extent. At the same time we have a smaller amount of conjugated linoleic acid. Figure 2 gives a diagrammatic illustration of the different reactions. At 235°C. reactions 1, 2, and 6 seem to be predominating. At lower temperatures 3 and 4 will predominate.

It is well known that high temperature promotes the selectivity of the nickel-hydrogenation catalyst. The present investigation indicates that the dependence of the conjugation on the temperature may be an explanation. High temperature during hydrogenation causes high isomerization and increased reactivity toward hydrogenation.

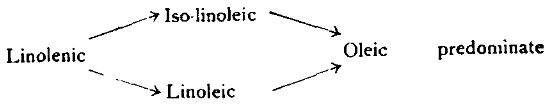
We recognize that by the calculation of composition by difference (see Table II) a relatively small error can greatly influence the final results. We know however that our experimental data are of sufficient accuracy for the purposes of discussion in this paper.

Summary

The fatty acid composition of hydrogenated linseed oil has been calculated from the iodine value, ultraviolet absorption with and without alkali isomerization, and the contents of saturated acids determined by the Bertram oxidation method. The hydrogenations are carried out at 160°C. and 235°C. The isomerization effect of the catalyst at the two temperatures is shown by heating the oils with 0.2% Ni-catalyst at 160°C. and 235°C. in CO_2-atm. The analyses show:

1. At 235°C. the conjugation of the double bonds is very large compared with 160°C.
2. The hydrogen seems to serve as an additional activator for the isomerization.
3. By hydrogenation at 235°C. 14% conjugated linoleic acid is found in the hydrogenated fat. By 160°C. only 3.6% of the acid is found. The contents of conjugated linoleic acid are due to the isomerization effect of the catalyst.
4. By 235°C. the reactions: linolenic → conjugated linolenic → conjugated linoleic → oleic, predominate.

By 160° C the reactions:



5. A theory is advanced that the temperature influences the selectivity through the conjugation of the double bonds.

REFERENCES

1. Mitchell, J. H., Kraybill, H. R., and Zscheile, F. P., Ind. Eng. Chem., Anal. Ed., *15*, 1-3 (1943).
2. Lemon, H. W., Can. J. Research, *22 F*, 191-198 (1944).
3. Mattil, K. F., Oil and Soap, *22*, 213-215 (1945).
4. Daubert, B. F., and Filer, L. J., Oil and Soap, *22*, 299-302 (1945).
5. Fischer, G. S., O'Connor, R. T., and Dollear, F. G., Am. Oil Chem. Soc., *24*, 382-387 (1947).
6. Bailey, A. E., and Fischer, G. S., Oil and Soap, *23*, 14-18 (1946).
7. Bailey, A. E., Am. Oil Chem. Soc., *26*, 644-648 (1949).
8. Rebello, D., and Daubert, B. F., Am. Oil Chem. Soc., *28*, 183-185 (1951).
9. Thompson, S. W., Am. Oil Chem. Soc., *28*, 339-341 (1951).
10. Radlove, S. B., Teeter, H. M., Bond, W. H., Cowan, J. C., and Kass, J. P., Ind. and Eng. Chem., *38*, 997-1002 (1946).
11. Blekkingh, J. J. A., Faraday Soc. Discussion No. 8 (1950).

[Received December 27, 1951]

Report of the Seed and Meal Analysis Committee, 1951-1952

THE current work of the Seed and Meal Analysis Committee is conducted by five subcommittees. They are: Screen Test for Soyflour; Analysis of Copra and Copra Meal; Analysis of Flaxseed and Linseed Meal; Analysis of Tung Fruit and Meal; and Analysis of Castor Beans and Pomace. Reports and recommendations of the last two subcommittees are as follows:

Analysis of Tung Fruit and Meal

DURING the past three years the present tentative methods for the sampling and analysis of tung fruit have been studied collaboratively. In addition, the forced draft oven method (Ad 2-48) has been compared with a vacuum oven method (A.O.A.C., 7th Edition, 1950, Moisture, 22.3, maintaining a vacuum of less than 50 mm. of mercury and heating for 2.5 to 3.0 hours), and Bidwell-Sterling distillation method (A.O.A.C., 22.4, using ground glass jointed apparatus). The results obtained by the members of the committee for samples of hulled tung fruit, tung kernels, and tung hulls by use of the A.O.C.S. method showed good agreement and were slightly lower than those obtained using the A.O.A.C. methods on the same samples. The moisture obtained by the distillation averaged 0.2% in units higher and those obtained by the vacuum oven method 0.4% in units higher than those obtained using the present tentative method. The present tentative method (Ad 2-48) appears satisfactory for evaluating samples for commercial transactions and control analysis at tung mills.

