ings have been put to practical use in the refinery for over a year with satisfactory results.

Acknowledgment

The author wishes to express his appreciation to K. S. Markley, Southern Regional Research Laboratory, New Orleans, for his advice in this work and assistance in the preparation of this report.

The author also wishes to express his appreciation to the following for their assistance in collecting the data for this report: W. S. Belden, Will Frech, F. P. Roullard Jr., and L. P. Barr, all of Producers Cotton Oil Company.

[Received November 10, 1950]

Report of the Seed and Meal Analysis Committee, 1950-1951

THE work of the committee is currently conducted by five subcommittees. Status and report of them are as follows:

The Subcommittee on the Analysis of Flaxseed and Linseed Meal has written up methods for sampling, moisture, oil content, protein, and ash in flaxseed, and methods for sampling, moisture, oil content, protein, ash, and crude fiber in linseed meal. These methods are now undergoing collaborative checking. The results of analytical testing have been encouraging, and it appears probable that the methods will require only minor editorial revision before submitting for approval to the Seed and Meal Analysis Committee.

The Subcommittee for the Analysis of Copra and Copra Meal has carried on some correspondence relative to its assignment. It met and discussed the problems at the time of the fall meeting of the Society. It was agreed that the subject of determination of moisture and volatile matter should be considered first. Plans were made for exchange of analytical methods, prior to carrying out collaborative work. It is anticipated that the first collaborative work will be started soon.

The Subcommittee on the Analysis of Tung Fruit and Meal is continuing its work on methods. A report on its efforts will be offered later as the season of harvest does not permit completion of the year's work at this date.

Report of Subcommittee on Peanut Analysis

Variations in analysis reported on the Smalley Foundation check samples of peanut kernels have led to doubt of uniformity in the mixing of the sliced samples. The requiring of the use of an instrument such as the velocity mixer is indicated. In order to determine the efficiency of the velocity mixer in the preparation of sliced peanuts for analysis, a series of samples were prepared and sent to five laboratories for analysis.

Three samples, A, B, and C, were prepared. Samples A were prepared by slicing several pounds of Southeastern Runner Peanuts and mixing by rolling back and forth on a sheet of paper. Samples A were then taken at random and sealed in 6-oz. salve tins. Samples B were similarly prepared from Virginia peanuts. These samples were so selected in order to obtain as wide a variation as possible in oil content so that inefficiency of mixing would be most pronounced. Samples C were prepared separately by weighing 20 grams each of Samples A and B and mixing for 30 seconds in the velocity mixer. These 20-gram portions were all taken at random from the larger preparations.

Each collaborator was instructed to make triplicate oil determinations on each sample and to mix 20 grams each of Samples A and B and determine oil content in triplicate, moisture to be determined simultaneously with the oil. The composite sample was to be mixed as was customary in each laboratory. All collaborators used the velocity mixer with the exception of No. 4. He reports that mixing was done by hand.

The results of all individual tests are shown in Table I. All results were calculated to a dry basis for purposes of comparison.

TABLE I

Percentage of Oil in Peanut Kernels, Moisture-Free Basis

Collaborator No.	Sample A	Sample B	Sample C	Mixed A & B
1	52.41	44.87	48.11	48.66
	52.41	44.66	48.63	48.56
	52.36	44.71	48.50	48.56
Avg.	52.39	44.75	48.41	48.59
2	53.05	45.92	49.08	49.24
	52.90	45.92	49.24	49.24
	52.90	46.02	49.24	49.09
Avg.	52.95	45.95	49.19	49.19
3	52.59	44.98	48.79	48.76
	52.63	45.05	48.78	48.81
	52.64	45.04	48.82	48.83
Avg.	52.62	45.02	48.80	48.80
4	52.63	45.04	48.94	48.63
	52.59	45.10	48.94	48.52
Avg.	52.61	45.07	48.94	48.58
5	52.62	45.22	48.79	48.74
	52.72	45.16	48.95	48.64
	52.77	45.00	49.00	48.69
Avg.	52.70	45.13	48.91	48.69

