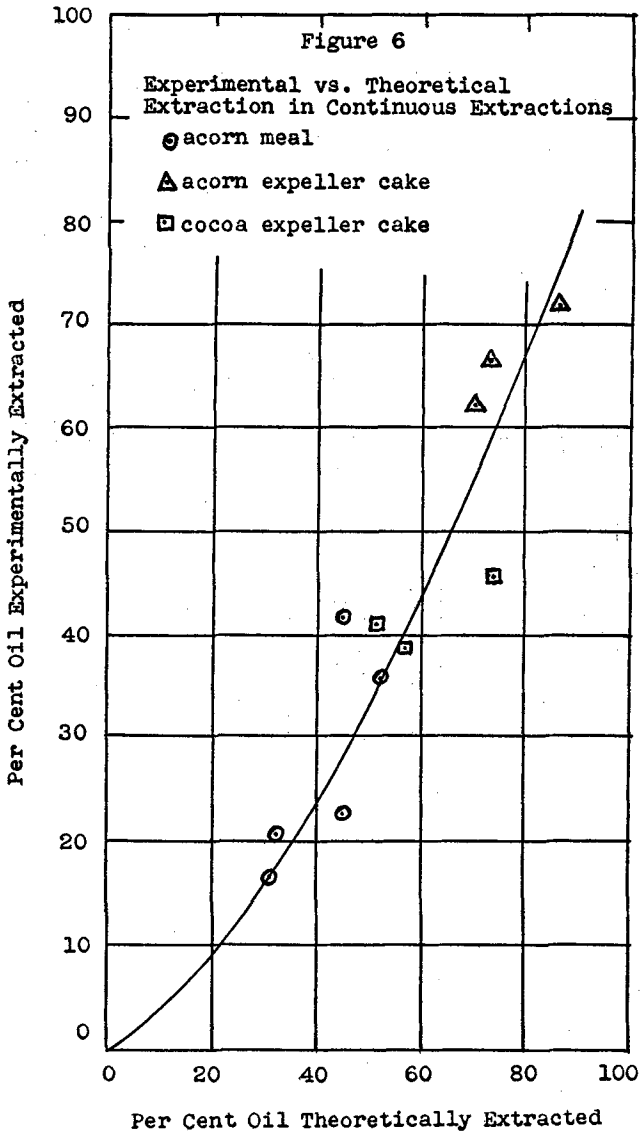
terion for the prediction of the results of an oil extraction. Solvent-extraction theory, however, can be used successfully to predict the results of such extractions. Experimental vs. theoretical extraction is plotted in Fig. 6 and this figure shows that this theory is capable of predicting the results of such extractions as those of acorn oil from acorn meal and acorn expeller cake as well as the extraction of cocoa butter from cocoa bean expeller cake. A comparison of the results for extraction of cocoa butter and acorn oil shows that, if the same extractor is operated under similar conditions on these materials, there are the same number of theoretical stages in that extractor whether the feed is acorn meal, acorn expeller cake, or cocoa bean expeller cake.



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Report of Seed and Meal Analysis Committee

Methods of Analysis of Soyflours

THE Seed and Meal Analysis Committee has been directed by the Governing Board of the Society to investigate and recommend methods for the determination of moisture, oil, nitrogen, ash, crude fiber, water absorption, phosphatides and particle size in soyflours. The initial step taken was to conduct a survey of experiences in the analysis of soyflours to direct the action of the Committee. The work, considerations, and recommendations regarding the first five of these methods are given in this report.

Moisture and Volatile Matter

It is recognized that oven moisture values do not actually represent moisture but a combination of moisture and volatile matter influenced by decomposition and oxidation that may occur under conditions of the test. For this reason the method is empirical and must be specific and followed rigidly. Majority of practices and experiences, revealed by the survey, indicated the satisfaction of the use of the forced draft oven (A.O.C.S. Specification H 1-39) and an oven temperature of 130° C. Two combinations of sample weight and time of heating have been used, namely, 2 grams for one hour and 5 grams for 2 hours. This point was studied collaboratively by four laboratories using samples of each of full fat, low fat, and de-fatted soyflours and heating at 130° C. in the forced draft oven. Observations made from the results reported are as follows:

1. For individual periods of heating, varying from 30 minutes to 2 hours, there was but little difference in the results obtained on 2- and on 5-gram samples.
2. A low rate of loss which can be tolerated in approaching a constant weight appears to occur at or soon after one hour of heating.

