Report of the Soybean Analysis Committee 1938-1939

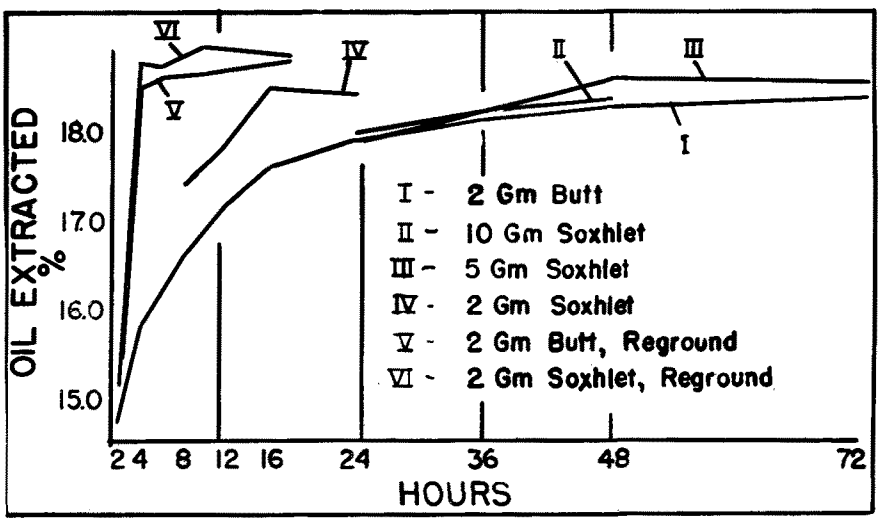
BEFORE adopting any method of analysis for soybeans as official, it is necessary to examine in some detail the factors which may influence the various determinations. From such a study it should be possible to select methods having the accuracy required by the referee chemist in a well-equipped laboratory, which are at the same time simple and rapid enough to be used by the control chemist either in a plant or a small laboratory where special equipment is not available. Unnecessary work or details should be avoided, and previous experience gained with other oil seeds should be examined with reference to its applicability to soybeans.

The determination of oil content of soybeans is probably the most important analysis made on this material. The chief factors which may affect this determination are: (1) Solvent; (2) apparatus used; (3) grinding of sample; and (4) pretreatment of sample.

(1) *Solvent.* The American Oil Chemists' Society recognizes, in its present official methods, only one solvent for fat extraction. This solvent is composed chiefly of normal hydrocarbons containing about 80 percent pentane and 20 percent hexane. It was selected on the basis of careful work reported in Oil and Fat Industries, *8*: 311 (1931). It is firmly entrenched and will probably remain in general use unless strong reasons are advanced for its abandonment. One disadvantage of this solvent is that it is a mixture, and its composition is subject to change on distillation or on long storage. For fat extraction some pure hydrocarbon such as hexane or heptane may offer advantages. No work, however, has been done with respect to the use of other solvents.

(2) *Apparatus used.* The Soxhlet type apparatus is probably in most general use, although the Butt type extractor is cheaper and the simplest of those commonly in use in the oil industry. Figure 1 shows a comparison of the rates of extraction of oil from 2-gram samples of soybeans in a Butt type extractor and from 2-, 5-, and 10-gram samples in a Soxhlet type extractor. Soybeans of approximately 6 percent moisture were ground in a Wiley mill and extracted without regrinding with the results shown in Curves I, II, III and IV. These results as well as all others given in this paper are averages of two or more closely agreeing determinations. Extraction in the Soxhlet apparatus removes oil somewhat more rapidly than extraction in the Butt apparatus, as can be seen by comparing Curves I and IV. However, the total amount of oil extracted was the same in both cases.

