Report of Seed and Meal Analysis Committee, 1947-48

THE Seed and Meal Analysis Committee has eight subcommittees actively engaged in study of and/or collaborative testing of methods of analysis. Their interest, activity, and recommendations are given in this report.

REPORT OF THE SUBCOMMITTEE ON SOY FLOUR SAMPLING

The Subcommittee on Soy Flour Sampling has agreed tentatively on a method of sampling soy flour for production purposes. However, due to certain complications the method cannot be submitted at this time. As far as bag sampling of soy flour is concerned, the subcommittee has concurred that the A.O.A.C. method for sampling wheat flour could be adapted to soy flour with certain modifications. In this connection work is now progressing on the construction and testing of a proposed new trier. Automatic sampling devices are also under consideration by the committee.

There was hope that definite recommendations could be made at this time, but it is evident that the work is more ponderous than was initially expected; therefore, it would seem desirable that the work of the subcommittee be carried over into the coming year.

L. R. BROWN — T. C. SMITH
LEONARD GERHART — M. W. DIPPOLD, chairman

REPORT OF THE SUBCOMMITTEE ON SOY FLOUR SIEVING METHOD

The subcommittee has done considerable work on its assignment to study and develop a suitable sieving method for soy flours. A number of procedures have been studied, and although we have no specific recommendations to make at this time, we do have sufficient data to indicate that the commonly used brushing or shaking methods are not satisfactory. In view of this situation methods involving the use of solvents and various washing procedures are being studied. It is recommended that the assignment of the subcommittee be extended for another year.

R. E. ANDERSON — W. F. GEDDES
M. W. DIPPOLD — J. K. GUNTHER
F. R. EARLE — V. C. MEHLENBACHER
E. B. FREYER — L. R. BROWN, chairman

REPORT OF THE SUBCOMMITTEE ON DETERMINATION OF THE WATER ABSORPTION OF SOY FLOUR

The subcommittee has been working on its assignment of investigating determination of water absorption of soy flour. In this investigation we are attempting to determine the significant factors involved in the determination of water absorption of soy flour from which, when completed, we will be able to decide whether or not a suitable method can be presented to the Society or whether any method is practical. While considerable information has been developed, the committee does not feel that the work has been completed. We therefore suggest a continuation of the problem. It is quite possible that within another year we can arrive at a decision and make a definite recommendation to the Society.

J. K. GUNTHER — L. R. BROWN
M. L. LAING — V. C. MEHLENBACHER, chairman

SUBCOMMITTEE ON DEVELOPMENT OF A METHOD FOR LECITHIN IN SOY FLOUR

Three members of this subcommittee have made collaborative analyses for phosphorus on the alcohol soluble portions of extracted, low-fat, and high-fat soy flours using a modification of the well-known volumetric method for determining percentages of phosphorus. This modification of the method provides for the following: 1. Continuous extraction for 16 hours of filter paper-wrapped 5-gram samplings of soy flour with 95% ethyl alcohol, using "Butt" extraction equipment; 2. addition of magnesium nitrate solution and a cotton wick to the alcohol solution of lipids and phosphorus bearing material; 3. burning off the excess alcohol and lipids and ashing of the charred remainder in a muffle furnace; 4. alkalimetry determination of phosphorus as the molybdate; and 5. calculation of the percentage of phosphorus to the percentage of lecithin. The results on these three samples are shown in the following table:

	Laboratory 1		Lab. 2	Lab. 3
	% Lecithin		% Lecithin	% Lecithin
	October[1]	April[2]		
Extracted flour	2.31 2.31	2.20 2.18	2.25 2.25	2.28 2.30
Low-fat flour	2.42 2.42	2.31 2.31	2.47 2.37 2.47 2.47	2.36 2.39
High-fat flour	2.18 2.18	2.11 2.10	2.19 2.19	2.09 2.13
Averages	2.30	2.20	2.29	2.26

[1]Analyses made in October, 1947.
[2]Analyses made in April, 1948.

Within a given laboratory the method as modified seems to work quite well; however, the report shows more variation within a given laboratory and between laboratories than is desirable.

The subcommittee is of the opinion that in order to develop a method and evaluate it properly before recommending it for adoption, at least one collaborator must be found who is using phosphatides in a process that will permit the correlation of amount of alcohol soluble phosphorus material and of added phosphatides with the effects produced. If it is decided to continue collaborative work on methods for determining lecithin in soy flour, it is suggested that a rapid colorimetric procedure might be found that would be more satisfactory than the time-consuming volumetric method which has had preliminary testing.

F. R. EARLE — W. D. POHLE
T. J. POTTS — F. I. COLLINS, chairman

REPORT OF THE SUBCOMMITTEE ON METHOD FOR DETERMINING CRUDE FIBER IN SOY FLOUR

Examination of methods for the determination of crude fiber in soy flour shows two possible procedures which might apply. A considerable amount of investigation remains to determine the acceptability of either of these procedures. It is therefore recommended that the present subcommittee be assigned to continue the study of this problem.

J. K. GUNTHER — L. R. BROWN
W. F. GEDDES — T. J. POTTS
V. C. MEHLENBACHER — R. E. ANDERSON, chairman

REPORT OF SUBCOMMITTEE FOR PEANUTS AND PEANUT MEAL

The report of this subcommittee in 1947 pointed out 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 recommendations were approved by the Society for the deletion of the methods for the analysis of whole peanuts for moisture, oil, and nitrogen in the whole nuts. At the same time revised methods were offered and adopted as tentative for the determination of moisture and oil in peanut kernels, and a slight editorial change was made in the method for the determination of nitrogen in regard to the reference for the preparation of the sample for analysis.

