The infrared method is suggested as a necessary tool to investigators conducting research on the oxidation, isomerization, polymerization, composition, and hydrogenation of fats and their components and derivatives, and on the preparation of pure unsaturated acids and esters.

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Report of the Seed and Meal Analysis Committee, 1949

THIS special report of the Seed and and Meal Analysis Committee is submitted because lateness of the season of harvesting and processing tung fruit does not allow time for the Subcommittee on the Analysis of Tung Fruit and Meal to report immediately at the annual meeting.

Report of the Subcommittee on the Analysis of Tung Fruit and Meal 1948-49

During the last year the members of the Subcommittee on the Analysis of Tung Fruit and Meal have continued the study of the analysis of samples of tung fruit by the whole fruit procedure and by the component procedure.[1] Six lots of tung fruit, varying widely in moisture, oil, and hull content and weighing about 100 pounds each, were thoroughly mixed, and each lot was divided by the use of a riffle into seven large subsamples of 200-250 fruit each. Each subsample was immediately placed in a 50-pound lard can, which was sealed by using cellulose tape around the edge of the top and was shipped to the collaborators for analysis. Four collaborators analyzed these samples by the whole fruit procedure, one collaborator used the component procedure while the sixth collaborator, who received two large subsamples from each lot of tung fruit, analyzed one subsample from each lot by the whole fruit procedure and the other subsample from each lot by the component procedure. The results obtained by the collaborators are given in Table I. It will be noted that no correction has been applied to the oil content obtained by the whole fruit procedure because of the extractable material in the hulls and shells of the tung fruit which is not oil. The results for oil content have been recalculated to include the foreign matter. As two collaborators did not report foreign matter separately, it was necessary to make these calculations in order to include the results of all the collaborators on the same basis.

A statistical study of the results reported for oil content of the tung fruit samples by the collaborators in 1947-48 [1] and in 1948-49 have shown no significant difference between the results obtained using the same procedure or between the results obtained using the whole fruit procedure and the component procedure for the oil determination. In addition, no correction was indicated in the oil content obtained by the whole fruit procedure to obtain results in agreement with those obtained by the component procedure. Using no correction in the oil content obtained by the the whole fruit procedure, the average of the results of the collaborators for oil content of the tung fruit samples by the whole fruit procedure and by the component procedure was 20.24% and 20.34%, respectively, for 1947-48, and was 18.14% and 18.22%, respectively, for 1948-49.

In contrast to the good agreement shown by the collaborators in the results reported for the oil content of the tung fruit samples, the results reported for moisture content of the tung fruit samples have shown a rather wide variation, particularly when the component procedure was used. The differences between the average moisture content of the samples obtained by the collaborators are highly significant and indicate that serious differences might be encountered when the oil content of the tung fruit is calculated to a dry basis. It appears that the variations in the moisture results are probably due to the use of several different methods in the moisture determinations by the collaborators either because of the

TABLE I

Analysis of Collaborative Samples by Component and Whole Fruit Procedures, 1948-49

Collaborator	Per Cent Oil in Tung Fruit						
	Sample Number						
	1	2	3	4	5	6	Average
1[1]	16.9	11.6	17.8	23.6	19.3	21.0	18.37
2[1]	16.1	11.7	18.2	23.8	19.2	19.4	18.07
2[2]	16.3	12.1	18.3	22.9	19.4	19.5	18.08
3[2]	16.4	11.4	18.3	23.3	18.7	20.2	18.05
4[2]	15.8	11.7	17.5	22.8	19.7	20.1	17.93
5[2]	15.9	11.8	18.9	23.0	19.2	19.9	18.12
6[2]	16.7	11.3	18.3	23.6	20.2	21.1	18.53
Average[1]	16.50	11.65	18.00	23.70	19.25	20.20	18.22
Average[2]	16.22	11.66	18.22	23.12	19.44	20.16	18.14
Average	16.30	11.66	18.19	23.29	19.39	20.17	18.16
	Per Cent Moisture in Tung Fruit						
1[1]	26.4	32.0	25.0	10.0	18.0	11.0	20.40
2[1]	29.8	33.3	27.7	9.1	19.6	14.6	22.35
2[2]	29.0	32.2	25.8	9.0	18.3	12.8	21.18
3[2]	27.7	31.4	25.1	9.7	18.0	12.4	20.72
4[2]	28.5	30.9	26.8	10.0	18.2	12.4	21.13
5[2]	28.9	32.3	25.1	10.3	18.0	11.8	21.07
6[2]	28.7	33.5	27.2	10.1	18.4	12.8	21.78
Average[1]	28.1	32.7	26.4	9.6	18.8	12.8	21.42
Average[2]	28.6	32.1	26.0	9.8	18.2	12.4	21.18
Average	28.4	32.2	26.1	9.7	18.4	12.5	21.23