FIGURE 1



(3) *Grinding of sample.* The present tentative method requires regrinding of the sample in a mortar after a two-hour extraction and then extracting for three additional hours. The regrinding is a slow and quite laborious process, and efforts have been made to avoid this operation. The results shown in Curves V and VI (Figure 1), when compared with those of Curves I and IV, indicate the need for regrinding. The fineness to which the sample was ground originally has considerable influence on the necessity for regrinding. Grinding of soybeans on three types of mills was studied, and the results of the corresponding sieve analyses are shown in Table I. The moisture content of the beans prior to grinding had no influence on the relative efficiencies of the three mills, but the moisture content of the ground beans was altered by grinding in both the Bauer and coffee mills. The Wiley mill, which is designed to cut rather than grind, was used with a screen having 1-mm openings. The coffee mill used was of the customary type and was set with grinding discs just in contact with each other; the Bauer mill (3600 r. p. m.) was of standard type and requires no description. Three settings of the Bauer mill were used, namely, B_1 where the grinding discs were barely in contact, B_3 where the clearance was .003 inches, and B_{11} where the clearance was .011 inches.

The sieve analyses were made on extracted soybean meal and are not absolutely accurate owing to the tendency of the finer sizes to ball up during sieving. However, it is apparent that no marked difference exists between grindings made with the B_1 and B_3 settings, and in subsequent determinations these two settings gave the same results. Through the kindness and cooperation of Dr. Lawrence Zeleny of the Bureau of Agricultural Economics, it was also possible to use another type of mill. Soybeans were ground on an experimental flour mill having differential speed rolls with forty corrugations to the inch. The beans were coarsely

TABLE I.
SIEVE ANALYSES OF GROUND SOYBEANS

Grinding	Ground soybeans passing through sieve — U. S. Standard Mesh				
	20 mesh	35 mesh	60 mesh	80 mesh	100 mesh
	Percent	Percent	Percent	Percent	Percent
Wiley 1-mm	100.0	71.0	33.3	24.6	18.0
Coffee mill	99.0	93.5	74.4	53.0	41.6
Bauer B_1	99.7	96.8	73.3	62.6	53.3
Bauer B_8	99.6	94.4	67.2	51.9	37.8
Bauer B_{11}	98.7	63.6	39.6	32.2	16.6

TABLE II.
PERCENTAGE OF OIL IN SOYBEANS CALCULATED TO MOISTURE-FREE BASIS

	Wiley 1-mm Butt 2-g sample Reground 4 hrs.	Bauer B_1 Butt 2-g sample Reground 4 hrs.	Bauer B_8 Soxhlet 10-g sample 20 hrs.	Rolls Butt 2-g sample Reground 4 hrs.	Rolls Butt 2-g sample 20 hrs.	Rolls Soxhlet 10-g sample 20 hrs.
	Percent	Percent	Percent	Percent	Percent	Percent
1	18.70	18.93	18.72[1]			18.73[1]
2	20.80			21.20	21.26	21.06
3	18.51			18.60	18.43	18.22
4	15.88			16.26	16.29	15.94

1 Analysis by Dr. Lawrence Zeleny's laboratory.

TABLE III.
EFFECT OF PRETREATMENT ON THE PERCENTAGE OF OIL EXTRACTED, CALCULATED TO A MOISTURE-FREE BASIS

	No treatment	Preheated and predried 2 hrs. -130° C. (2.4 pct. H_2O)	Preheated only 2 hrs. -130° C. (6.0 pct. H_2O)	Predried only — desiccator (2.6 pct. H_2O)	Predried in desiccator then preheated 2 hrs. -130° C. (2.0 pct. H_2O)
	Percent	Percent	Percent	Percent	Percent
Wiley 1-mm	18.70	19.00	18.70	18.08	19.00
Bauer B_8	19.05	19.13	19.27	18.26	18.65
Bauer B_{11}	18.06	19.04	19.09	18.38	18.71

ground on a coffee mill and then reground by passing through the roller mill three times. The results of the oil extraction obtained with soybeans ground by the various methods are given in Table II.