3. The results obtained by drying in the forced draft oven with 5-gram samples heated at 130° C. for 2 hours appeared to be reasonably near the true moisture content. They checked reasonably well with the Fischer volumetric values. However, the apparent residual moisture by the Fischer method in the dried samples plus the oven-drying results equalled more than the original Fischer values. The presence of volatile matter, decomposition and oxidation is offered as an explanation.

Additional work done in one of the laboratories showed for the same soyflours from 1.2 to 1.7% of residual moisture by the Fischer method in 5-gram samples dried at 101° C. in a Brabender forced draft oven and from 1.0 to 1.6% of residual moisture by the same method in 5-gram samples dried in a vacuum oven at 101° C. at about 20 inches of mercury for 6 hours.

It is recommended by a majority vote of the committee that the following method be adopted as tentative for the determination of moisture and volatile matter in high fat, low fat, and de-fatted soyflour:

Weigh accurately and as rapidly as possible a 5-gram portion of the well mixed sample of soyflour into a tared official moisture dish. Slip the cover on the bottom of the dish and place the uncovered dish in the forced draft oven (A.O.C.S. Specification H 1-39) and dry at 130° C. for 2 hours, timing from return of the oven to 130° C. after the dish and sample have been placed therein. Remove the dish and sample from the oven, cover immediately, cool in a desiccator containing an efficient desiccant (A.O.C.S. Specification H 9-45) and weigh. Calculate and report loss in weight as percentage of moisture and volatile matter.

Oil

The A.O.C.S. official method for the determination of oil in cottonseed and oilseed cake, meal, and meats, with some variation in size of sample and period of extraction has been used by the soyflour trade with considerable satisfaction for some period of time and with agreement on these points should continue to serve the trading and inspection needs. The influence of time of extraction is shown by some data given in Table 1.

TABLE 1.

Influence of Time of Extraction on the Butt Extraction of Oil from Soyflours.

Soyflour	Weight Sample	Hours Extracted			
		3	4	5	6
	g.	%	%	%	%
Full fat	2	20.14	20.27	20.38	20.26
Full fat	5	20.20	20.16	20.40	20.22
Low fat	5	4.84	4.83	4.80	4.82
De-fatted	5	0.81	0.76	0.80	0.79

These data and experience in the use of the method demonstrate that the extraction is essentially complete at the end of three hours and that an additional hour or two assures precision and reproducibility.

It is the majority vote of the committee to recommend adoption of the A.O.C.S. official method for determining oil in cottonseed as tentative for the determination of oil in soyflours but specifying an extraction period of 5 hours and the use of a 2-gram sample for full fat and a 5-gram sample of low fat and de-fatted soyflours.

Ash

The determination of ash in such materials as soyflours is subject to some variables which are difficult if not impossible to adequately control. These variables include loss of carbon dioxide from carbonates, volatilization, unequal heating and ventilation of furnace chamber, and retention of free carbon. Hence, the necessity of specific conditions for the test is implied.

The A.O.A.C. method for grains and stock feeds, which specifies the incineration of a 2-gram sample at 600° C. for 2 hours in a muffle furnace with a control pyrometer, has had some use for the analysis of soyflours for ash. Results of some tests of the applicability of this method to soyflours are given in Table 2. A Hoskins furnace with a control pyrometer was used and the ashing dishes employed were porcelain combustion capsules (Coors No. 170, size No. 3). The furnace was heated to specified temperature before the dish and sample were put into it, and the dish and ash were removed after an incineration period of 2 hours.

TABLE 2.

Determination of Ash in Soyflours.