These tentative methods have been used by the Commodity Credit Corporation for the third marketing season for evaluating peanuts. They were extensively used during the past season as an adjustment basis on export peanuts. The seven cooperative check samples of the Smalley Foundation were analyzed by these tentative methods for moisture and oil. The reports of the results of 12 chemists in as many laboratories are given in Tables I and II. The data indicate the satisfaction of the methods and rates the chemists. Fourteen of the 96 values were outside the tolerance of ± 0.3% for moisture. Ten values were high and four were low. Twenty-seven of the 96 values were outside the tolerance of ± 0.3% for oil. Fourteen were high and 13 were low, indicating the possibility of failure to remove all of the solvent from the extracted oil or to properly regrind the sample to facilitate extraction.

For several years the committee has been working on a more satisfactory method of grinding in the preparation of peanut kernels for analysis. The Henry slicer was thus developed. It apparently eliminates much of the objection to the food chopper in that the prepared sample is not gummy and free oil is not present. This slicer has been used as official for the U.S.D.A. and all export analyses for the past six months. Comparative collaborative results of analyses of carefully selected duplicate samples by three chemists are given in Table III. These results indicate the satisfaction had in the use of the slicer in kernel sample preparation. An average deviation of 0.23% is observed from the average of the values reported for Smalley Foundation check sample No. 6 (Table II). A lot of the same peanuts were sliced in the Henry slicer and thoroughly mixed samples were sent to the same 12 chemists. For these duplicate samples, sample 6A (Table II), the mean deviation from the average was 0.15%. Similarly, the mean deviation from the average was 0.055% ammonia for the food chopper prepared sample, and 0.037% ammonia for the Henry slicer prepared sample. In this case 16 chemists participated in the check sample analysis.

The use of crimp sealed tin cans for storage and shipping of samples in the Smalley Foundation has continued to show the value of this procedure in retaining the moisture content of the samples. The agreement observed among chemists in the analysis of check samples for moisture, Table I, is in a large measure due to packing and distributing the samples in crimp sealed cans. No such agreement would be possible if the procedure did not assure the chemists received samples as near as possible at the same moisture content.

In view of the above, which is supported by experience greater than cited, it is recommended:

1. That "Sample containers as follows: *for whole nuts* ½ gallon, open-mouthed cans equipped with covers suitable for sealing by machine crimping; *for shelled stock* (kernels) No. 2 open-mouthed cans equipped with covers for sealing by machine crimping" be substituted for paragraph 5, section A, of method Ab-1-38 for sampling peanuts. All containers shall be filled to capacity before sealing for sending to laboratories. Containers shall be sealed immediately on filling.
2. That "Mix the cleaned sample thoroughly and fill to capacity proper containers as specified in A (5) above and seal immediately by machine crimping" be substituted for paragraph 4, section C, of method Ab-1-38 for the sampling of peanuts.
3. That the tentative method (Ab-2-47) for the determination of moisture in peanut kernels be made official.
4. That the tentative method (Ab-3-47) for the determination of oil in peanut kernels be amended to specify the use of the Henry Nut Slicer instead of the Universal food chopper No. 1 with the peanut butter blade to be made official.
   If this recommendation be approved changes in the text of the printed procedure will be:
   a) Paragraph 4 of section A will be made to read "Henry Nut Slicer (Davidson-Kennedy Co., Atlanta, Ga.)."
   b) Paragraph 2 of section C will be made to read "Cool the sample to room temperature and then pass through the nut slicer. Utmost care is required that the slicing blade is set so as to prevent the expressing of any oil. Completely mix the sliced sample. The Law and Company Viscosity Mixer is recommended for mixing the sliced sample."
   c) Changing the word "ground" to "sliced" elsewhere in the text of the written procedure.
5. That the official method (Ab-5-38) for the determination of free fatty acids be amended to specify the use of the

TABLE I

Per Cent Moisture Found in Peanut Kernel Check Samples

Chemist	Check Sample Number							
	1	2	3	4	5	6	6A	7
1	6.0	6.6	6.0	5.6	5.6	6.8	6.2	7.5
2	5.6	6.5	5.9	5.6	5.8	6.8	5.7*	7.4
3	5.9	6.8	5.5*	5.8	5.8	6.8	8.6*	7.2
4	6.3*	7.3*	6.0	6.2*	5.9	7.3*	6.1	7.7
5	5.9	7.0	5.8	5.7	5.8	7.1	6.4	7.1
6	6.1	6.3*	6.0	5.7	5.7	7.1	6.1	7.5
7	5.9	7.1	6.0	5.8	5.7	6.6	6.1	7.2
8	6.0	6.8	6.0	5.7	6.0	6.8	6.2	7.4
9	6.1	7.1	7.6*	6.2*	5.9	7.0	6.2	7.5
10	5.6	7.4*	6.1	5.3	5.6	6.8	5.8	7.5
11	5.7	6.7	6.0	5.1*	5.6	6.9	6.5*	7.9*
12	5.8	6.6	5.9	5.3	5.7	6.9	6.2	7.2
Average	5.9	6.8	6.0	5.6	5.7	6.9	6.1	7.4

* Outside tolerance of Smalley Foundation Committee of ± 0.3%.

NOTE: Value of chemist No. 3 omitted from average for Sample 6A. Sample 6A represents an additional sampling of the mixed lot of peanut kernels sampled for check sample No. 6.

TABLE II

Per Cent Oil Found in Peanut Kernel Check Samples

Chemist	Check Sample Number							
	1	2	3	4	5	6	6A	7
1	46.3	45.2	44.8	46.5	45.8	47.5	48.1	46.7
2	46.2	45.0	44.4*	46.3	46.0	47.2*	48.0	46.4*
3	46.0	44.8	44.9	46.1	45.5	48.2*	48.1	47.3
4	46.3	45.5*	45.4	46.3	45.7	47.9	48.2	47.4*
5	46.0	45.1	44.8	46.0	45.1*	47.4*	48.0	46.7
6	47.4*	45.5*	44.7	45.9	45.9	47.8	47.5*	47.0
7	45.8*	44.9	44.1*	46.1	45.5	48.1	48.0	47.4*
8	45.9	44.5*	44.5	45.7	44.8*	47.9	47.8	46.3*
9	48.0*	44.6	44.5	44.5*	47.2*	47.6	48.1	46.9
10	46.2	45.0	44.6	46.7*	45.6	47.9	48.3	46.7
11	46.6	45.1	45.5*	45.9	46.4*	48.0	48.1	47.5*
12	46.6	44.6	44.8	46.0	45.1*	47.7	47.9	47.6*
Accepted Average	46.4	45.0	44.8	46.0	45.7	47.8	48.0	47.0

* Outside tolerance of Smalley Foundation Committee of ± 0.3%.