The precision attained at any one time may be judged from the fact that all of the twelve extractions of 2-gram samples in the Butt apparatus, followed by regrinding and reextraction, gave between 18.61 and 18.77 percent of oil. As can be seen from Table II, it is necessary either to regrind during the extraction or to continue the extraction for at least 20 hours. Even with the finest grindings shorter periods of time gave lower results than were obtained with the regrinding procedure.

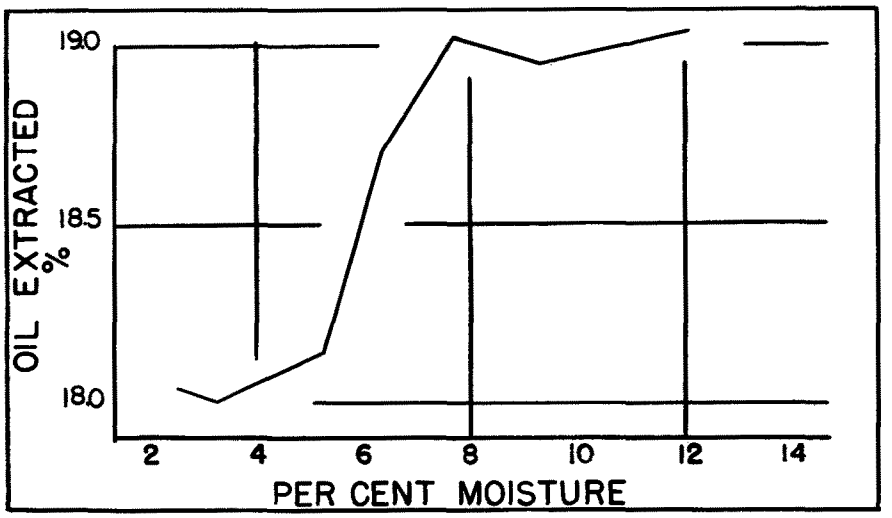
(4) *Pretreatment of sample.* The previous history or treatment of the soybean sample may have an effect on the quantity of oil extracted. In the present tentative method it is required that the beans be dried at 130° C. before grinding. The results in Table III show the effect of this treatment (Column 3), as well as those of preheating in a closed container without drying (Column 4), predrying at room temperature without heating (Column 5), and predrying with subsequent heating (Column 6), which were studied on one sample of soybeans.

The moisture content of the untreated samples was 6.3 percent when ground in the Wiley mill and 5.2 percent and 6.5 percent when ground in the Bauer mill at settings B_8 and B_{11}, respectively. As may be observed from the results in Table III, preheating alone tends to raise the apparent oil yield whereas, in general, predrying at room temperatures appreciably lowers it. The latter point was investigated in more detail, using samples ground on the Wiley mill, with the results shown in Figure 2. The samples used for this work were not heated, and, as can be seen, a difference of 1 percent in apparent oil content is found in going from 5 to 8 percent moisture content. One explanation of this apparent increase in the yield of oil with increasing moisture of the sample is that increased removal of phosphatides results when the oil is extracted from samples of high moisture content. In the above samples the oil extracted at 5.3 percent water content contained .0013 percent phosphorus, while the oil extracted at 12.1 percent water content contained .0178 percent phosphorus. The difference in phosphorus content, computed in terms of lecithin, is not sufficient to account for the increased oil yield, but it does suffice to show that the material extracted at high moisture content is not identical with that extracted at low percentages of moisture.

CONCLUSION

In considering the results obtained in this study it would appear that, for the referee laboratory or control chemist in the plant working on commercial samples, the present tentative methods will introduce no appreciable errors in the determination of the oil content of soybeans. For the research laboratory in plant physiology, the agronomist, or the geneticist, the use of a Bauer mill and the heating and drying of the sample before extraction cannot be followed since, in many cases, the maximum weight of the sample available may amount to only 10 to 20 grams. In any case, to obtain complete extraction of the oil from soybeans in four or five hours, it is necessary to regrind after partial extraction. These results are in agreement with those of McKinney, Cartter, and Jamieson, Oil and Soap, *11;* 252 (1934).