It is obvious that if results obtained on Sample C (mixed prior to sending to each collaborator) and the mixed sample (prepared from A and B by each collaborator) are in agreement, the method or methods of mixing are satisfactory. This agreement is shown in Table II, where the high and low results on C and mixed A and B samples are shown for each col-

TABLE II

Highest and Lowest Result Obtained on Either Sample C or Composite of A and B

Collaborator	1	2	3	4	5
High	48.66%	49.24%	48.83%	48.94%	49.00%
Low	48.11%	49.08%	48.76%	48.52%	48.64%
Total Spread	0.55	0.16	0.07	0.42	0.36

laborator. It should be pointed out here that these variations reflect differences due to analytical technique as well as differences due to imperfect mixing. It should also be noted that these differences are the spread between maximum and minimum and not between these figures and a mean result.

Since it is possible without resorting to averages to show excellent agreement between samples prepared by mixing in the velocity mixer two such widely differing materials in different laboratories and at different times, it is recommended that Official Method Ab 3-49 be revised to require that the sliced peanut kernels be mixed by use of the velocity mixer before the analytical sample is drawn for analysis, and that this method be continued as official.

THOS. C. LAW, chairman
E. C. AINSLIE PAUL D. CRETIEN
C. H. COX M. L. HARTWIG

Report of Subcommittee on Screen Test for Soyflour

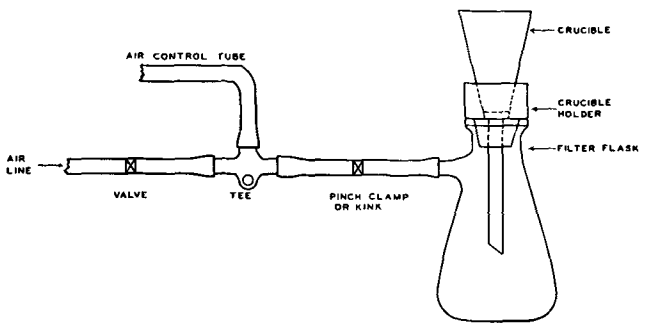
During the past four years much effort has been directed toward the development of a suitable screen test applicable to all grades and types of soyflour. The chief difficulty has been due to the fact that the fat content of the soyflours varies from 0.5 or less to 22.0%. In addition, some soyflours are reconstituted from de-fatted flour by the addition of oil or lecithin, or both. Because of the variability in fat content, the ordinary brushing and/or shaking procedures for screen size are not suitable. A wide range of flour types was examined by all of the presently used methods. Large discrepancies were found in the results reported by different laboratories. Subsequently a method was developed which involves the use of a fat solvent and washing the flour through a standard screen. This method is an adaptation of at least two somewhat similar methods which are in limited current use, one of which appears in the handbook of methods of the Soy Flour Manufacturers Association. The specifications of the method developed are as follows:

Definition. This method determines the quantity of material coarser than specified screen size under the conditions of the test.

Scope. Applicable to high fat, low fat, de-fatted and reconstituted soyflours.

A. Apparatus:

1. Metal crucible 2" high, top diameter 2", bottom diameter 1", fitted with specified U. S. Standard (or Tyler equivalent) wire screen, 200, 325, or other mesh as the need may be.
2. Walter crucible holder.
3. 250-ml. filter flask.
4. Air supply, tubing, and tee.



B. Preparation of Sample:

1. Mix sample thoroughly in a closed container by turning the container manually or mechanically. Reduce the sample to suitable size and store in an air-tight container.