Good agreement has been found between the results for the analysis for the oil content by the whole fruit method (Ad 3-48) and those obtained by use of the physical analysis method (Ad 4-48) and the kernel method (Ad 5-48). The average oil content of six samples was 19.98% and 20.02%, respectively. The analysis of samples of hulled tung fruit for oil content by the kernel method (Ad 5-48) showed considerable variation in reported results, with standard deviations varying from ± 0.7 to ± 2.7% and an average of 1.5% oil.

As growers are marketing hulled tung fruit in increasing quantities, interest was directed toward developing a method for their analysis. An adaptation of the whole fruit methods for moisture (Ad 2-48) and oil (Ad 3-48) was investigated. It involved adding an equal weight of ground tung fruit hull to the Wiley-ground hulled tung fruit. The results of the initial analysis of six samples by the six members of the subcommittee were rather disappointing as the standard deviation of the results was ± 2.3% oil. In view of the excellent results by use of the whole fruit method (Ad 3-48) it appeared probable that faulty sampling techniques were responsible for this variation.

Studies conducted in the laboratory of one of the committeemen on the new procedure for the analysis of hulled tung fruit indicated the necessity of carefully riffling the Wiley-ground hulled fruit and the Bauer-ground mixture of hulled fruit and hulls to obtain uniform aliquot portions. It was also indicated that the aliquot to be used as the sample for analysis should be mixed in a Velocity Mixer to obtain a homogeneous mixture from which representative samples can be weighed for analysis. In addition, it was found essential to take into account the petroleum ether extractable material in the hulls added to the Wiley-ground hulled tung fruit before grinding the mixture in the Bauer mill. The quantity of extractives varies between lots of hulls.

The procedure later submitted to collaborative testing is given in this report. The oil contents of six samples of hulled tung fruit obtained by use of the kernel method (Ad 4-48 and Ad 5-48) by two members of the committee are compared with those obtained by use of the proposed new method in Table I. It is deemed that the proposed new method is satisfactory and will fill a definite need of the tung industry.

TABLE I

Oil Contents of Hulled Tung Fruit

Sample No.	Methods and laboratory							
	Kernel method			New method				
	1	2	Mean	3	4	5	6	Mean
	%	%	%	%	%	%	%	%
1	43.5	42.4	43.0	43.0	43.0	44.0	43.6	43.4
2	36.9	34.4	35.6	35.6	38.6	37.0	36.5	37.0
3	41.4	40.5	41.0	40.6	41.2	39.8	40.2	40.4
4	40.3	39.3	39.8	40.6	39.8	39.2	38.7	39.6
5	42.2	40.2	41.2	41.3	43.6	43.4	43.1	42.8
6	37.5	37.3	37.4	......	37.6	38.0	38.8	38.1

Adoption of the proposed method for the analysis of hulled tung fruit for oil will entail revision of the sampling procedure (Ad 1-48) and the method for the determination of moisture and volatile matter (Ad 2-48) to provide for the sampling and analysis of hulled tung fruit. The statements of the revisions of these two methods and the proposed method for the determination of oil in hulled tung fruit are as follows:

Sampling

Scope. Applicable to sampling tung fruit or hulled 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 in.
4. Scales, capacity 2,000 to 3,000 g. and sensitive to 1 g.
5. Riffle, convenient size. One with 12 chutes, about 1¼ in. in width, is satisfactory. Three metal boxes, *ca.* 2 in. high and of such dimensions that the riffle may stand and discharge into these boxes, are necessary.

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 Samples of Fruit

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:

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

D. Riffling Laboratory Sample of Hulled Fruit

1. Pass the entire sample over the riffle. Repeat until two subsamples of about 1 gal. are obtained.
2. Place laboratory samples in 1-gal. cans and close covers tightly.

Moisture and Volatile Matter

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

Scope. Applicable to tung fruit, hulled tung fruit, hulls, and mixture of the last two materials.