FIGURE 2



In conclusion, it is recommended that collaborative work be done on (1) the influence of moisture on the yield of oil extracted, (2) the fineness of grinding which is necessary to obtain complete extraction without regrinding, and (3) the nature of the constituents removed with the oil when extractions are carried out with soybeans having a high moisture content.

T. L. Rettger
S. O. Sorenson
M. M. Durkee
R. T. Milner, Chairman

SAMPLING COMMITTEE REPORT

THE Sampling Committee presents the following report covering the work done in the past year. While the committee has been very busy during the year, no work has been finished to the point where we can make definite recommendations, and this will be definitely a progress report only.

The Sampling Committee has been organized with the following members: Messrs. Victor Serbell, Procter Thomson, A. D. Rich, G. A. Crapple, V. C. Mehlenbacher, H. H. Mueller, P. W. Tompkins, and H. P. Trevithick, Chairman.

Most of the New York members had a meeting at Mr. Serbell's office, September 21, 1938, and discussed sampling rules in general, as applied to fats and oils only. We did not consider cottonseed, meal, nor cake, although these materials may be taken up later.

There has been considerable discussion regarding soap stock sampling, and the committee has definitely voted to recommend writing two sets of rules, by divorcing raw soap stock and acidulated soap stock.

Acidulated soap stock offers no particular difficulties, and can be handled both before and after loading, by any of the methods which are satisfactory for any fat.

Raw soap stock, however, is difficult to melt, it coagulates into lumps, is liable to ferment, etc., all of which renders it practically impossible to sample satisfactorily, after the material has been in the container, whether drum or tank car, any length of time. The committee would probably recommend that the only satisfactory time for sampling soap stock is when it is being loaded.

Our methods for soap stock sampling are very brief, but provide for sampling by the "dip" method and by the "bleeder" method. The committee by correspondence definitely decided to modify both of these methods somewhat, and hoped to have a definite report for this meeting. However, it later developed that the accuracy of "bleeder" sampling was questioned. It was claimed that even after going through the pump, soap stock contains both very fine material, and also lumps of hard material. These lumps will plug the bleeder unless the opening is very large. Upon opening the valve to release the lumps, very thin material will flow through rapidly, and destroy the accuracy of the sample. This question is now being investigated by the committee, and if this criticism proves justified, we will probably recommend that the bleeder method be eliminated entirely.

There has also been discussion of the question of sampling tank cars containing free water and/or sediment, and also of the use of the two inch tryer, since a two inch core of a tank car does not allow anything for the varying width of the tank car. The committee is investigating the accuracy of the two inch tryer sampler, and also a number of other samplers, comparing the results given with that obtained from the same tank car after unloading and mixing in scale tanks. The committee has further investigated a number of samplers for use with barrels or drums.

The committee has also obtained the methods of sampling used by the British Standards Institution, etc. It is very interesting to know that the British methods of sampling, developed without any cooperation between the two countries, are very similar to ours.

It may be that we can finish some of our work for submission at the Fall Meeting.

While this is a progress report only, the committee has done a considerable amount of work, and should be able to present a good report later.

The Chairman would like to express his appreciation of the active and interested cooperation of all the committee.

H. P. Trevithick, Chairman
Sampling Committee, A.O.C.S.

Report of the Referee Board

FOR the year 1938-9 the Referee Board granted 33 Referee Certificates. Five check samples of crude cottonseed oil were distributed and ten check samples of cottonseed were sponsored. Law and Company as usual prepared and distributed the seed samples. This year Mr. R. T. Doughtie of the Bureau of Agricultural Economics tabulated the reports on each of the seed samples, thereby greatly lightening the burden of work of the Referee Board. There were as many voluntary collaborators as referee chemists receiving each series of samples. The board has no recommendations to offer and no report to make beyond giving this account of its activities and expressing its appreciation of the cooperaton of everyone concerned.

C. H. Cox
N. C. Hamner
J. P. Harris
J. J. Vollertsen
A. S. Richardson, Chairman.