C. Procedure:

1. Weigh 1.000-gram sample into crucible.
2. Crack air valve to give maximum agitation without causing sample to slop over. (20-30 ml./sec.)
3. Place crucible tightly in holder in filter flask and close air line to flask. (Clamp, stop cock, or tubing may be made long enough to permit operator to hold a kink in the tubing.)
4. Add 15 ml. CCl_4 around sides of crucible by means of burette, graduated pressure bottle, or graduated cylinder.
5. Place finger over air control tube as air line to flask is opened. When flour is well mixed due to aeration (less than ½ ml. solvent should have passed through screen at this point), release air control tube for fraction of a second. Continue manipulating air control tube with finger so that air pressure is on crucible 2 seconds and released fraction of a second until only about 1-2 ml. of solvent remains in the crucible. At this point remove finger from air control tube and shake or rotate crucible and flask until the remaining solvent has drained into filter flask.
6. Repeat above steps using successively 10-ml. and four 5-ml. portions of solvent.
7. Remove crucible from holder and wash residue 3 times with 1½ ml. of solvent.
8. Wipe sides and bottom of crucible with a towel. Let solvent evaporate in air or oven for a few minutes and transfer grits to small tared pan. Heat in oven ½ hour at 110°C. or 15 min. at 135°C. and determine weight of grits. If desirable, weighing may be made directly in crucible.

D. Calculation:

% Coarser than specified screen size = grams retained × 100.

TABLE III

Collaborative Results on Screen Tests on Soyflour
(Per cent retained on mesh indicated)

Laboratory	Results of Replicate Tests						Average	Range
	1	2	3	4	5	6		
	%	%	%	%	%	%	%	%
	Extracted flour—200 mesh							
1	4.00	4.14	3.73	3.96			3.96	0.41
2	4.74	4.70	4.55	4.21	4.25		4.49	0.53
3	3.98	4.07	4.37	3.92	4.16	4.22	4.12	0.45
	Expeller low fat flour—200 mesh							
1	7.54	7.30	6.59	7.14			7.14	0.95
2	6.59	7.10	6.77	6.33	6.54		6.67	0.77
3	5.13	5.12	5.27	5.16	5.42	5.25	5.22	0.30
	Full fat flour—200 mesh							
1	7.00	6.40	6.85	6.75			6.75	0.60
2	6.80	6.96	7.20	7.00	6.75		6.94	0.45
3	6.30	6.41	6.64	6.67	5.99	6.60	6.43	0.68
	Reconstituted extracted flour—5% fat—200 mesh							
1	2.70	2.04	2.03	2.26			2.33	0.67
2	2.00	2.06	2.12	1.89	2.14		2.04	0.25
3	2.19	2.07	2.23	2.03	2.08	2.25	2.14	0.22
	Reconstituted extracted flour—14% fat—200 mesh							
1	10.95	9.50	11.20	10.55			10.55	1.70
2	9.15	9.20	8.66	8.39	9.00		8.88	0.81
3	8.10	7.79	8.99	8.42	8.03	8.87	8.37	1.20
	Lecithinated extracted flour—8% fat—200 mesh							
1	10.50	8.94	9.05	9.49			9.47	1.56
2	9.66	9.13	9.41	9.33	9.18		9.34	0.53
3	9.35	8.81	8.42	9.80	8.80	9.08	9.04	1.38
	Extracted flour—325 mesh							
1	0.50	0.50	0.50	0.50			0.50	0
2	0.61	0.74	0.59	0.61	0.62		0.63	0.15
3	0.78	0.88	0.83	0.72	0.62	0.48	0.72	0.26

E. Notes:

1. At no time should solvent be applied directly on the screen.
2. After adding first 5 ml. wash, any clump of flour floating on top of the solvent should be broken up with small spatula, wire, or rod. The spatula may be rinsed off when adding the second 5-ml. wash portion.
3. When necessary to use sieve such that more than 15% grits remain on the sieve, two 10-ml. washings instead of one portion should be used.
4. The method depends on:
   a) Forcing air back through the screen to clean the screen and obtain complete mixing of solvent and flour and
   b) Permitting only small increments of the solvent and flour to pass through the screen at one time so that no filter cake is built up in the screen.

The amount of data accumulated during the collaborative study is voluminous. It is not submitted with this report. A tabulation of the results of tests made by three laboratories on the final set of seven samples is given in Table III. Results of a fourth laboratory, inexperienced in the use of the method, were approximately 1% higher than those of the ones reporting, indicating the need of some experience before one will obtain command of the techniques involved to obtain comparable results.