NOTE: Sample 6A represents an additional sampling of the mixed lot of peanut kernels sampled for check sample No. 6. In this case the samples were prepared by use of the Henry shaver before distribution.

TABLE III

Subcommittee Cooperative Work on Peanuts

	Oil and Ammonia Results Calculated to 7% Moisture Basis											
	Food Chopper-Prepare Preheated				Slicing Machine-Prepare Preheated				Slicing Machine-Prepare Unheated			
	2nd H_2O	Oil	NH_3	FFA	2nd H_2O	Oil	NH_3	FFA	2nd H_2O	Oil	NH_3	FFA
Cox	4.28	47.0	5.60	1.1	4.07	47.2	5.56	1.1	4.53	47.3	5.49	1.1
	4.36	47.4	5.57		3.91	47.5	5.54		4.58	47.2	5.57	
	4.09	47.5	5.59		5.12	47.6	5.54		4.63	47.4	5.55	
	4.30	47.4	5.51		4.26	46.9	5.54		4.60	47.6	5.50	
	4.05	47.5	5.47		4.03	46.8	5.60		4.51	47.1	5.54	
	4.54	47.1	5.57		3.82	47.4	5.58		4.65	47.2	5.64	
Average	4.3	47.3	5.55	1.1	4.2	47.2	5.56	1.1	4.6	47.3	5.55	1.1
Oil Spread		0.5				0.8				0.5		
Ainslie	2.8	47.9	5.63	1.1	2.4	47.7	5.56	1.0	5.3	48.0	5.60	0.8
	2.6	48.1	5.56		2.4	47.9	5.55		5.5	47.7	5.64	
	2.3	47.7	5.61		2.6	47.7	5.53		5.3	48.0	5.52	
	2.6	47.8	5.57		2.5	48.0	5.55		5.3	47.7	5.59	
	2.6	47.3	5.58		2.3	48.3	5.56		5.4	48.0	5.54	
	2.5	46.7	5.62		2.5	47.9	5.58		5.3	48.1	5.56	
Average	2.6	47.6	5.59	1.1	2.5	47.9	5.56	1.0	5.4	47.9	5.58	0.8
Oil Spread		1.4				0.6				0.4		
Law	2.3	47.9	5.56	0.8	2.5	47.9	5.60	0.8	6.2	47.9	5.65	1.1
	2.1	48.0	5.59		2.7	48.1	5.56		6.3	47.8	5.65	
	2.4	47.8	5.56		3.1	48.0	5.65		6.2	48.1	5.65	
	2.3	47.6	5.60		2.6	48.0	5.60		6.3	47.9	5.59	
	2.5	47.8	5.53		2.6	48.1	5.56		6.3	47.9	5.64	
	2.6	47.5	5.60		2.7	48.1	5.58		6.2	48.4	5.59	
Average	2.3	47.9	5.57	0.8	2.6	48.0	5.59	0.8	6.3	48.0	5.63	1.1
Oil Spread		0.5				0.2				0.6		
Average All Samples	3.1	47.6	5.57	1.0	3.1	47.7	5.57	1.0	5.4	47.7	5.59	1.0
Average Oil Spread		0.80				0.53				0.50		

Henry Nut Slicer instead of the Universal food chopper for the preparation of the peanut kernel sample and that the method be continued as official. If this recommendation is approved, paragraph 1 of section A of the written method will be changed to read "Henry Nut Slicer (Davidson-Kennedy Co., Atlanta, Ga.)" and paragraph 2 of section C will be changed to read "Pass ca 150 grams of kernels through the nut slicer. Mix the sliced sample thoroughly."

E. C. AINSLIE
C. H. COX
T. J. POTTS
G. CONNER HENRY
T. C. LAW,
chairman

REPORT OF SUBCOMMITTEE ON TUNG FRUIT AND MEAL ANALYSIS

Definition

In presenting the results of the work of the subcommittee it is felt that the samples encountered in research and in marketing and processing must be defined. Tung fruit (known commercially as tung nuts) is the whole ripened fruit organ of the tung tree and consists usually of four or five seeds and the surrounding hull. The hull is the outer covering or husk in which the seeds are borne. The tung shell is the outer woody covering of the seed enclosing the oily tung kernel. The tung kernel is the inner oily portion of the tung seed. The fruit, hull, seed, shell, and kernel are illustrated in Fig. 1.

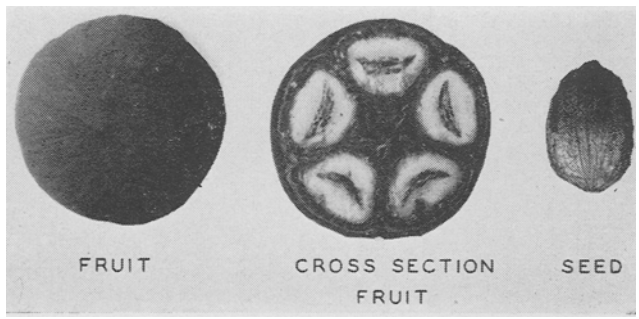

FIG. 1

Sampling

In studies on the sampling of tung fruit, duplicate samples were obtained from five loads of fruit, taking in each case at least 15 two-quart samples from the center of the unloading chute with a sampler at regular intervals to collect samples of sufficient size to fill a 50-pound lard can. These duplicate samples

were analyzed for moisture and oil content by one of the collaborators and the results obtained are given in Table I.