A. Apparatus and Supplies

1. Forced draft oven, A.O.C.S. *Specification* H 1-39.
2. Riffle, convenient size. One with eight 1¾-in. chutes is satisfactory. Three metal boxes are necessary, *ca.* 2 in. high, and of such dimensions that the riffle may stand and discharge into these boxes.
3. Sieve, 6-mesh, diameter 12 to 15 in.
4. Wiley sample grinding mill with ¼-in. 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 in. (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.
8. Ground tung hulls (available at some tung mills during processing season).

B. Removal of Foreign Matter

TUNG FRUIT:

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

TUNG FRUIT:

1. Grind whole tung fruit picked at random from the gross sample through the Wiley mill using a ¼-in. screen. If oil is to be determined on the whole fruit, grind 200-250 of the tung fruit. If moisture and volatile matter only is 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 lb. 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.

HULLED TUNG FRUIT:

1. Grind 1-gal. sample of hulled tung fruit through the Wiley mill using a ¼-in. screen.
2. Mix ground hulled fruit and riffle three times by pouring a thin stream of the ground material evenly over the riffle to yield two subsamples of about 150 g. each and place in air-tight containers.

D. Procedure

1. Weigh duplicate 5-g. samples of the Wiley-ground tung fruit or hulled tung fruit and ground hulls 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

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

Oil

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

Scope. Applicable to hulled tung fruit.

A. Apparatus and Supplies

1. Bauer Mill No. 148 with plates No. 6912 so adjusted as to produce a fine meal.
2. Wiley sample grinding mill with ¼-in. 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. Velocity Mixer (A. S. Aloe Co., St. Louis, Mo.).
4. Butt type extraction apparatus, assembled as indicated in the illustration, A.O.C.S. Method Aa 4-38.
5. Filter paper, S&S No. 597, Reeve Angel No. 211, Whatman No. 2 or equivalent, 150 mm.
6. Absorbent cotton, free of petroleum ether extract.
7. Air-tight sample containers for holding ground samples.
8. Forced draft oven, A.O.C.S. Specification H 1-39.
9. Ground tung hulls (available at some tung mills during processing season).

B. Reagents

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

C. Preparation of Sample

1. Use a 150-g. portion of Wiley-ground sample prepared as directed in Ad 2-52 C (hulled tung fruit).
2. Weigh portion of Wiley-ground sample, add an equal weight of ground hulls, and mix thoroughly.
3. Grind mixture of ground hulled tung fruit and hulls through the Bauer mill with plates set about 0.008 in. apart. Mix thoroughly and riffle twice to yield a subsample of about 75 g. and mix in a Henry Velocity Mixer operated at full speed for one minute.
4. Grind a portion of the ground hulls through the Bauer mill with plates set about 0.008 in. apart and place in an air-tight container.

D. Procedure

1. Weigh accurately duplicate 5-g. 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 the a) Wiley-ground hulled fruit, b) ground hulls, c) Bauer-ground mixture of hulled fruit and hulls, and d) Bauer-ground hulls as described in A.O.C.S. Tentative Method Ad 2-48, Section D.

E. Blank Determination

1. Determine petroleum ether extractable material on duplicate 5-g. samples of the Bauer-ground hulls using the procedure as described under D.
2. Determine moisture in Bauer-ground hulls as described in Ad 2-48, D.

F. Calculation

Oil in hulled fruit, % =

$$\left[\left(2 \times \frac{A \times (100 - B)}{D \times (100 - C)}\right) - \frac{E \times (100 - F)}{D' \times (100 - G)}\right] \times 100$$

A = Grams extract from Bauer ground hulled fruit and hulls.

B = Percentage of moisture in mixture Wiley-ground hulled fruit and hulls (Section D, paragraph 5).

C = Percentage of moisture in Bauer-ground mixture of hulled fruit and hulls (Section D, paragraph 5).

D = Weight of Wiley-Bauer ground sample.

D' = Weight of Bauer-ground hull sample (Section E, paragraph 1).

E = Grams extract from Bauer-ground hulls.

F = Moisture in ground hulls.

G = Moisture in Bauer ground hulls.

The methods for the determination of moisture (Ad 2-48), the determination of oil in whole tung fruit (Ad 3-48), the physical analysis of tung fruit (Ad 4-48), and the determination of oil in tung kernels (Ad 5-48) have proven adequate and should be advanced to official status.

Recommendations

1. That method Ad 1-48 be revised to include sampling of hulled tung fruit and be continued as tentative.

2. That method Ad 2-48 be revised to include the determination of moisture in hulled tung fruit, made official, and designated as Ad 2-52.