Type of Flour and Temperature	Position in Furnace			Average	Residual Carbon*	Average Carbon-Free Ash
	Front	Center	Rear			
	%	%	%	%	%	%
Full fat						
550° C.	5.11	4.94	5.17	5.07	0.182	4.89
600° C.	4.71	4.65	4.71	4.69	0.038	4.65
650° C.	4.41	4.48	4.61	4.50	0.003	4.50
Low fat						
550° C.	6.00	5.80	5.92	5.90	0.088	5.81
600° C.	5.55	5.36	5.67	5.53	0.033	5.50
650° C.	5.35	5.52	5.43	5.43	0.010	5.42
Fat free						
550° C.	6.16	6.20	6.17	6.18	0.103	6.08
600° C.	5.68	5.71	5.87	5.73	0.043	5.69
650° C.	5.41	5.67	5.64	5.57	0.013	5.56
Fat free at 600° C.						
a	6.11	5.78	5.91			
b	6.05	5.88	5.95			
c	6.05	5.78	5.99			
Average	6.07	5.81	5.92			
Residual carbon*	0.067	0.042	0.048			
Average carbon-free ash	6.00	5.77	5.87			

*Residual carbon calculated on basis of original sample of soyflour.

The ashes from triplicate determinations indicated in Table 2 were composited and digested with dilute hydrochloric acid to eliminate carbonate carbon and dissolve the greater portion of the ash. The solutions were filtered on asbestos in micro sintered glass filters. The residual free carbon was determined by micro techniques.

The ashes obtained at 550° C. were dark and contained considerable free carbon. Those from the 650° C. ashing contained very little free carbon, but their appearance definitely showed that some fusion had occurred. Position in the furnace appears to influence the results. Increase in temperature of incineration reduces the corrected ash values. This is assumed as being due to loss of carbon dioxide and volatilization.

It is well known that ash results may be influenced by the shape and size of the ashing vessels used. Porcelain combustion capsules were used in the above investigations as they were the broadest vessels available that were considered practical. They are low in height and the ash obtained is a thin wafer about seven-eighths of an inch in diameter.

The method is empirical in nature and is recognized as a compromise. It is supported by the collaborative work done by the A.O.A.C. and has already had some general application in the soyflour trade.

It is recommended by a majority vote of the committee that the following method be adopted as tentative for the determination of ash in soyflours:

Weigh 2 grams of the well mixed sample into a previously heated and tared porcelain combustion capsule (Coors

No. 170, size No. 3). Place the capsule and sample in muffle furnace previously heated to 600° C. and maintain at this temperature (±15°C.) for 2 hours with pyrometer control. Transfer capsule to a desiccator containing an efficient desiccant (A.O.C.S. Specification H 9-45), cool to room temperature and weigh immediately thereafter. Calculate and report residue as per cent of ash.

Nitrogen and Protein

The present official method of the Society for the determination of nitrogen, ammonia, and protein in soybeans and cottonseed has had long and satisfactory use. There appears to be no reliable reason for the approval of a method of slightly different basic specifications for soyflour. It is the recommendation of a majority of the committee that the present official method for soybeans and cottonseed and their cake and meals be adopted as tentative for the determination of nitrogen and protein in all soyflours, with the sample weight of 0.7005 to 1.4010 grams and with the results reported as nitrogen or/and crude protein ($N \times 6.25$). The soybean and soyflour trade customarily express the results in this manner and has adopted apparently the food and feed conversion factor of 6.25 for calculating the protein from the determined nitrogen value. Reports should indicate that the protein values are $N \times 6.25$.

Crude Fiber

Crude fiber is specifically defined as the group of organic constituents quantitatively evaluated by strict application of the Official A.O.A.C. Method. This method is empirical and requires strict adherence to its specifications. Values obtained by any modified procedure should not be called "crude fiber" unless it has been demonstrated that they represent the same percentage of the sample and the same group of substances as those obtained by use of the A.O.A.C. method. This method has a legal standing in the food and feed trade requiring its use for inspection purposes.

