled flours and two commercial all-purpose flours were treated with varying amounts of chlorine-air mixtures as well as pure chlorine under controlled conditions. The results are shown in Table 18.

Table 18.--Effect of chlorine on the falling number and pH value of laboratory-milled and commercial all-purpose flours

Sample No. 10/	Class of Wheat	Chlorine treatment % Control F.N.	pH	0.25 F.N.	pH	1.00 F.N.	pH	100.00 F.N.	pH
1	HRW	439	5.90	434	5.55	397	4.75	99	2.10
2	SRW	282	5.30	304	5.15	311	4.45	102	1.50
3	SRW	360	5.70	357	5.65	385	4.55	94	1.90
4	SW	324	5.80	333	5.65	347	4.35	111	2.00
11/5		117	5.60	115	5.30	128	4.70	79	2.00
6		111	5.65	105	5.40	131	4.90	71	2.20

FLOUR BLENDING

Frequently millers desire to blend wheats or flours to prepare a flour with special characteristics. From that point of view, we determined whether falling number is an additive property.

Two samples of flour having falling numbers 79 and 372, respectively, were blended in different proportions. The falling number of each blend was determined and the relationship between falling number and blend ratio was plotted as shown in Figure 3. Flour sample A, falling number 79, had a more pronounced effect in lowering the falling number than would be surmised from its percentage in the blend. If the falling numbers were additive, a mixture of 10 percent A and 90 percent B should produce a falling number value of 342; whereas, the actual value was 177.

10/ Samples 1, 2, 3, and 4 are laboratory-milled flours.

11/ Samples 5 and 6 are commercial all-purpose flours.

Perten (3) recommends that flour blends be made on the basis of liquefaction numbers which function linearly rather than on the basis of falling numbers which show a curvilinear relationship. He recommends the following formula for calculating the liquefaction number:

$$\text{Liquefaction number (L.N.)} = \frac{6000}{\text{falling number} - 50}$$

Utilizing the liquefaction number of the components and the blend, the percentage of each component required can be computed from the following equation:

$$\frac{X\ (\text{LN of A})}{100} + \frac{(100-x)\ \text{LN of B}}{100} = \text{L. N. of blend}$$

where x = % of component A
100-x = % of component B

Example: Falling Number of A = 79; L. N. = 206.9
Falling Number of B = 372; L. N. = 18.6
Falling Number of blend = 177; L. N. = 47.2

By substitution in above equation:

$$\frac{x\ (206.9)}{100} + \frac{(100-x)18.6}{100} = 47.2$$

x = 15.2% of flour A
100-x = 84.8% of flour B

The values obtained from the equation are the percentages of each flour needed for a blend with falling number 177. They agree within about 5 percent of the experimental blend (10% A-90%B) read from Figure 3.

RECOMMENDED FALLING NUMBER PROCEDURE

1. Fill apparatus with distilled water to the water level control fixed at 1 inch below cover. Place rubber stopper containing a National Bureau of Standards thermometer or its equivalent in the tube-well and permit to remain until temperature reading becomes constant. If the temperature of the bath is between 98.0° and 99.8° C., adjust it to 100.0° C. with ethylene glycol or glycerol. The quantity required is shown in the following table:

Require Temperature Elevation (° C.)	Quantity to be added (percent by volume)	
	Ethylene Glycol	Glycerol
0.2	1.9	1.9
0.4	3.9	4.9
0.6	5.8	7.4
0.8	7.8	9.8
1.0	9.7	12.3
1.2	11.3	14.2
1.4	12.9	16.1
1.6	11.4	18.1
1.8	16.0	20.0
2.0	17.6	21.9

If the bath temperature is below 98.0° C., the falling number determination cannot be made at 100.0° C. by temperature adjustment because of danger of boiling out the contents of the tube. Instead, the falling number may be estimated as follows: Determine the falling number at the observed boiling point, assumed 96.0° C. Then adjust the temperature to 97.5° by adding 13.6% ethylene glycol and determine the falling number again. Plot both values on graph paper in relation to temperature and extend the slope of the curve to 100.0° C. Read the falling number from the graph at this point.

2. Weigh 7.0 g. of sample on a 14-percent moisture basis (see Table 19 for weights corrected to a 14-percent moisture basis) into a dry falling number tube and tip to a 45° angle. Add 25 ml. of distilled water with an automatic pipet. Insert a rubber stopper and shake tube vigorously in upright position 10 times (up and down) making sure that all of the flour is in suspension by upending.