3. That method Ad 3-48 for the determination of oil in whole tung fruit be made official and designated as Ad 3-52.

4. That method Ad 4-48 for the physical analysis of tung fruit be made official and designated as Ad 4-52.

5. That method Ad 5-48 for the determination of oil in tung kernels be made official and designated as Ad 5-52.

6. That the proposed method described in this report for the analysis of hulled tung fruit be adopted as tentative and designated as Ad 6-52.

7. That the subcommittee be continued for further collaborative work on the tentative methods for the analysis of tung fruit and study of substitution of vermiculite for tung hulls in the method for determining oil in hulled tung fruit.

R. S. McKINNEY, chairman
G. WORTHEN AGEE
G. F. BAILEY
C. R. CAMPBELL
G. CONNER HENRY
P. L. PHILLIPS

Analysis of Castor Beans and Pomace

As a result of a request of the Production and Marketing Administration of the U. S. Department of Agriculture to the president of the Society for standard methods for the analysis of castor beans and pomace a subcommittee of the Seed and Meal Analysis Committee was appointed. It was requested to develop and recommend adoption of methods for the determination of moisture, oil, and free fatty acids in castor beans and moisture and oil in castor pomace.

The present methods of the A.O.C.S. for the analysis of vegetable oil source materials and the results of the investigations of Demint *et al.*[1] were used as a basis for developing specifications of procedures for collaborative study. They were tested by the analysis of a sample of castor beans and a sample of castor pomace, obtained from the Production and Marketing Administration and the Baker Castor Oil Company, respectively. The large sample lots were mixed and then subdivided by use of a Boerner sample divider in an attempt to provide each member of the subcommittee identical samples. The results of the collaborative tests are tabulated in Table I.

TABLE I

Collaborative Analysis of Castor Beans and Pomace

Sample and laboratory no.	Original moisture	Oil content		Free fatty acid in oil
		Original moisture basis	H_2O-free basis	
Castor beans	%	%	%	%
1	5.45	47.98	50.74	1.0
2	5.46	48.17	50.95	1.1
3	5.30	49.50	52.27	0.6
4	4.50	47.90	50.16	0.80
5	5.55	47.79	50.60	0.84
6	5.10	48.95	51.58	0.90
7	5.20	47.58	50.19	0.60
8	5.51	47.21	49.96	0.76
9	5.47	50.12	53.02	0.70
10	5.00	47.75	50.26	1.0
11	5.46	46.65	49.34	0.68
Average	5.27	48.14	50.82	0.82
Castor pomace				
1	8.78	2.35	2.58	
2	9.15	2.47	2.72	
3	8.71	2.48	2.72	
4	8.90	2.00	2.20	
5	9.03	2.30	2.58	
6	8.94	2.48	2.72	
7	9.10	2.30	2.53	
8	9.11	2.25	2.48	
9	9.00	2.10	2.31	
10	8.55	2.25	2.46	
11	9.12	3.38	3.72	
Average	8.94	2.40	2.64	

In outlining the collaborative test procedures attention was given to the specification of apparatus and solutions mentioned in the A.O.C.S. methods for the analysis of vegetable oil source materials. Carbon tetrachloride was chosen as the extraction solvent for the determination of oil on the recommendation of those experienced in the analysis of castor beans. Petroleum solvents are not miscible with castor oil in all proportions. Methyl alcohol was found best for the extraction of castor oil from the beans[1] for the determination of free fatty acids. The methyl alcohol extracts are less viscous, and, in removing the solvent, the nonoil material collects on the sides of the vessel and a clear oil sample for analysis for free fatty acids can be obtained.

Comments of members of the subcommittee were considered in drafting the final statement of five methods. The full text of these methods are as follows:

Moisture and Volatile Matter

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

Scope. Applicable to castor beans.

A. Apparatus

1. Forced draft oven, A.O.C.S. Specification H 1-39.
2. Metal moisture dishes $3\frac{3}{4}$ x 1 in. with tight fitting slip-over covers.
3. Desiccator containing an efficient desiccant. Calcium chloride is not satisfactory. (See A.O.C.S. Specification H 9-45.)
4. Sample riffle, the riffle should have eight $1\frac{3}{4}$-in. slots. Three metal boxes are necessary, *ca.* 2 in. high and of such dimensions that the riffle may stand and discharge into these boxes.
5. Screen, 10 in. in diameter having round hole perforations $\frac{10}{64}$ of an inch in diameter.
6. Air-tight sample containers of convenient size.