TABLE I

Analysis of Duplicate Samples Obtained by Recommended Sampling Method

Sample	Oil %	Moisture %
1A	20.3	16.4
1B	20.4	16.1
2A	18.2	21.0
2B	17.8	21.2
3A	18.2	23.8
3B	18.2	21.0
4A	19.4	24.3
4B	19.5	23.8
5A	18.1	23.9
5B	17.6	24.2

Analytical

During the last three years the members of the Subcommittee on Tung have made an intensive study of methods of analysis of tung fruit. During 1946 the subcommittee members made collaborative analyses on samples of tung fruit prepared and sent out by the subcommittee chairman. The procedures used were based upon methods of analysis developed in the Bureau of Agricultural and Industrial Chemistry (1) with the exceptions that moisture determinations were made on the whole fruit or carpel samples instead of the components, and the moisture content of samples of ground kernels was determined by drying five-gram samples of this material for 1 hour in a forced draft oven at 101°C. Preliminary tests (see Table II) had shown that a 24-hour drying period at 101°C. was sufficient to completely remove the moisture from the whole fruit samples and that concordant moisture results could be obtained by drying the ground kernels for 1 hour at 101°C. in a forced draft oven.

TABLE II

Moisture Data on Tung Fruit Dried for 24 and 28 Hours at 101°C.

Sample	Drying Time	
	24 Hours % Moisture	48 Hours % Moisture
1	13.9	14.0
2	14.3	14.4
3	14.6	14.7
4	11.7	11.9
5	10.9	11.0
6	11.5	11.7

Analysis of Ground Kernels for Moisture in Forced Draft Oven for One Hour at 101°C.

Sample	Collaborator		
	1	2	3
1	5.66	5.6	5.42
2	4.2	3.4	3.9
3	9.1	....	9.4
4	4.0	4.2	4.3
5	5.3	6.2	5.8
6	5.8	6.4	6.0
7	4.1	4.5	2.6

In the procedure employed on the first series of samples the moisture was determined by drying 5- or 8-fruit subsamples for 24 hours at 101°C. and 25-fruit subsamples were separated into components and the separated kernels used in the oil determination. In the preparation for the oil determination the separated kernels were ground twice in a No. 71 Universal Feed Grinder or once in a Bauer No. 148 mill, with No. 6912 plates at 3600 r.p.m. adjusted to produce a fine meal. The moisture in the ground kernels was determined by drying 5-gram samples in a vacuum oven at 50 mm. of Hg. pressure and 101°C. for 3 hours, or in a forced draft oven at 101°C. for one hour with redrying for half hour periods until the loss of weight was not more than 5 mg. In the oil determination a five-gram sample of the undried ground kernels was extracted for 4 hours in a Butt-type extraction apparatus with petroleum ether of American Oil Chemists' Society specifications and the extracted sample was reground with mortar and pestle for 5 minutes with one gram of fine sand and re-extracted in a similar manner for 2 hours. In a second series, consisting of the last three samples, one carpel was taken from each of the 35 fruits in the sample and used for the oil determination, while another carpel was taken from each fruit for the moisture determination. The results obtained by the collaborators using the two procedures are given in Table III. Considerable variations in the oil and moisture contents of the first five samples occurred, with standard deviations of 0.32%-0.90% oil and 0.42%-0.97% moisture, which could be attributed to sampling errors on the basis of a recent study on the sampling of tung fruit (2). Considerably higher standard deviations in the oil content of the tung fruit samples occurred when carpels were used instead of the kernels of the fruits.

TABLE III

Analysis of Collaborative Samples by Component Procedure

Sample	Per Cent Oil in Tung Fruit								S. D.
	Collaborators								
	1	2	3	4	5	6	7	Average	
1	19.5	21.0	19.8	19.8	19.1	20.4	19.5	19.8	0.63
2	19.6	19.9	19.2	19.7	20.5	19.1	18.9	19.6	0.55
3	19.9	19.9	20.2	19.8	20.4	19.5	19.0	19.8	0.48
4	22.4	22.7	22.6	23.0	22.1	22.7	23.0	22.6	0.32
5	22.7	23.2	23.6	23.0	21.0	22.7	23.7	22.8	0.90
6	19.4	21.8	20.6	19.2	18.9	20.9	19.4	20.0	1.10
7	21.8	21.8	20.5	19.7	18.3	22.0	20.8	20.7	1.35
8	20.7	20.5	18.7	20.2	20.5	21.3	20.1	20.3	0.80
	Per Cent Moisture in Tung Fruit								
1	15.1	16.4	16.3	15.6	16.0	15.0	14.7	15.6	0.67
2	15.0	14.8	16.8	15.3	14.3	15.3	14.5	15.1	0.82
3	11.9	11.6	12.1	11.9	12.9	11.8	12.1	12.0	0.42
4	13.0	12.6	10.4	13.4	12.5	12.4	12.8	12.4	0.97
5	11.8	12.5	9.6	11.7	11.2	11.3	11.1	11.3	0.89
6	11.3		9.4	10.6	10.5	10.5	10.2	10.4	0.69
7	11.5	10.7	10.0	10.7	10.8	10.5	10.5	10.7	0.42
8	10.1	10.5	9.8	10.8	10.0	10.1	10.6	10.3	0.35

It appeared that the accuracy could be improved only by the use of a much larger sample than that usually used in analyses by the component procedure. Increasing the size of the sample in the component procedure introduced the problem of the proper hulling and shelling of the large sample, as skilled workers required for the task objected to the tediousness involved. Therefore consideration was given to the possibility of the devlopment of a new procedure wherein the whole sample of tung fruit would be ground and moisture and oil determinations made on portions of the ground fruit.