[1] Used component procedure.
[2] Used whole fruit procedure.

[1] Report of Subcommittee on Analysis of Tung Fruit and Meal, J. Am. Oil Chem. Soc., *25*, 321 (1948).

lack of a forced draft oven in the laboratory or perhaps to a preference for another moisture procedure other than that specified in the tentative methods of the Society for the moisture determination of tung fruit. In this method the moisture determination of tung fruit is made on duplicate 5-gram samples of the Wiley-ground tung fruit in a forced draft oven at 101°C., for one hour and subsequently at one-half hour periods until the loss of weight between successive weighings is not more than 5 mg. or until a gain in weight is noted. In our report for 1947-48 [1] it was shown that the moisture method used will affect the moisture results but will not affect the estimation of the oil content, provided the same moisture method is employed with the Wiley-ground and the Wiley-Bauer ground samples.

Studies in the laboratory of one of the collaborators have indicated that the accurate determination of moisture in tung fruit is a difficult operation. The Bidwell-Sterling procedure probably gives the most accurate estimation of the water content of tung fruit although only slightly less reliable results can be obtained by the use of a vaccum oven if a proper drying time is used. With a pressure of 5 mm. of mercury it usually requires a three-hour period for the coarsely ground tung fruit samples from the Wiley mill while a 2½-hour period is required for the finely ground material from the Bauer mill. The Subcommittee Chairman hopes that all the collaborators will have their laboratories equipped with a forced draft oven by next year so that they can all use the tentative moisture method of the Society in the collaborative work next year. It is believed that this will result in better agreement in the moisture results.

Five lots of tung press cake were coarsely ground and thoroughly mixed; six subsamples were drawn from each lot using random sampling. Each subsample was placed in a 2-quart friction top can, the lid tightly sealed, and was shipped to the collaborators for analysis for moisture, oil, nitrogen, and ash content. The results obtained are given in Table II.

In the report of the Subcommittee on the Analysis of Tung Fruit and Meal last year [1] alternate methods for the analysis of tung fruit and in some instances alternate equipment were specified. As this does not conform to the policy of the Society, these methods have been modified to eliminate this objection. The specifications of the methods for sampling tung fruit, for analysis of tung fruit, for the physical analysis of tung fruit for components, and for the analysis of tung kernels are:

TABLE II

Analysis of Collaborative Samples of Tung Press Cake, 1948-49

Collaborator	Sample Number					
	1	2	3	4	5	Average
	Per Cent Moisture					
1	(1.6)	(1.0)	(3.3)	....	....	....
2	4.2	3.6	8.6	4.8	5.6	5.4
3	3.6	2.7	7.5	4.6	4.8	4.6
4	4.1	3.1	7.3	4.8	4.6	4.8
5	4.0	2.4	7.6	4.5	4.5	4.6
6	3.7	2.7	7.7	4.6	....	....
Average	3.9	2.9	7.7	4.7	....	....
	Per Cent Oil					
1	4.9	6.7	5.9	....	....	....
2	4.4	6.4	5.8	4.8	5.5	5.4
3	4.8	6.1	5.6	4.8	5.5	5.4
4	4.5	5.9	5.7	4.6	5.5	5.2
5	4.7	6.1	5.6	4.7	5.4	5.3
6	4.9	6.2	5.8	4.8	....	....
Average	4.7	6.2	5.7	4.7	5.4	5.3
	Per Cent Nitrogen					
1						
2	2.89	2.85	3.12	2.84	3.27	2.97
3	3.18	3.25	3.02	3.15	3.31	3.18
4	3.20	3.28	3.08	3.10	3.34	3.20
5	3.21	3.23	2.94	3.05	3.29	3.14
6	3.63	3.45	3.18	3.23	....	....
Average	3.22	3.21	3.05	3.07	3.30	3.12
	Per Cent Ash					
1	4.85	4.79	4.20	....	....	....
2	4.15	4.40	4.15	4.35	4.15	4.24
3	4.47	4.25	3.99	4.39	4.02	4.22
4	4.38	4.23	4.04	3.88	4.05	4.12
5	4.38	4.20	3.92	4.02	4.02	4.11
6	4.7	4.7	4.5	4.40	....	....
Average	4.49	4.43	4.13	4.21	4.06	4.17