B. Preparation of Sample

1. Re-examine the sample for foreign matter. Place a 300-g. portion of the sample on the 10-in. screen and gently shake back and forth to remove the fine material passing through the $\frac{10}{64}$ of an inch in diameter holes. Hand-pick the sample for free and attached whole capsules (hull), detaching the latter, and capsule fragments of mature beans, green or immature capsules, and capsule segments both filled and unfilled, unfilled beans (usually referred to as "poppers"), stems, rocks, etc. Shells (seed coats) remaining on the screen and cracked and broken beans are considered part of the sample for analysis. The foreign matter is considered as the total of the material passing the screen and removed by hand-picking as described above. It is weighed and reported as a percentage of the original sample.
2. Reduce the cleaned sample through the riffle to a size sufficient for the number of tests that will be required.

C. Procedure

1. Weigh 40- to 50-g. samples of castor beans into tared moisture dishes. Slip the covers on the bottoms of the dishes and dry in a forced draft oven at 130° ± 3°C. for 4 hours.
2. Remove from the oven, replace covers immediately, cool to room temperature in a desiccator, and weigh. Calculate moisture as directed in D.

D. Calculation

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

Caution. Castor beans contain an alkaloid, ricinine. They also contain a proteose to which some individuals are allergic. It is strongly recommended that workers wear rubber gloves when preparing and handling analytical samples and that they avoid inhaling any of the dust arising from such samples by working near an air exhaust or in a well ventilated laboratory hood.

Oil

Definition. This method determines the substances extracted by carbon tetrachloride under the conditions of the test.

Scope. Applicable to castor beans.

A. Apparatus

1. Butt type extraction apparatus, assembled as indicated in the illustration (A.O.C.S. Official Method Aa 4-38).
2. Filter paper, S&S No. 597, Reeve Angel No. 211, Whatman No. 2, or equivalent, 150 mm.
3. Absorbent cotton, free of carbon tetrachloride extract.
4. Porcelain mortar, *ca.* 4 in. i.d. with unglazed inside surface, and pestle.
5. Universal food chopper No. 1 with 12-tooth blade (Landers, Frary, and Clark, New Britain, Conn.).

B. Reagents

1. Carbon tetrachloride, reagent grade, free of nonvolatile residue.
2. Sand, 60- to 80-mesh.

C. Procedure

1. Place *ca.* 50 g. of castor beans from the original sample in a convenient drying dish and dry at 130°C. for not more than 20 minutes in a forced draft oven.
2. Cool the sample to room temperature and then grind through the food chopper, feeding the beans slowly to prevent expressing of any oil.
3. Mix the ground sample thoroughly by rolling on a glazed paper or gently rolling in a large glass jar.
4. Weigh accurately, duplicate samples of 5 g. each of the ground sample onto a tared filter paper so that the paper will absorb any oil on the surface of the ground beans. Fold the paper containing the sample and enclose in a second filter paper folded in such a fashion as to prevent escape of the ground beans. (See illustration in A.O.C.S.

---

[1] Demint, R. J., Cucullu, A. F., and Hoffpauir, C. L., J. Am. Oil Chem. Soc. (in press).

Official Method Aa 4-38.) 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 on the sample.

5. Extract the ground sample with carbon tetrachloride as directed in A.O.C.S. Official Method Aa 4-38, Section D, paragraphs 2 to 5, inclusive, except that hot plates are used as a source of heat and the extraction is halted after 2 hours.
6. Remove the thimble from the Butt tube, allow the carbon tetrachloride to evaporate from the filter paper and sample at room temperature, and then carefully transfer the sample to the mortar so that there will be no loss. Add *ca.* 0.5 g. of 60- to 80-mesh sand and regrind the sample in the mortar with at least 200 vigorous strokes of the pestle during a period of *ca.* 2 minutes.
7. Return the ground sample to the same filter paper and continue the extraction as before for another 2 hours.
8. Discontinue the extraction, cool, and disconnect the extraction flask. Evaporate the carbon tetrachloride on a steam bath for an hour after boiling ceases, then place the flask in a forced draft oven at 101°C. for 2 additional hours. Cool to room temperature, carefully remove any foreign material from the outside of the flask, and weigh. Repeat heating in the forced draft oven for 30-minute intervals until constant weight is obtained.
9. Determine the moisture content of the partially dried and ground sample as follows:
   a. Weigh 5 g. into a tared A.O.C.S. moisture dish.
   b. Slip the cover on the bottom of the dish and place in a forced draft oven. Dry at 130° for 2 hours.
   c. Remove the dish from the oven and cover immediately. Cool in a desiccator containing an efficient desiccant for 30 minutes and weigh.