The procedure developed by McKinney, Halbrook, and Agee (3) employs a sufficiently large sample to eliminate to a considerable extent the sampling errors which occur with the relatively small samples usually used in the component procedure. In this procedure a sample of 200- to 250-fruit is ground in a Wiley mill, using a ¼-inch screen and, after thorough mixing, drawing two portions of about 1½ quarts each. One portion is used in the moisture determination and the other portion, after regrinding in a Raymond or Bauer mill, is used for the oil determination. In the new procedure three methods may be used for deter-

mining the moisture content of the tung fruit. The first method consists of drying a 5-gram portion of the Wiley-ground sample for 4 hours at 101°C. in an oven at not more than 50 mm. of Hg. pressure. After cooling and weighing, the sample is redried under similar conditions for 1-hour periods until a loss of weight of not more than 2 mg. occurs. In the second method a 5-gram sample of the Wiley-ground material is dried for 1 hour in a forced draft oven at 101°C., cooled and weighed, then redried under the same conditions for half-hour periods until the loss of weight is not more than 5 mg. Two redrying periods are usually required. The third method, that of Bidwell-Sterling (4) using a 20- or 100-gram sample and run for 1¼-1½ hours, probably gives the most accurate estimate of the moisture content of tung fruit as some oxidation may occur in the two oven methods.

As some drying occurs in the preparation of the Wiley-ground material for the oil determination, it is necessary to make a moisture determination on the thoroughly mixed Raymond- or Bauer-ground material using the same method employed on the Wiley-ground material. Redrying in the oven methods is not usually required. A 5-gram sample of the Raymond- or Bauer-ground material is extracted for 4 hours in a Butt-type extraction apparatus using petroleum ether of American Oil Chemists' Society specifications and the oil content of the tung fruit is calculated to the original moisture basis.

TABLE IV

Effect of Moisture Method on Estimation of Oil Content of Tung Fruit

Sample	Moisture Method Bidwell-Sterling		Oil	Moisture Method Forced-Draft Oven		Oil
	Wiley-Ground	Bauer-Ground		Wiley-Ground	Bauer-Ground	
	%	%	%	%	%	%
1	15.3	12.4	19.90	14.16	11.24	19.93
2	14.3	10.8	20.65	13.1	9.2	20.72
3	17.5	12.1	22.00	16.68	11.34	21.95
4	12.05	10.65	21.67	10.66	8.89	21.65
5	17.4	15.2	19.41	16.33	13.9	19.45
6	15.1	13.3	20.45	13.6	12.1	20.38
Average	15.3	12.4	20.68	14.1	11.1	20.68

To determine the effect of the moisture method employed on the estimation of the oil content of samples of tung fruit using the Wiley-Bauer grinding technique, six samples of tung fruit were analyzed for oil and moisture content, using both the Bidwell-Sterling method and the forced draft oven method. Data obtained in these analyses are given in Table IV. It is clear that variations in the per cent moisture obtained with the forced draft oven have no appreciable effect upon the estimation of the oil content. This is important since current prices of tung fruit (nuts) are based upon oil content. Therefore use of the forced draft oven instead of the Bidwell-Sterling method will not affect the purchase of fruit.

Comparison of the New Procedures with the Component Procedure

The tung industry has used the component procedure for the analysis of tung fruit for a number of years and the price paid for the fruit has been based upon the results obtained by this procedure. Therefore for the new procedure to be acceptable to the industry it had to yield results comparable to those obtained by the component procedure.

A difficulty in the development of the whole fruit procedure was that an appreciable amount of non-oil constituents, soluble in petroleum ether, occurs in the shell and hull portions of the tung fruit. In the component procedure the oil content of the fruit is calculated from the per cent kernels and the per cent oil in the kernels, the oil being located entirely in the kernels. In a preliminary study of the new procedure in the laboratory of one collaborator six samples were drawn from commercial lots of tung fruit at a mill, and each sample was thoroughly mixed and quartered into two portions of about 100-fruit each. One portion of each sample was analyzed by the component procedure while the other portion was analyzed by the new procedure using the Wiley-Raymond grinding technique. The results obtained are listed in Table V. The average results obtained for the oil and moisture content of the samples of tung fruit by the component procedure and by the new procedure using the Wiley-Raymond grinding technique are in good agreement while the variations between the results for individual samples are about that to be expected from the previously mentioned sampling study (2) when 100-fruit samples are used.

TABLE V

Comparison of Oil and Moisture Content of Tung Fruit by Component Procedure and by New W-R Procedure

Sample	Component Procedure		New W-R Procedure	
	Moisture	Oil	Moisture*	Oil
	%	%	%	%
1	16.37	20.27	16.57	20.10
2	16.62	20.29	16.71	19.93
3	13.85	20.48	13.56	21.21
4	15.67	19.25	16.16	19.62
5	11.46	19.98	11.38	19.69
6	11.67	20.04	11.54	19.43
Average	14.27	20.05	14.32	20.00

* Using vacuum oven method.

In connection with the collaborative analyses on samples of tung fruit during 1947 it appeared desirable to compare the results obtainable with the new procedure using the Wiley-Bauer or the Wiley-Raymond grinding technique with those obtained by the component procedure. Five lots of tung fruit were thoroughly mixed and each lot was divided into three large samples of 200 to 250 fruit each. One of the large subsamples of each lot was then subdivided into six small subsamples which were analyzed by the members of the Subcommittee on Tung using the component procedure. The results obtained by the collaborators are given in Table VI. The other two large subsamples of each lot of tung fruit were analyzed for moisture and oil content by two collabo-