A unanimous vote of the committee recommends the adoption of the A.O.A.C. crude fiber method as tentative for all soyflours. Work has been initiated for the purpose of developing a method for determining fiber or fibrous materials which will be specific for soyflours.

In considering these five methods of analysis of soyflours the committee expresses a majority opinion that they should be adopted as tentative without further investigative or collaborative work.

The contributions of V. C. Mehlenbacher of Swift and Co., of F. I. Collins of the U. S. Soybean Laboratory, of F. R. Earle of the Northern Regional Research Laboratory, and of Lamont Hagan, L. E. Brown, J. F. Jurgens, and R. R. Mod of the Southern Regional Research Laboratory, in connection with the investigational work reported and referred to, are acknowledged.

In considering these methods L. R. Brown, N. F. Kruse, G. P. Davidson, C. P. Long, W. L. Taylor, F. I. Collins, and L. Zeleny have served as associates of the Committee. Thus, the needs of the soyflour trade and the National Soybean Processors Association were given wider consideration.

Determination of Ash and Crude Fiber in Oilseed Meals

The Governing Board has directed that the Seed and Meal Analysis Committee consider and recommend procedures of analysis for the determination of ash and crude fiber in oilseed meals. State inspection laws specify the official methods of the A.O.A.C. for the evaluation of these commodities. In line with the methods recommended for the determination of ash and crude fiber in soyflours, it is recommended by a vote of the Committee that the A.O.C.S. method for ash in grains and stock feeds and the A.O.A.C. method for the determination of crude fiber be adopted as tentative by the Society for oilseed meals. The background work already done in connection with these two methods, their general use in the feed trade, and the investigations herewith reported on the determination of ash in soyflour, considering the retention of residual free carbon, are offered in support of this recommendation.

Report of the Subcommittee for Cottonseed and Cottonseed Meal

In the 1946 report of this subcommittee it was recommended that, as there was need for a quicker optional method than the official method for moisture in cottonseed, drying whole seed for 2½ hours at 130° C. should be studied. This was done during the fall of 1946.

Eight analysts made duplicate moisture determinations by the two methods on a random sample twice weekly during the seed harvesting season, reporting the two moisture results in duplicate and the F.F.A. content of the seed. The advantage of this program was that analyses were made under normal routine conditions on cottonseed varying geographically, in time of harvesting, and in quality.

In the tabulation of results (Table 3) a number of averages were made. It will be seen that while 2½ hours drying at 130° C. gives slightly higher results, none of the compared averages by all analysts differs by more than .05%. The greatest difference found by an individual analyst was .14%.

The results were split into two groups: (1) Those in which both figures were below 12% moisture and (2) those above 12%. Low moistures ranged down to a minimum of 7.3%; high ones up to 18.5%. (Only the first 20 reports were used from the analyst's data on 89 samples so as to give each analyst's results approximately the same weight.) Averages C and D show no significant difference in comparison between the two methods on low and high range moisture content seed.

It was planned to secure data on deteriorated, or high free fatty acid content seed, but the general good quality of the 1946 crop limited the obtainable data. Average E is on six samples containing from 2.5% to 7.0% F.F.A. The widest difference in moisture found was .2%. The average difference of .04% is about the same as on other groupings. While the amount of data is small there is no indication that seed deterioration, as measured by F.F.A. content, would affect the comparison of the two moisture methods.

On the basis of the data presented herewith, this committee recommends that the A.O.C.S. Official Method Aa 3-38 be supplemented to permit the use of 2½ hours drying at 130° C. when quick moisture determinations on cottonseed are needed. It is not proposed that this procedure be made an official alternative method for the overnight drying pro-

TABLE 3.

Determination of Moisture in Cottonseed.

Overnight Drying at 101° C. vs. 2½ hrs. at 130° C.

