

TABLE III

First Cut Linters		
Sample	A.O.C.S. Standard	Proposed Procedure
1.....	78.7	78.4
2.....	80.1	80.4
3.....	82.0	82.6
4.....	81.4	81.3
5.....	82.0	81.7
6.....	82.5	81.6
Average.....	81.1	81.0
Second Cut Linters		
1.....	69.8	69.8
2.....	70.3	69.8
3.....	70.3	71.4
4.....	68.9	68.6
5.....	68.6	68.9
6.....	69.5	69.5
7.....	70.1	69.8
8.....	72.3	72.6
9.....	72.0	72.6
10.....	71.2	71.2
11.....	67.2	66.6
12.....	68.6	68.6
13.....	69.5	69.5
14.....	69.5	69.2
15.....	69.3	69.9
Average.....	69.8	69.9

In order to be sure that this clarification of the procedure does not alter the values obtained with high yield linters, the following table shows the yield results obtained with the standard A.O.C.S. method and with the recommended procedure.

No differences were found between the two procedures with the high yield linters, but the proposed procedure can be used for both high and low yield linters and hull fiber whereas the Standard A.O.C.S. procedure, as now written, cannot be used without some clarification on the low yield celluloses.

### Recommendations

We recommend that the proposed procedure, as outlined above, be adopted this year so that it can be used as soon as possible to clear up the discrepancies which are obtained at times by some laboratories on the low yield materials.

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## Sieve Analysis of Ground Soybeans and Soy Flour<sup>1</sup>

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THE strong tendency to agglomerate, exhibited by soy flour, particularly those samples finely ground or containing oil, has prevented the satisfactory use of mechanical sieving to determine the particle size distribution.

Methods used in the past, when it has been necessary to get at least an approximate measure of the particle size, have included brushing the sample through sieves with a soft brush, washing the sample through with a liquid, usually carbon tetrachloride, and combinations of these two techniques. If an operator carefully standardizes his procedure, he can obtain consistent results on the coarser screens. As an example of the difficulties encountered in attempts to use finer screens however, three replicates of soy flour brushed through a 200-mesh sieve showed 18.8, 14.5, and 13.4% retained on the sieve. Other replicates of the same flour treated by a combination of washing and brushing showed 19.2, 12.9, and 15.0% retained. These data are in agreement with the statement of the Subcommittee on Soy Flour Sieving Methods that "the commonly-used brushing or shaking methods are not satisfactory" (1).

During testing of washing methods a procedure was developed which has given acceptable, though not perfect, results. The apparatus (Figure 1) consisted of an aluminum sprinkler, such as is commonly used in the home laundry, connected by Tygon tubing to a 4-liter aspirator bottle in which air pressure was controlled by a finger placed over a vent in a compressed air line. The sprinkler was mounted above a 10-inch glass funnel which collected the used liquid and discharged it into a container. The operation should be carried out in a hood or in a well-ventilated place.

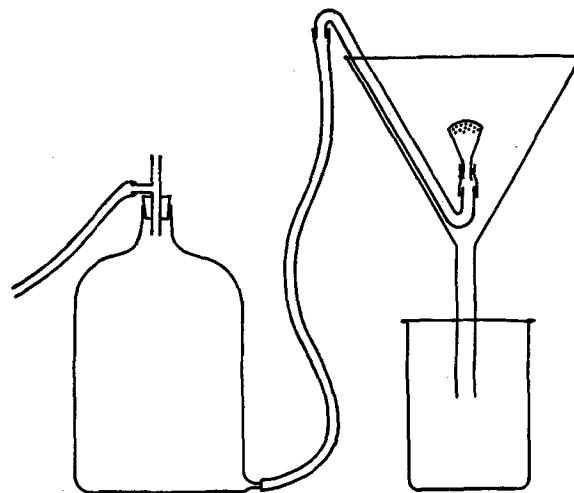


FIG. 1. Sieve-washing apparatus.

For the determination, two-gram samples of the ground soybeans or flour were suspended in 50 ml. of carbon tetrachloride and, unless low in fat, were allowed to stand 30 minutes. Lumps were broken with a stirring rod, and the sample transferred with additional liquid to a standard three-inch sieve. The sieve was held over the sprinkler and the sample was washed by directing a spray of carbon tetrachloride against the bottom of the screen with enough force to cause the liquid to penetrate the screen but not enough to cause splashing over the top of the sieve. After having been washed with from two to four liters, the residue was transferred to a Selas XFF crucible, dried at 100°C. for one-half hour, cooled, and weighed. (The Selas crucible was used because it can be cleaned by ignition.)

Data obtained in comparing two laboratory mills are presented in Table I to illustrate the results produced by the method. The hammermill was one de-

<sup>1</sup> Presented at the San Francisco fall meeting, American Oil Chemists' Society, Sept. 26-28, 1950.