The subcommittee agrees that the method is not entirely satisfactory in all respects. As it feels confident however that it is superior to any known method investigated, it recommends that the method be adopted as tentative by the Society.

L. R. BROWN, chairman — M. W. DIPPOLD
R. E. ANDERSON — F. R. EARLE

As the recommendations of the two subcommittees have received a favorable vote by all except one member, in each case, of the Seed and Meal Analysis Committee, it is recommended that they be adopted by the Society.

T. H. HOPPER, chairman

E. C. AINSLIE — T. C. LAW
L. R. BROWN — R. S. MCKINNEY
C. H. COX — V. C. MEHLENBACHER
F. R. EARLE — T. J. POTTS
E. B. FREYER — T. L. RETTGER
J. C. KONEN — T. C. SMITH

Buckwheat Leaf Meal Fat. I. Its Physical and Chemical Characteristics and the Constituents of the Water-Soluble and Unsaponifiable Fractions of the Saponified Fat

CHARLES F. KREWSON and JAMES F. COUCH, Eastern Regional Research Laboratory,[1] Philadelphia 18, Pennsylvania

DURING the preparation of rutin from fresh green buckwheat plant (5) or from a leaf meal made by dehydrating the green plant (5, 6, 20), a fatty fraction is obtained as a by-product. The composition of this fraction has not been reported, and since the material is available in quantity, it was examined primarily to determine whether it contains constituents of economic importance. Furthermore few reports have been made on the critical examination of fats from the tissue of green plants by recently improved methods. The results of this study are reported in two papers. This paper describes the physical and chemical characteristics of the buckwheat leaf meal fat and includes a report on the composition of the water-soluble and the unsaponifiable constituents of the saponified fat. The examination and identification of the water-insoluble constituents of the saponified fat will be described later.

The characteristics of the leaf meal fat were determined either by official methods (1) or by methods referred to in the references. The results are recorded in Table I.

Japanese buckwheat leaf meal fat was used in this investigation. It does not differ greatly in chemical composition from that obtained from Tartary buckwheat, the variety now preferred for rutin manufacture (7). This was demonstrated by spectrophotometric examination of the crude fats for polyunsaturated acids (2, 3). Table II shows the results of this exami-

[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, United States Department of Agriculture.

TABLE I

Characteristics of Fat from Japanese Buckwheat Leaf Meal [1]

Fat content,[2] %	5.8
Moisture and volatile matter,[3] %	6.6
Specific gravity, 25°/25°C.	1.0823
Saponification equivalent (1)	298.7
Hydroxyl value (25)	7.6
Acid value (1)	21.3
Iodine number (1)	123.9
Reichert-Meissl value (1)	7.04
Polenski value (1)	0.41
Water-soluble acids (calc. as butyric, %), (1)	12.5
Insoluble acids,[4] % (1)	64.9
Unsaponifiable matter, % (30)	14.9
Chlorophyll, % (1)	2.8
Red pigment, % (28)	0.50
Carotene,[5] % (31)	0.30
Choline, % (10)	0.30
Ash, % (1)	1.98
Nitrogen, % (33)	1.22
Phosphorus, % (1)	0.45
Sulfur, % (26)	0.21
Magnesium, % (16)	0.13

[1] All values reported are on a moisture-free basis. Numbers in parentheses refer to literature citation.

[2] Benzene-soluble portion of the concentrated ethanol extract of the meal.

[3] By vacuum drying to constant weight at room temperature.

[4] Free of unsaponifiable matter but contained 28.5% petroleum ether insoluble material.

[5] Sample of fat stored for 3 years.

TABLE II

Spectrophotometric Analysis of Polyunsaturated Fatty Acids in Buckwheat Fat

	Japanese	Tartary
	%	%
Linoleic acid	6.6	9.6
Linolenic acid	16.3	13.9
Conjugated dienoic acid	6.0	4.2
Total	28.9	27.7