

follows: saturated acids 9.0% (7% in Table III); oleic acid 46.5% (44% in Table III); total linoleic acid 44.3% (49% in Table III); conjugated linoleic acid 6.8% (12% in Table III). The fact that conjugated linoleic acid recovery and also total linoleic acid recovery is about 5% low is probably due to polymerization of this amount of the conjugated ester during the longer period of heating at the higher pot temperatures involved in the detailed fractional distillation. The agreement of the two sets of figures as given above is fairly good and is even closer if allowance is made for the loss of 5% of conjugated linoleic acid by polymerization.

The main points which the fractional distillation reveals are that saturated acids are mostly palmitic, that there may be approximately 1% of palmitoleic acid, and that the conjugated linoleic ester is somewhat higher boiling than the normal C<sub>18</sub> esters and can be separated and concentrated by fractional distillation. The detailed fraction distillation essentially confirmed the previously determined composition of the fat acids of tall oil.

### Summary

The composition of the fat acids of six samples of American tall oil has been determined. They are all quite similar in fat acid composition. The average values were: linoleic acid 48%; oleic acid 45%; saturated acids 7%. There is present approximately

11% of conjugated linoleic acid, probably formed by the action of alkali and heat during the cooking of the pulp from which the tall oil was formed.

Detailed fractional distillation of a sample of the methyl esters of the fat acids showed that the saturated acids are mostly palmitic, that there may be about 1% of palmitoleic, and that the conjugated linoleic acid present can be separated and concentrated by fractional distillation.

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## Report of the Soybean Analysis Committee 1944-45

THIS Committee carried out collaborative analyses for oil on seven samples during the past year. In addition, several members of the Committee carried on extensive investigations of the various factors influencing the determination of oil. The results of the collaborative tests are listed in Table 1. These analyses for oil content were carried out in accordance with the amplified official A.O.C.S. method as published in Oil & Soap, 21, 306 (1944). Samples Nos. 11, 12, 13, and 1945-1 were whole soybeans which each collaborator ground in his own laboratory. Portions of Sample 1945-1 were dried, ground in a hammer mill at one laboratory, and then sent to each collaborator. The results are shown in columns 6, 7, and 8 of Table 1.

The standard deviation between collaborators ranges from 0.18 to 0.09 for the four samples of whole soybeans. This indicates very good reproducibility of the method in the hands of these seven laboratories. When the variables introduced by grinding and regrinding are eliminated, however, the standard deviation for a 2-gram sample is 0.07 and for a 5-gram sample is 0.04. This latter value is a goal to aim for. Consideration of the results in Table 1 led one of the Committee, F. I. Collins of the U. S. Regional Soybean Laboratory, to carry out some experiments on grinding which are summarized in Table 2. Most of these results represent the average of 16 separate oil determinations (four samples of each of four varieties of soybeans). The results in

Table 2 indicate again the importance of fine initial grinding, and show that good regrinding is essential for coarsely ground soybeans. Table 2 also shows that if the initial grinding is sufficiently fine, regrinding may not be necessary.

Elimination of the regrinding operation would speed up the determination of oil, reduce greatly the labor involved, and, as shown by Table 1, improve the reproducibility of results, especially if 5-gram samples were used for extraction. Although repeated efforts have been made, it has been impossible to devise a method of specifying objectively the fineness of initial grinding necessary to eliminate regrinding. As Table 2 shows, any one of several types of mills

TABLE 2  
Per Cent Oil Content (14% Moisture Basis) as Affected by  
Grinding and Regrinding

Mill	Strokes Regrinding		No Regrind
	200	100	
Wiley (2-mm.).....	18.99	18.82	.....
Wiley (1-mm.).....	18.90	18.78	.....
Wiley (35-mesh).....	19.02	18.92	.....
Hammer.....	19.08	19.04	18.87
Bauer.....	19.06	19.02	18.88
Arcade.....	19.12	19.11	19.10

will grind satisfactorily, but these samples were ground by those skilled in the adjustment and operation of the various mills used. Of these mills, only the Wiley and hammer types require no adjustment by the operator. The Wiley mill does not grind suffi-

TABLE 1  
Per Cent Oil Content (14% Moisture Basis). Collaborative Samples 1944-45.