TABLE VI

Analysis of Collaborative Samples by Component Procedure

Sample	Per Cent Oil in Tung Fruit							S. D.
	Collaborators							
	1	2	3	4	5	6	Average	
1	20.4	21.3	20.8	19.9	20.1	19.3	20.3	0.70
2	19.4	19.3	19.5	(17.2)	18.5	19.3	19.2	0.40
3	18.5	19.0	21.0	18.9	19.1	19.8	19.4	0.90
4	20.8	20.5	20.6	21.0	20.9	20.4	20.7	0.24
5	19.3	19.3	20.2	18.4	19.4	20.6	19.5	0.84
	Per Cent Moisture in Tung Fruit							
1	14.1	13.0	14.5	14.0	13.5	14.6	13.9	0.48
2	10.5	10.7	12.2	10.2	11.7	11.3	11.1	0.80
3	11.2	11.8	12.5	11.2	11.7	11.9	11.7	0.49
4	14.2	14.7	14.8	14.2	14.9	14.1	14.5	0.37
5	14.1	15.6	15.6	14.2	14.8	15.5	15.0	0.70

rators using the Wiley-Bauer and the Wiley-Raymond grinding technique. Because of the sampling errors in the component procedure with such small samples, the average of the results of the collaborators were calculated and used for comparison. In Table VII are given the average results of the subcommittee members on the samples analyzed by the component procedure and by the new procedure using both Wiley-Bauer and Wiley-Raymond ground samples. The average of the results of the collaborators for oil content (19.82%) by the component procedure was found to be in good agreement with the average results obtained on the Wiley-Raymond ground samples (19.81%), but the average results obtained on the Wiley-Bauer ground material (20.19%) are appreciably higher than those obtained by the other procedures. These results indicate that a correction of 0.37% must be subtracted from the per cent oil obtained on samples ground in the Wiley-Bauer mills to obtain results comparable to those obtained by the component procedure.

TABLE VII

Per Cent Oil and Moisture Content by Component and New Procedures

Sample	Per Cent Oil in Tung Fruit		
	Collaborators' Component Procedure	Wiley & Raymond Ground Sample	Wiley & Bauer Ground Sample
	(Average Results)		
1	20.30	20.74	20.80
2	19.20	19.57	20.30
3	19.38	19.47	19.80
4	20.70	20.42	20.25
5	19.50	18.85	19.80
Average	19.82	19.81	20.19
	Per Cent Moisture in Tung Fruit		
Sample	Collaborators' Component Procedure (18-24 hrs. @ 101°C.)	Wiley & Raymond Ground Sample (4 hrs. Vac. Oven)	Wiley & Bauer Ground Sample (B-S *Method*)
	(Average Results)		
1	13.9	13.6	14.5
2	11.1	11.4	11.5
3	11.7	11.8	12.4
4	14.5	13.6	14.9
5	15.0	14.9	15.3
Average	13.2	13.1	13.7

In this collaborative work it was found that the Bidwell-Sterling method gave the highest results (average, 13.7%) for moisture content of tung fruit; drying the whole fruit for 24 hours yielded moisture results (average, 13.2%) which were slightly higher than those obtained by drying the Wiley-ground fruit in a vacuum oven (average, 13.1%). As has been shown by Table IV, the variations in the analysis of tung fruit by the new procedure using the three moisture methods should not have an appreciable effect upon the estimation of the oil content of the tung fruit, provided the same moisture method is employed with the Wiley-ground and the Wiley-Bauer or Wiley-Raymond ground samples.

During 1948 the chairman of the Subcommittee on Tung sent out six collaborative samples of tung fruit to seven laboratories for check analysis, two laboratories using the component procedure and five laboratories using the whole fruit procedure with the Wiley-Bauer grinding technique. In the component procedure the moisture was determined by drying 8-fruit subsamples for 24 hours at 101°C., while in the whole fruit procedure the moistures were determined by drying samples of the ground materials in a forced draft oven at 101°C. The samples sent to each laboratory contained between 200 and 250 fruits. The average results of the laboratories are given in Table VIII. With a few exceptions the agreement of the laboratories for the past year were good as seen by these figures.

TABLE VIII

Analysis of Collaborative Samples by Component and Whole Fruit Procedures

Collaborator	Per Cent Oil in Tung Fruit					
	Sample Number					
	1	2	3	4	5	6
1[1]	21.64	19.27	20.79	18.64	20.50	21.33
2[1]	21.93	19.00	20.50	19.32	20.52	20.98
3[2]	21.76	19.08	20.44	18.86	20.96	20.97
4[2]	21.65	19.41	20.45	18.82	20.39	20.70
5[2]	22.10	19.26	21.20	18.70	20.00	20.80
6[2]	(21.60)	18.91	21.10	19.03	20.26	21.68
7[2]	.....	(18.1)	20.70	(17.70)	20.10	20.80
	Per Cent Moisture in Tung Fruit[3]					
1[1]	11.71	18.00	14.80	15.13	17.46	14.61
2[1]	11.33	18.62	14.93	14.26	19.01	15.35
3[2]	10.97	16.62	14.30	14.35	16.29	13.93
4[2]	10.64	16.33	13.64	13.85	16.15	13.62
5[2]	10.70	16.30	13.70	14.20	16.50	14.00
6[2]	11.18	16.64	13.64	13.27	15.94	13.74
7[2]	.....	16.50	14.30	14.40	16.20	13.70

[1] Used component procedure.
[2] Used whole fruit procedure.
[3] Samples dried 24 hours at 101°C. in component procedure; ground material dried at 101°C. in forced draft oven in whole fruit procedure.

The industry laboratories have used and are now equipped for use of the component part procedure of analysis. The inability of the commercial testing laboratories to use this method, because of the expense and difficulty of shelling the kernel, led to the development of the whole fruit method. These two methods have been used and systematically checked against each other during the past season, during which a price-support program has been maintained with gratifying results. Until all laboratories of the industry and official or commercial testing chemists can be equipped for a single method, such as the whole fruit method, it is anticipated that both methods may necessarily be used.

The specifications of the methods for sampling, for analysis by components and for analysis of the whole fruit are:

Sampling

A. PROCEDURE:

Carload, truck, or wagon lots during unloading.

Take at least 15 two-quart samples from the center of the unloading chute with an approved sampler at regular intervals so that at least a 50-pound lard can shall be collected. Each portion of the sample as drawn shall be immediately placed in metal container and the tight fitting cover promptly replaced. The gross sample shall be weighed and weight recorded and sample shall be stored safely until analyzed.