A.O.C.S. Tentative Method Ad 1-48

SAMPLING

Scope: Applicable to sampling tung fruit during unloading from truck, wagon, or carload lots.

A. APPARATUS:

1. Sampling bucket which may be prepared by attaching a bucket of about 6 x 6 x 6 inches to a pole of convenient length.
2. Sample container of convenient size and with a tight-fitting cover. A conventional 50-pound, lard or shortening can is usually satisfactory.
3. Sieve, 6-mesh, diameter 12 to 15 inches.
4. Scales, capacity 2,000 to 3,000 grams and sensitive to 1 gram.

B. PROCEDURE:

1. Take bucket full from the center of the unloading chute at regular intervals such that the gross sample will fill the 50-pound can.
2. Collect the samples in the can and keep tightly covered at all times except when samples are being placed therein.

C. CLEANING LABORATORY SAMPLE:

1. Pass the entire sample over a 6-mesh sieve to remove foreign matter. Remove by hand-picking any foreign matter which is not removed by the sieve. Collect and weigh the foreign matter as well as the cleaned sample.
2. Record the net weight of the sample on the identification tag which is submitted with the sample to the laboratory.
3. Calculate the foreign matter as follows:

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

A.O.C.S. Tentative Method Ad 2-48

MOISTURE AND VOLATILE MATTER

Definition: This method determines moisture and any material which is volatile under the conditions of the test.

Scope: Applicable to whole tung fruit.

A. APPARATUS:

1. Forced draft oven, A.O.C.S. Specifications H 1-39.
2. Sample riffle, the riffle should have eight 1¾-inch slots. Three metal boxes are necessary, ca 2 inches high, and of such dimensions that the riffle may stand and discharge into these boxes.
3. Sieve, 6-mesh, 12- to 15-inch diameter.
4. Wiley sample grinding mill with ¼-inch screen. Equip the mill with an auxiliary hopper fitted above the regular one to prevent material from being thrown out. Also provide a tight-fitting chute to connect from the bottom of the mill through the cover of a large can into which the ground material is delivered. This is to insure against loss of ground sample and drying out.
5. Air-tight sample containers for holding ground samples.
6. Aluminum moisture dishes, 30-gauge, 2 x ¾ inches (50 x 19 mm.) with tight-fitting slip-over covers.
7. Desiccator, containing an efficient desiccant. Calcium chloride is not satisfactory. See A.O.C.S. Specification H 9-45.

B. REMOVAL OF FOREIGN MATTER:

1. Weigh the gross sample and compare with original weight recorded on tag to see if there has been any change in moisture.
2. Pass the entire sample over a 6-mesh sieve to remove foreign matter. Remove by hand-picking any foreign matter which is not removed by the sieve. Collect and weigh the foreign matter as well as the cleaned sample.
3. Calculate the foreign matter as follows:

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

C. PREPARATION OF SAMPLE:

1. Grind whole tung fruit picked at random from the gross sample through the Wiley mill using a ¼-inch screen. If oil is to be determined on the whole fruit, grind 200-250 of the tung fruit. If moisture and volatile matter only are to be determined, grind 25 of the tung fruit.
2. Break up any lumps of the ground material and then mix thoroughly.
3. Reduce the ground sample, either through a riffle or by quartering to a subsample of about 2 pounds and store in air-tight container. Quartering is done by dividing the ground and well-mixed sample pile into four approximately equivalent quarters with a spatula. Discard two diagonally opposite quarters. Combine and remix the two remaining quarters and then re-quarter as before. Continue in this manner until the sample is reduced to an appropriate size.