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TABLE I

Grain	Grind	Retained on Screen		
		325-mesh %	270-mesh %	100-mesh %
Soybeans	Hammermill .020" slots	31.8	27.0	10.3
		30.5	29.0	10.3
		31.0	29.0	10.9
		31.3	28.6	11.0
		30.9	29.0	.....
	Ave.	31.1	28.5	10.6
Soybeans	Hammermill .027" holes	19.7	16.0	3.1
		18.4	16.6	3.2
		18.5	16.1	3.5
		18.3	16.9	3.6
		18.6	17.0	.....
	Ave.	18.7	16.5	3.4
Soybeans	Attrition mill Position 2	33.0	31.4	25.1
		33.9	32.2	24.8
		33.6	33.3	25.6
		34.0	31.8	26.7
		33.0	31.8	.....
	Ave.	33.5	32.1	25.6
Soybeans	Attrition mill Position 5	42.6	44.1	37.8
		42.0	43.9	37.2
		44.6	42.2	38.2
		43.6	43.5	36.3
		42.3	42.2	.....
	Ave.	43.0	43.2	37.4
Soybeans	Attrition mill Position 15	64.9	61.2	55.5
		59.8	61.7	58.0
		62.8	61.9	57.8
		61.0	59.7	55.3
		61.2	61.5	.....
	Ave.	61.9	61.2	56.6
Corn	Attrition mill Position 5	64.8	63.8	53.0
		64.7	63.3	52.9
		65.7	64.2	54.2
		66.1	63.2	53.7
		66.3	64.6	.....
	Ave.	65.5	63.8	53.4
Corn	Hammermill 1/16" screen	61.0	59.0	40.6
		62.2	60.0	39.0
		62.1	59.8	39.7
		61.8	58.9	39.7
		62.4	59.9	.....
	Ave.	61.9	59.5	39.8
Soybeans	Commercial	12.5	9.3	None
		12.3	9.6	
		11.8	9.2	
		11.7	8.7	
		11.6	9.2	
	Ave.	11.9	9.2	

scribed by Ross and Hardesty (2), and the attrition mill was a laboratory model commercially available. Of the 23 groups of data only four show a range greater than 2%, and, of these, three are on the same sample—the coarsest in the series. The hammermill used with the screen having the 0.027-inch round holes produced the finest grind, and the same mill with the slotted screen produced the next finest. The attrition mill at its finest setting (Position 2) produced essentially as much material passing the 325-mesh sieve as did the hammermill with the slotted screen but contained more material retained by the 100-mesh sieve. The progressive variation in particle size is shown very clearly for the three positions of the attrition mill. The difference between the corn and soybeans ground in the attrition mill at Position 5 presumably reflects the differences in the structure and hardness of the grain although no tests were made to determine if a given setting of the mill could be reproduced. The data in Table I show very definite differences between mills and between grinding conditions in each mill. The commercial soy flour was included for comparison.

The method has disadvantages. It requires a rather large volume of carbon tetrachloride although the amount lost per sample is not excessive. Further washing with an additional seven liters causes a continuing transfer of sample through the screen. Results are reported as a percentage of the original sample although the residue weighed contains less moisture and oil than the starting material. If the material retained by the screen were dried for one hour at 130°C. and analyzed for oil, results could be calculated to any desired basis. For the present purpose the effect of the small amount of residual oil and moisture was considered unimportant. It could be important however in other applications of the procedure. The change in size of particles treated with carbon tetrachloride is unknown.

## REFERENCES

1. Report of the Seed and Meal Analysis Committee, J. Am. Oil Chem. Soc., 25, 319 (1948).
2. Ross, W. H., and Hardesty, J. O., J. Assoc. Off. Agr. Chem., 25, 238-246 (1942).

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## Extraction of Distillers' Dried Grains in a Soybean Solvent Extraction Plant

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**D**ISTILLERS' dried grains are a by-product of the whiskey industry prepared by drying the slop remaining after the fermented grains have passed through the beer stills. The grains put into the fermentation and distillation process consist of about 75% corn, 15% barley, and 10% rye. The slop is reduced to 5% to 9% moisture by filtration, multi-effect evaporation, and rotary drying. In most cases all of the solid material in the slop is recovered. The principal use of the dried grains is as a constituent in dairy feeds.

The dried grains contain 9% to 10% oil, which originally was in the germ of the grains put into

process. Naturally the oil consists largely of corn oil since this grain constitutes the bulk of those used.

During July, 1938, a submerged marc type extraction plant was completed and put in operation on distillers' dried grains (3). Later however the entire project was abandoned, and the plant was moved to Chile for use on another oil-bearing material (1). Since that time there have been considerable improvements in methods of extraction and solvents used. Also the feed markets have come to accept extracted meal to a much greater extent than in the earlier days of solvent extraction. More recently, laboratory and pilot plant studies have been made on the extraction of dried brewers' grains, but in this case the oil recovered was not of edible quality (2).

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