Table 19.--Weight of Sample corrected for moisture content

Moisture content (Pct.)	Weight of sample 12/ grams	Moisture content (Pct.)	Weight of sample 12/ grams	Moisture content (Pct.)	Weight of sample 12/ grams
8.0	6.54	10.8	6.75	13.6	6.97
.2	6.56	11.0	6.76	8	6.98
.4	6.57	2	6.78	14.0	7.00
.6	6.59	4	6.80	2	7.02
.8	6.60	6	6.81	4	7.03
9.0	6.62	8	6.83	6	7.04
.2	6.63	12.0	6.84	8	7.07
.4	6.64	2	6.86	15.0	7.08
.6	6.66	4	6.87	2	7.10
.8	6.67	6	6.89	4	7.12
10.0	6.69	8	6.90	6	7.13
.2	6.70	13.0	6.92	8	7.15
.4	6.72	2	6.94	16.0	7.17
.6	6.73	4	6.95	16.2	7.18

If the temperature of the boiling water-bath is above 100.0° C., add 0.1% of isopropyl alcohol to the water for each 0.1° C. of excess temperature. This will reduce the boiling temperature to 100.0° C. It is not important to make this adjustment if the original boiling temperature is not higher than 100.2°C.

3. Scrape down the upper part of the tube with the viscometer-stirrer.

4. Remove stopper from bath well. Simultaneously start the timer and place the tube in the bath, lock into position, taking no more than 5 seconds.

5. Stir the sample with the viscometer-stirrer at the rate of two strokes per second (down and up is one stroke), until the clock reaches 60 seconds, for a total of 110 strokes. Stop with the stirrer in the up position.

6. If an automatic timer is used, set the wire against the stirrer so that the buzzer and shut-off mechanism will be activated at the conclusion of the test.

7. Stop the buzzer by pushing the small top knob, which controls contact wire, counterclockwise.

12/ 14.0-percent moisture basis.

8. Record the time in seconds.

9. Quickly remove the test tube from the bath and insert a stopper or another empty test tube to prevent water evaporation.

10. Hold the tube under cold water and remove stirrer. The starch gel is easily removed from the tube when cold by means of a spatula with an extended handle. Clean the stirrer with a test tube brush. Use an air jet to remove any remaining gel and water from the orifice.

11. If the falling number is determined on an "as is" moisture basis, convert to 14-percent moisture basis using the formula:

$$\text{Falling Number* (as is)} \times \frac{100-14}{100-\text{moisture }(\%)} = \text{Falling Number (14\% moisture basis)}$$

* Refer to Source No. 8, Moisture Content of Sample.