D. PROCEDURE:

1. Weigh duplicate 5-gram samples of the ground tung fruit into tared moisture dishes.
2. Slip cover on the bottom of the dish and place the uncovered dish in the oven and dry at 101°C. for one hour.
3. Remove the dishes from the oven, cover promptly, cool in a desiccator to room temperature and weigh.
4. Repeat with heatings of ½-hour periods until the loss in weight between successive weighings does not exceed 5 mg., or until a gain in weight is noted. Report as moisture the greatest loss found.

E. CALCULATION:

$$\text{Moisture and volatile matter, \%} = \frac{\text{Loss in weight} \times 100}{\text{Weight of sample}}$$

A.O.C.S. Tentative Method Ad 3-48

OIL

Definition: This method determines the substances extracted by petroleum ether under the conditions of the test.

Scope: Applicable to whole tung fruit.

A. APPARATUS:

1. Bauer Mill No. 148 with plates No. 6912 so adjusted as to produce a fine meal.
2. Wiley sample grinding mill with ¼-inch screen. Equip the mill with an auxiliary hopper fitted above the regular one to prevent material from being thrown out. Also provide a tight-fitting chute to connect from the bottom of the mill through the cover of a large can into which the ground sample is delivered. This is to insure against loss of ground sample and drying out.
3. Butt type extraction apparatus, assembled as indicated in the illustration, A.O.C.S. Method Aa 4-38.
4. Filter paper, S & S No. 597, Reeve Angel No. 211, Whatman No. 2 or equivalent, 150 mm.
5. Absorbent cotton, free of petroleum ether extract.
6. Air-tight sample containers for holding ground samples.
7. Forced draft oven, A.O.C.S. Specification H 1-39.

B. REAGENTS:

1. Petroleum ether, A.O.C.S. Specification H 2-41.

C. PREPARATION OF SAMPLE:

1. Use a 2-pound portion of Wiley-ground sample prepared as directed in A.O.C.S. Tentative Method Ad 2-48, C.
2. Grind this entire portion through the Bauer Mill. Mix carefully by rolling on a large sheet of paper and place in air-tight container.

D. PROCEDURE:

1. Weigh accurately duplicate 5-gram ground samples into filter papers and enclose each sample in a second paper, folded in such a manner as to prevent escape of meal (see illustration in A.O.C.S. Method Aa 4-38). The second paper is left open like a thimble. A piece of absorbent cotton may be placed in the top of the thimble to distribute the solvent as it drops on the sample.
2. Place wrapped samples in Butt extraction tubes and assemble the apparatus as shown in A.O.C.S. Method Aa 4-38. Place 25 to 30 ml. of petroleum ether in the extraction 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 loss due to evaporation. Extract for 4 hours.
4. 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.
5. Determine moisture in Wiley-Bauer ground sample as directed in A.O.C.S. Tentative Method Ad 2-48, D.

E. CALCULATION:

$$\text{Oil in whole tung fruit, \%} = \frac{A \times (100 - B)}{D \times (100 - C)}$$

A = Grams oil extracted from D.

B = Per cent moisture from Wiley-ground portion, A.O.C.S. Tentative Method Ad 2-48, E. (Original Moisture)

C = Per cent moisture from Wiley-Bauer-ground portion, A.O.C.S. Tentative Method Ad 3-48, D. (Second Moisture)

D = Weight of sample from A.O.C.S. Tentative Method Ad 3-48, D.

A.O.C.S. Tentative Method Ad 4-48

PHYSICAL ANALYSIS OF TUNG FRUIT

Definition: This method determines the kernel content of tung fruit under the conditions of the test.

Scope: Applicable to whole tung fruit.