B. CLEANING AND SEPARATION OF LABORATORY SAMPLE:

It is recommended that if possible the dirt be removed from the load of tung fruit and be added to the tare. The sample shall be examined and if found not to have been thoroughly cleaned shall be recleaned by the use of 6-mesh screen and by hand picking of all remaining particles of foreign matter. Weigh foreign matter and calculate per cent as follows:

$$\text{Foreign matter } \% = \frac{\text{Weight of foreign matter} \times 100}{\text{Weight of sample}}$$

Analysis by Components

A. PREPARATION OF SAMPLE:

1. Foreign matter: Reweigh laboratory sample, noting any loss of moisture and examine sample for foreign matter

which is to be removed from that portion to be used for analysis. Pass the sample over a 6-mesh screen to remove as much foreign matter as possible and pick out the remainder by hand after spreading out on a clean, dry surface. Calculate foreign matter as above.

B. MOISTURE:

1. Preparation of sample: Grind a sample of 25 tung fruit in a Wiley mill using a ¼-inch screen. The Wiley mill shall be equipped with an auxiliary hopper over the regular hopper to prevent material from being thrown out and also a tight fitting chute from the bottom of the mill through the cover of a large can into which the ground material is delivered without possibility of spilling or drying out. The whole sample of ground tung fruit shall be thoroughly mixed, first breaking up any lumps by hand, by rolling on a large piece of paper. The sample is then subdivided by quartering with a large spatula or with a riffle to a subsample of about 1 quart.

2. Moisture determination:

a) Weigh duplicate 5-gram samples of Wiley-ground tung fruit into moisture dishes and place dishes with the samples in an approved forced draft oven for one hour at 101°C. Remove dishes from the oven, cover promptly, cool and weigh. Replace the dishes in the oven for one-half hour, remove from the oven, cover, cool and weigh as before. Repeat the process until loss in weight between successive weighings is not more than 5 mg., or until a gain of weight is noted. The moisture to be reported is calculated from the greatest loss found.

b) Calculations: Moisture and volatile matter in tung fruit,

$$\% = \frac{\text{Loss in weight} \times 100}{\text{Weight of sample}}$$

C. OIL:

1. Determination of per cent kernels and preparation of sample:

a) Weigh a sample of at least 100 tung fruit. Remove the hulls and shells from the kernels and weigh the tung kernels, shells and hulls.

$$\text{Kernels } \% = \frac{\text{Weight of kernels}}{\text{Weight of fruit sample}}$$

b) Grind the kernels twice in a Universal Feed Grinder No. 71, using the 16-tooth blade or grind in a Bauer Mill No. 148 with No. 6912 plates, which has been adjusted to produce a fine meal.
If the sample is very large, the ground kernels may be quartered to a small sample which is placed in a sample bottle with tight stopper.

2. Determination of moisture in prepared kernels:

a) Forced draft oven procedure:
Weigh 5 grams of ground kernels into a tared American Oil Chemists' Society dish. Place uncovered dish in a forced draft oven for 1 hour at 101°C. Remove dish from oven, cover at once, cool in a desiccator and weigh. Replace dish with cover removed in oven for one-half hour, remove the dish from oven, cover it at once, cool and weigh as before. Repeat the procedure until the loss in weight is not more than 5 mg. between successive weighings, or until a gain in weight is noted. The moisture to be reported is calculated from the greatest loss in weight found.

3. Determination of oil in prepared kernels:

a) Apparatus:
a. Butt-type extraction apparatus, assembled as for cottonseed and similar analyses.
b. Filter paper, S. & S. No. 597, Reeve Angel No. 211 or equivalent, 125 or 150 mm.
c. Absorbent cotton.
d. Air-tight sample containers for holding ground samples.

b) Reagent:
Petroleum ether, American Oil Chemists' Society specifications.

c) Procedure:
1. Weigh accurately 5 grams of the ground sample into a filter paper and enclose in a second paper or papers, folded in such a manner as to prevent escape of meal. The second paper is left open at the top like a thimble. A piece of absorbent cotton may be placed in the top of the thimble to distribute the solvent as it drops onto the sample.
2. Place wrapped sample in the Butt extraction tube and assemble the apparatus in the usual manner. Place 25 to 30 ml. of petroleum ether in the Soxhlet flask before attaching to the tube.
3. Heat on a water bath at such a rate that the solvent will drop from the condenser into the thimble at a rate of at least 150 drops per minute. Keep the volume of solvent fairly constant by adding enough to make up for any that may be lost due to evaporation. Extract for 4 hours.
4. Cool and disconnect the extraction flask and tube and remove wrapped sample from tube. Empty the sample into a mortar, add 1 gram of fine sand and grind with pestle for 5 minutes. Rewrap the sample and continue extraction for an additional 2 hours. Occasionally check the efficiency of extraction by regrinding sample 5 minutes and re-extracting.
5. Cool and disconnect the extraction flask. Evaporate the solvent from the oil extract on a water bath until no trace of the solvent remains. Evaporation of the solvent should be complete within approximately 20 minutes. In case of doubt, allow flask to remain on the water bath for an additional 15 minutes and rotate the flask slowly. Remove the flask from water bath, cool to room temperature and weigh.

4. Calculations:

a) Oil in ground kernels, $\% = \frac{\text{Weight of oil} \times 100}{\text{Weight of sample}}$

The per cent oil is calculated to any desired moisture basis with the following formula:

b) Oil, moisture desired basis, % =

$$\frac{\text{F } (100 - \% \text{ moisture desired})}{100 - \% \text{ moisture in ground sample}}$$

F = % oil determined in ground sample

c) Oil in tung fruit, % = % oil in tung kernels × % kernels in tung fruit.