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Fig. 1

Relationship Between Bath Temperature and Falling Number

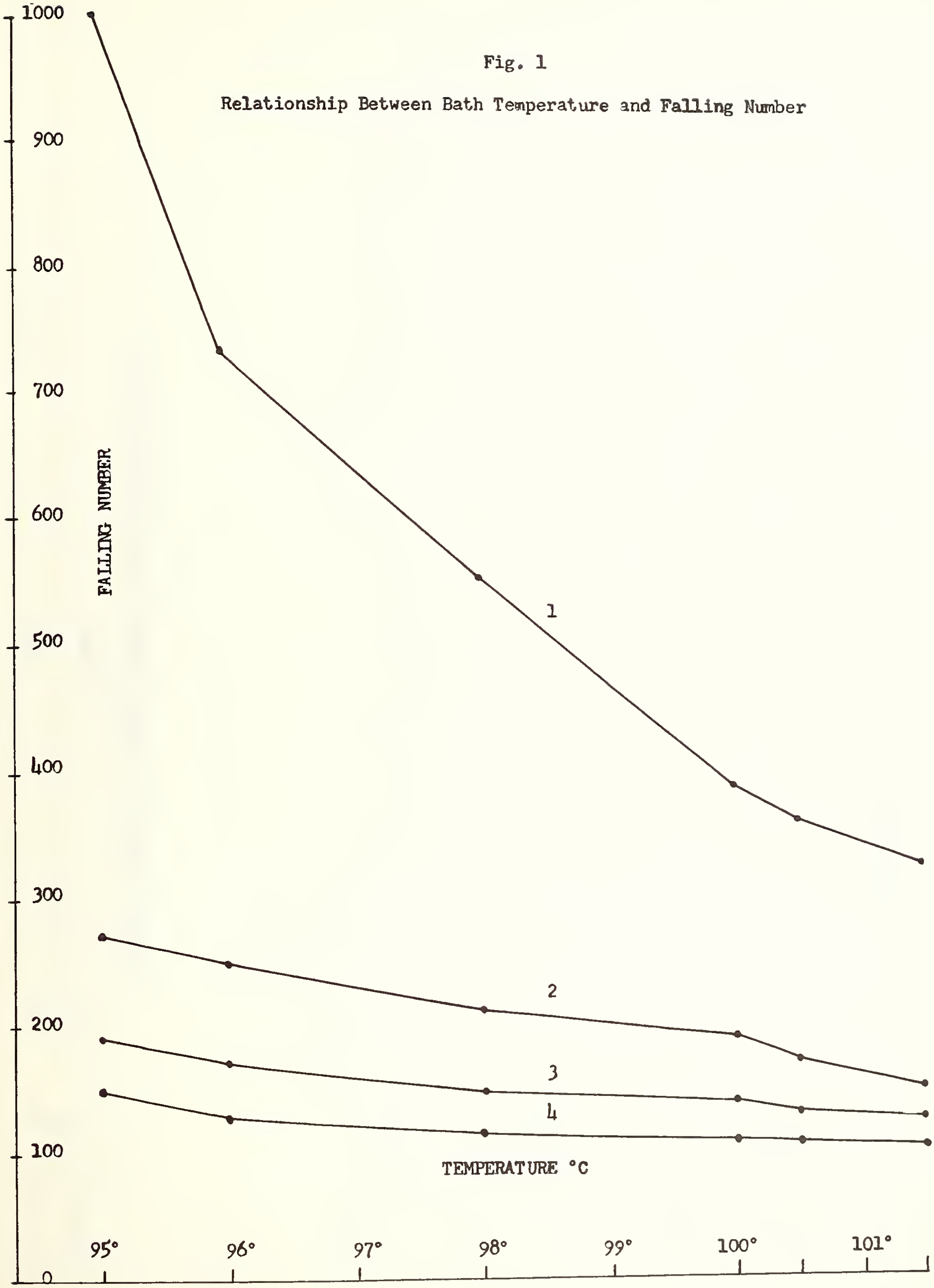
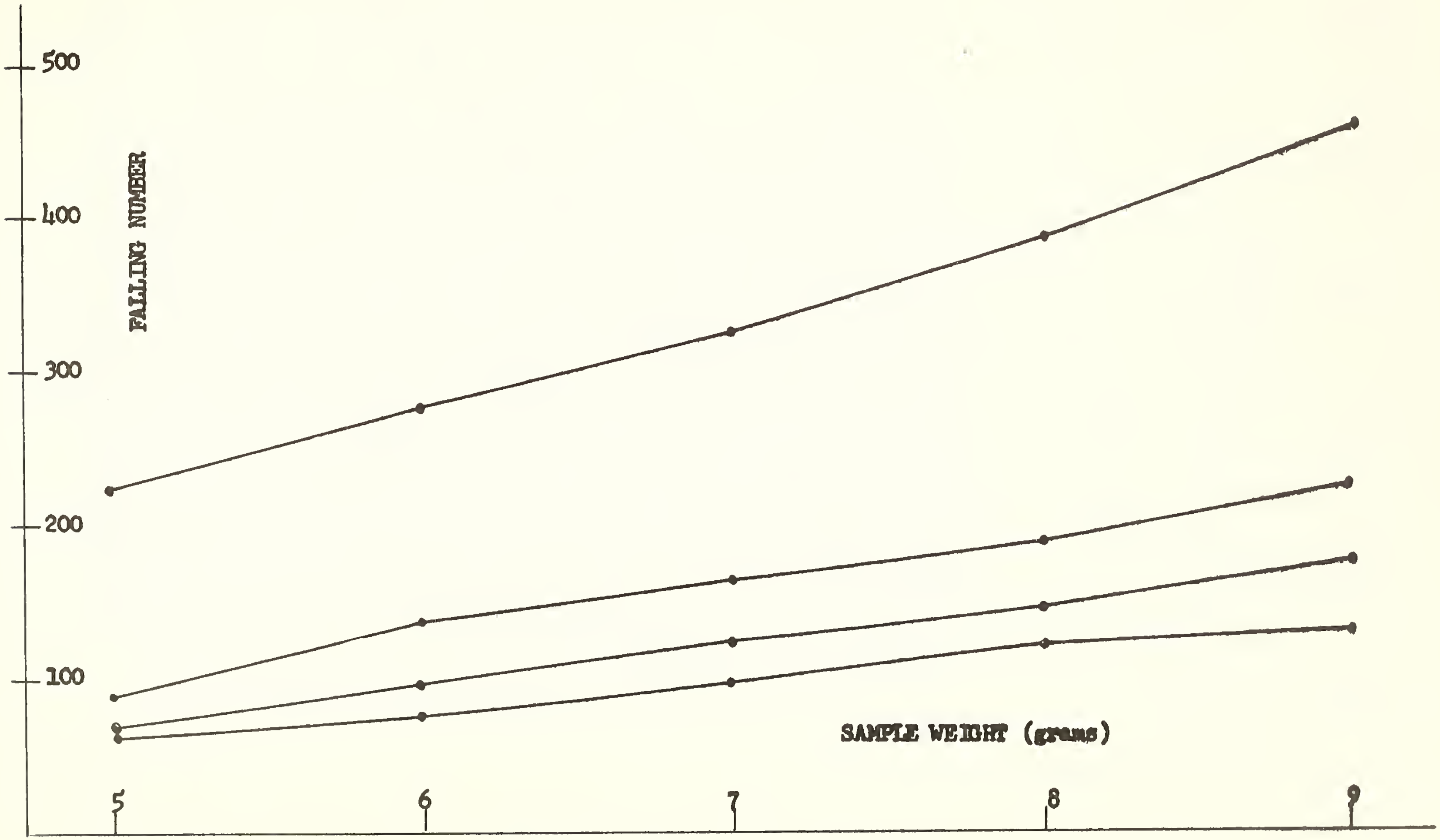