A. APPARATUS:

1. Scales, capacity 5000-gram and sensitive to 1 gram.

B. PROCEDURE:

a) Determination of the amount of kernels:

1. Weigh a sample of at least 100 tung fruit. Manually remove the hulls and shells from the kernels and weigh the tung kernels.

$$\text{Kernels in tung fruit, \%} = \frac{\text{Weight of kernels} \times 100}{\text{Weight of whole fruit sample}}$$

A.O.C.S. Tentative Method Ad 5-48

OIL

Definition: This method determines the substances extracted by petroleum ether under the conditions of the test.

Scope: Applicable to tung kernels.

A. APPARATUS:

1. Universal Food Chopper No. 71 with 16-tooth blade or Bauer Mill No. 148 with plates No. 6912 so adjusted as to produce a fine meal, without extrusion of oil.
2. Butt type extraction apparatus, assembled as indicated in the illustration, A.O.C.S. Method Aa 4-38.
3. Filter paper S & S No. 597, Reeve Angel No. 211, Whatman No. 2 or equivalent, 150 mm.
4. Absorbent cotton, free of petroleum ether extract.
5. Air-tight sample containers for holding ground samples.
6. Forced draft oven, A.O.C.S. Specification H 1-39.
7. Porcelain mortar and pestle, the mortar must be at least 4 inches i.d. at the top. The pestle handle must be large

enough to afford a firm hand grip. The inner surface of the mortar is kept rough by occasionally grinding sand in it.

8. Sand, fine (Sea Sand, Merck, Reagent grade, or equivalent).

B. REAGENTS:

1. Petroleum ether, A.O.C.S. Specification H 2-41.

C. PROCEDURE:

a) Preparation of sample:

1. Grind the kernels separated from at least 100 tung fruit twice in a Universal Food Chopper, using the 16-tooth blade or in a Bauer Mill with No. 6912 plates.
2. Immediately place in sample bottle of convenient size and stopper tightly. If sample is too large, quarter down to desired quantity.

b) Determination of oil in kernels:

1. Weigh accurately duplicate 5-gram ground samples into filter papers and enclose each sample in a second paper, folded in such a manner as to prevent escape of meal (see illustration in A.O.C.S. Method Aa 4-38). The second paper is left open like a thimble. A piece of absorbent cotton may be placed in the top of the thimble to distribute the solvent as it drops on the sample.
2. Place wrapped samples in Butt extraction tubes and assemble the apparatus as shown in A.O.C.S. Method Aa 4-38. Place 25 to 30 ml. of petroleum ether in the extraction 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 loss 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. Re-wrap the sample and continue extraction for an additional 2 hours. Occasionally check the efficiency of extraction by regrinding sample 5 minutes and extracting for another 2-hour period.
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 water bath for an additional 15 minutes and rotate the flask slowly. Remove the flask from water bath, cool to room temperature and weigh.
6. Determine moisture on the ground sample as directed in A.O.C.S. Tentative Method Ad 2-48, D.

D. 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, $\% =$

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

$F = \%$ oil determined in ground sample.

Recommendations: It is recommended that:

1. The methods for the sampling and analysis of tung fruit, the physical analysis of tung fruit, and the analysis of tung kernels as described in this report be continued as tentative.
2. That the assignment of the Subcommittee on the Analysis of Tung Fruit and Meal be extended for another year.
3. That samples of tung fruit be sent out during the next year at least six times for analysis.
4. That attention be paid by the Subcommittee during the next season to determining the "true" moisture content of tung products and to the grinding of tung kernels and seeds for analysis.
5. That no correction be applied to the oil content obtained by the whole-fruit procedure since there has been found to be no difference between the results reported for oil content of tung fruit samples analyzed using the whole fruit procedure and the component procedure.

G. WORTHEN AGEE — CHARLES RUSSELL CAMPBELL
G. F. BAILEY — G. CONNER HENRY
BRICE L. CALDWELL — R. S. MCKINNEY, chairman

The recommendations of the Subcommittee on the Analysis of Tung Fruit and Meal have received unanimous approval of the Committee and are recommended for approval by the Society.

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