Collaborator No.	Whole Soybeans				Ground Soybeans Sample No. 1945-1			Average
	Sample No. 11	Sample No. 12	Sample No. 13	Sample No. 1945-1	Regrind	No Regrind	No Regrind 5-gram	
3.....	17.98	17.43	19.27	17.34	17.30	17.21	17.22	17.68
11.....	17.96	17.20	19.40	17.52	17.48	17.39	17.23	17.74
34.....	18.05	17.36	19.29	17.30	17.47	17.34	17.29	17.73
43.....	18.22	17.49	19.52	17.21	17.39	17.27	17.20	17.76
50.....	18.32	17.46	19.53	17.12	17.26	17.20	17.18	17.72
51.....	18.07	17.49	19.33	17.24	17.44	17.35	17.18	17.73
63.....	.....	.....	.....	17.30	17.34	17.21	17.17	.....
73.....	17.80	17.39	19.36	17.53	17.60	17.32	17.17	17.74
81.....	18.35	17.60	19.40	17.32	17.30	17.18	17.20	17.76
Average.....	18.09	17.43	19.39	17.32	17.40	17.27	17.20	.....
Maximum.....	18.35	17.60	19.53	17.53	17.60	17.39	17.29	.....
Minimum.....	17.80	17.20	19.27	17.12	17.26	17.18	17.17	.....
Standard deviation.....	.18	.11	.09	.13	.10	.07	.04	.....

ciently fine. One improved type of hammer mill has been tested by the chairman, and unfortunately in its present form this mill clogs too quickly to be used successfully for the routine grinding of dried soybeans. Further work on this type of mill should be carried out.

If the average results on the four portions of Sample 1945-1, analyzed by each collaborator, are compared, it will be noted that when 5 grams were extracted without regrinding, 17.20% oil was found as compared with 17.40% with regrinding. These two values are the extremes of the averages on this sample. It seems quite possible that the difference of 0.20% between these values may represent material which is not truly oil and which is removed by petroleum ether because of the exposure to moist air during the regrinding. This explanation should be studied.

This year's work has shown therefore that:

1. The present official A.O.C.S. methods for oil in soybeans, in the hands of experienced analysts, has a standard deviation of from 0.1 to 0.2.

2. Improvements in reproducibility may be obtained by eliminating the regrinding and increasing the size of the sample.

At the present time no change in the present official methods can be justified. It is recommended that future work to improve the methods be carried out as follows:

1. Study the use of a grinding mill which will eliminate regrinding and which need not be adjusted by the operator.

2. Study the composition and nature of the additional material which is extracted by petroleum ether after regrinding a sample which initially was very finely ground.

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## Report of the Cellulose Yield Committee 1944-1945

DURING the past year linter samples were sent out four times to ten laboratories. Two of the laboratories received only three sets of samples. The following table gives the averages of all the samples sent out:

Lab. No.	No. Sets Samples Tested	Samples			Overall Average for Year
		A Linters	B Linters	C Fiber	
1.....	4	78.6	72.5	70.8	74.0
2.....	4	78.5	72.6	70.9	74.0
3.....	4	78.6	72.7	71.0	74.1
4.....	4	78.5	72.4	71.5	74.1
5.....	4	78.9	71.8	71.0	73.9
6.....	4	79.1	72.5	71.3	74.3
7.....	4	78.6	72.7	70.8	74.0
9.....	4	79.0	73.6	72.3	75.0
10*	3	78.8	73.2	70.3	.....
11.....	4	79.1	72.6	71.3	74.3
12.....	4	78.9	72.4	71.2	74.2
13*	3	78.5	73.8	70.6	.....
Avg.....	.....	78.8	72.6	71.2	74.2

\*Three sets run, not included in average.

Laboratory No. 8, which is missing from the group, did not participate in the check samples during the past year.

It is noted that with the exception of laboratory No. 9 the averages of all three sets are very close.

We still believe that it is worthwhile to send these samples out three or four times a year in order to be sure that all laboratories are keeping their equipment in proper shape.

### Recommendations:

It is recommended that a sample be sent out at least four times during the next year to all laboratories who are equipped to run the tests and who desire to get in on the check analysis.

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