Fig. 2

Relationship Between Sample Weight and Falling Number



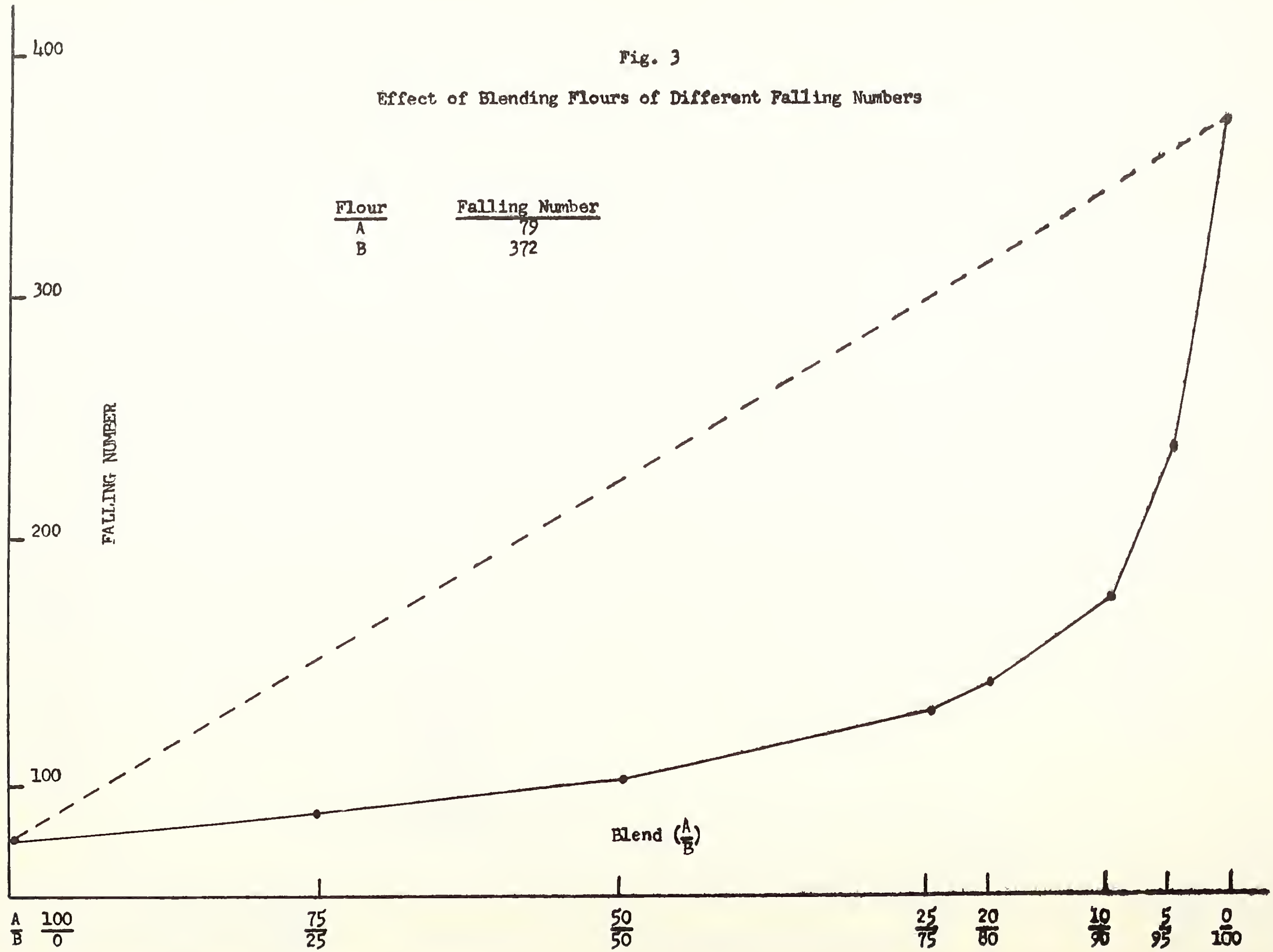


Fig. 3

Effect of Blending Flours of Different Falling Numbers

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