Analysis of Whole Fruit

A. PREPARATION OF SAMPLE:

1. Foreign matter: Reweigh laboratory sample, noting any loss of moisture and examine sample for foreign matter which is to be removed from that portion to be used for analysis. Pass the sample over a 6-mesh screen to remove as much foreign matter as possible and pick out the remainder by hand after spreading out on a clean, dry surface. Calculate foreign matter as shown in section on sampling.

2. Grinding for oil and moisture analysis: Grind a sample of 200-250 tung fruit in a Wiley mill using a ¼-inch screen. The Wiley mill shall be equipped with an auxiliary hopper over the regular hopper to prevent material from being thrown out and also with a tight fitting chute from the bottom of the mill through the cover of a large can into which the ground material is delivered without the possibility of spilling or drying. The whole sample of ground tung fruit shall be thoroughly mixed, first breaking up any lumps by hand, by rolling on a large piece of paper or preferably in a large Maclellen mixer (30 quarts). The sample is then subdivided by quartering with a large spatula or with a riffle, yielding duplicate portions of about 1½ quarts. One portion is used in the moisture determination and the other portion in the oil determination.

B. MOISTURE DETERMINATION (ORIGINAL):

1. Weigh duplicate 5-gram samples of Wiley-ground tung fruit into moisture dishes and place dishes with the samples in an approved forced draft oven for one hour at 101°C. Remove dishes from the oven, cover promptly, cool and weigh. Replace the dishes in the oven for one-half hour, remove from the oven, cover, cool and weigh as before. Repeat the process until loss in weight between successive weighings is not more than 5 mg., or until a gain of weight is noted. The moisture to be reported is calculated from the greatest loss found.

2. Calculations:

Moisture and volatile matter in tung fruit, % =

$$\frac{\text{Loss in weight} \times 100}{\text{Weight of sample}}$$

C. PREPARATION OF WILEY-GROUND TUNG FRUIT OIL DETERMINATION:

Grind the 1½-quart subsample of the Wiley-ground fruit in a Bauer mill No. 148 (Laboratory mill with No. 6912 plates) speed 3600 r.p.m. adjusted to produce a fine meal. Mix the ground material thoroughly by rolling on a sizable piece of paper and place in an air-tight sample container.

D. MOISTURE AND OIL IN BAUER GROUND SAMPLE:

1. Moisture: Determine moisture by the same procedure used on Wiley-ground sample.
2. Oil determination:
   a) Reagent: petroleum ether, American Oil Chemists' Society specifications.
   b) Apparatus:
      1. Butt-type extraction apparatus assembled as for cottonseed and similar analyses.
      2. Filter paper, S. & S. No. 597, Reeve Angel No. 211 or equivalent, 125 or 150 mm.
      3. Absorbent cotton.
      4. Air-tight sample containers.
   c) Procedure:
      1. Weigh duplicate 5-gram samples of the Wiley-Bauer ground material and wrap each portion in 125 or 150 mm. filter paper and rewrap in second paper or papers in such a manner as to prevent escape of meal, leaving top of the second paper open like the top of a thimble. A small piece of absorbent cotton may be placed in the top of the thimble to distribute the solvent as it drops onto the sample.
      2. Place the wrapped sample in the Butt extraction tube and assemble the apparatus in the usual manner. Place 25 to 30 ml. of petroleum ether in the Soxhlet flask before attaching the flask to the lower end of the Butt tube.
      3. Heat on a water bath at such a rate that the solvent will drop from the condenser into the thimble at approximately 150 drops per minute. Volume of solvent in the extraction flask should be kept fairly constant by adding additional solvent as necessary. Extract for 4 hours.
      4. Cool and disconnect extraction flask from Butt tube. Evaporate the petroleum ether by allowing the flask to continue to heat on the water bath until no trace of the solvent remains. In case of doubt, allow the flask to remain on the water bath for an additional 15 minutes and rotate the flask slowly. Remove flask from the water bath, cool to room temperature and weigh.
   d) Calculate oil content as shown in the following example:

Petroleum ether extract..... 0.9890 g.
Moisture (Wiley-ground).....11.4%
Moisture (Wiley-Bauer-ground).....10.0%
Weight of sample..... 5.000 g.

$$\text{Extract in fruit, \%} = \frac{0.9890}{5} \times \frac{88.6}{90.0} = 19.46\%$$

Oil in fruit, % = 19.46% − 0.40%* = 19.06%

* Correction for extractable material in hulls and shells.

Recommendations. It is recommended that:

1. The method of sampling of tung fruit which has been studied by the Subcommittee on Tung be designated as a tentative method.

2. The method of analysis, wherein the whole tung fruit are ground in a Wiley mill and subportions of the ground material used in the moisture determination and, after regrinding in a Bauer mill, used in the oil determination, be designated as a tentative method, with the use of a proper correction to be subtracted from the oil content obtained with the Wiley-Bauer ground fruit because of the extractable material from hulls and shells of tung fruit which is not oil.

3. The method of analysis, wherein the tung fruit is shelled and the moisture and oil are determined on the kernel, be designated as a tentative method.

4. Samples of tung fruit be sent out during the next season at least six times for analysis.

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METHODS OF ANALYSIS OF SOY FLOURS, OILSEED MEALS, AND COTTONSEED

At the 38th Annual Meeting the Society tentatively adopted the recommendations of this committee and the Uniform Methods Committee methods for the determination of moisture, oil, ash, nitrogen, and crude fiber in soy flours, methods for the determination of ash and crude fiber in oilseed meals, and a quick method of limited applicability for the determination of moisture in cottonseed. As these methods have not been printed with full specifications for insertion in the Official and Tentative Methods of the Society, it is recommended that they be continued on a tentative basis for another year.

At its 61st Annual Meeting (1947) the Association of Official Agricultural Chemists harmonized its tentative methods for soy flour with those adopted as tentative by the American Oil Chemists' Society [Jour. A.O.A.C., *31*, 58 (1948)].

The Subcommittee for the Analysis of Copra is making progress but is not ready to report.

This report and the recommendations have been given unanimous approval by the Seed and Meal Analysis Committee.

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