Analyst	Oven Type	% Moisture		% F.F.A.	No. Samples
		Overnight 101°	2½ hrs. 130°		
Boulware	Freas 101° DeKhotinsky 130°	10.74	10.67	.9	7
Cox	Despatch 101° Despatch 130°	13.09	13.20	1.0	20
Fix	Despatch 101° Despatch 130°	11.15	11.14	.8	20
Haire	Despatch 101° Freas 130°	12.64	12.72	.5	12
Pope	Precision Freas	10.43	10.57	1.0	15
Smith	Freas 101° Despatch 130°	12.48	12.46	.6	22
Wilkins	Despatch 101° Despatch 130°	11.78	11.75	.7	89
Rettger	Despatch 101° DeKhotinsky 130°	12.22	12.27	.7	15
(A) Avg. of Analysts Avg.		11.82	11.85		8 avgs.
(B) Weighted Average		11.87	11.88		200
(C) Avg. of Results under 12%		10.43	10.46		61
(D) Avg. of Results over 12%		13.78	13.83		55
(E) Avg. of Results over 2% F.F.A.		9.88	9.92	4.0	6

cedure. Therefore this recommendation is referred to the Editor of Analytical Methods for proper wording of the supplement to Aa 3-38.

Respectfully submitted,

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The report and recommendation of the Subcommittee for Cottonseed and Cottonseed Meal have been given a majority vote of approval by the Committee.

Report of Subcommittee for Peanuts and Peanut Meal

It is apparent that the intrinsic value of whole peanuts as an oilseed stock is based on the percentage of kernels obtained on shelling and the composition of the kernels. Hence, in considering tentative and official methods of analysis it is deemed that the analyses should be made on the kernel sample. In consideration of the methods for the determination of first and second moistures, reference is made to the report of Hoffpauir (Oil & Soap, XXII, 283-286). He reported results to show that some change in the moisture content was possible and generally probable when grinding the sample with a Universal food chopper. He also pointed out that it is essential to dry the sample material to the same degree of dehydration in the case of both the first and the second moistures in order to avoid further error, and reported data for moisture loss curves which indicate that a reasonable choice is to dry 50 grams of whole kernels for 3 hours and 5 grams of the predried and ground kernel sample for 2 hours at 130° C. in a forced draft oven.

For two marketing seasons, the Chemists' Committee of the Commodity Credit Corporation specified methods of analysis involving the analysis of whole kernels for moisture by the above-mentioned methods for use in trading with the oil mills. During the past season the Subcommittee on Oilseeds of the Smalley Foundation committee has used these moisture procedures in the analysis of the check samples of peanut kernels sent out under its jurisdiction. On the basis of the research work of Hoffpauir and the experience gained in the use of these moisture procedures during these several seasons, it is recommended that certain changes be made in the methods of the Society, and if these changes are adopted that the methods involved be made tentative for one year before being made official. These recommendations are:

1. That original moisture be determined on the whole kernels by weighing 40-50 grams of them into a moisture dish and drying in a forced draft oven at 130° ± 3° C. for 3 hours, and that where the moisture may be required on shells that a 20-30-gram sample be used following the same procedure as for the whole kernels.
2. That the present method for the determination of oil in whole nuts be deleted and that the method for determining oil in shelled nuts or kernels be revised with respect to the determination of the second moisture to specify that the second moisture shall be determined using a 5-gram sample and heating at 130° ± 3° C. for 2 hours in a forced draft oven.
3. In line with the above recommendations it is necessary to recommend that paragraph 1 of section C of the method for the determination of nitrogen, ammonia, and protein (Ab 4-38) be changed to read "Use a portion of the sample from A.O.C.S. official method Ab 3-38, section C, paragraph 2."

Respectfully submitted,

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The report and recommendations of the Subcommittee for Peanuts and Peanut Meal have been given a unanimous vote of approval by the Committee.

The Subcommittee for Tung Fruit and Meal is making progress, but is not ready to make any recommendations at this time. Its efforts are timely as the American Tung Oil Association is now taking an interest in official methods. The Subcommittee is in position to work closely with this Association.

The lack of a unanimous vote indicates the failure of one or more of the members of the Committee to vote on the recommendations.

The methods for sampling and screen test and for the determination of water absorption and phosphatides for soyflour are under consideration. Study is also being given to the development of a fiber test for soyflour which will be specific for this commodity.

Respectfully submitted,

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