D. Calculations

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

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

The percentage oil is calculated to any desired moisture basis by use of the following formula:

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

F = % oil determined in ground sample.

Free Fatty Acids

Definition. This method determines the free fatty acids in the oil removed from the seed by methyl alcohol extraction at room temperature.

Scope. Applicable to castor beans.

A. Apparatus

1. Universal food chopper No. 1 with 12-tooth blade (Landers, Frary, and Clark, New Britain, Conn.).
2. Oil sample bottles or flasks of convenient size.
3. Filtration apparatus of convenient size, consisting of either glass funnel and support or Buchner funnel and suction flask equipped with S&S No. 597, Reeve Angel No. 211, Whatman No. 2, or equivalent filter paper of appropriate size.

B. Reagents

1. Methyl alcohol—reagent grade—having no residual acidity or alkalinity.
2. Ethyl alcohol, 95% (U.S.S.D. Formulas 30 and 3A are permitted), or isopropyl alcohol, 99%. The alcohol must be neutralized with NaOH soln. to a faint pink color before use.
3. Phenolphthalein indicator soln., 1% in 95% alcohol.
4. Sodium hydroxide, 0.25 N, accurately standardized.

C. Procedure

1. Remove the foreign matter and reduce the sample to a convenient size as directed in A.O.C.S. Tentative Method Ae 2-52, Section B, paragraphs 1 and 2.
2. Grind *ca.* 150 g. of beans through the food chopper. Mix the ground sample thoroughly.
3. Suspend *ca.* 20 g. of the ground sample in 100 ml. of methyl alcohol at room temperature with occasional agitation for 30 minutes.
4. Filter by means of either a Buchner funnel or fluted paper. Remove the solvent by heating on a steam bath applying a vacuum to the flask during the entire period of heating to remove the last traces of solvent.
5. Weigh 7.05 g. of extracted and solvent-free oil into the oil-sample bottle or flask being careful to avoid transferring the non-oil material which adheres to the bottom of the original flask. Add 30 ml. of neutral alcohol and 1 ml. of indicator. Titrate with 0.25 N NaOH, shaking vigorously until a faint pink color is obtained which will persist for at least 1 minute.

D. Calculation

Free fatty acids, calculated as oleic, % = ml. of 0.25 N alkali used.

Moisture and Volatile Matter

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

Scope. Applicable to castor pomace.

A. Apparatus

1. Aluminum moisture dishes, 30-gauge, 2 x ¾ in. (*ca.* 50 x 19 mm.) with tight fitting slip-over covers.
2. Forced draft oven, A.O.C.S. Specification H 1-39.
3. Desiccator containing an efficient desiccant. Calcium chloride is not satisfactory. See A.O.C.S. Specification H 9-45.
4. Jones sampler, riffle type, 6 x 6 in.
5. Air-tight sample container, capacity *ca.* 100 g.

B. Preparation of Sample

1. Reduce the original 1,000-g. sample through the riffle to *ca.* 100 g. and immediately place in air-tight container. Weigh moisture sample immediately.

C. Procedure

1. Weigh *ca.* 5 g. of the sample into tared moisture dish.
2. Place the dish in the forced draft oven and dry at 130° ± 3°C. for 2 hours.
3. Remove from the oven, cover immediately, cool in a desiccator to room temperature, and weigh.

D. Calculation

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

Oil

Definition. This method determines the substances extracted by carbon tetrachloride under the conditions of the test.

Scope. Applicable to castor pomace.

A. Apparatus

1. Butt type extraction apparatus, assembled as indicated in A.O.C.S. Official Method Ba 3-38.
2. Filter paper, S&S No. 597, Whatman No. 2, Reeve-Angel No. 211, or equivalent, 150 mm.
3. Absorbent cotton, free of carbon tetrachloride extract.
4. Sieve, U. S. No. 20.
5. Laboratory mill suitable for grinding the samples to a maximum particle size of U. S. No. 20 sieve.

B. Reagents

1. Carbon tetrachloride, reagent grade.

C. Preparation of Sample

1. Grind the 100-g. portion from the method for moisture and volatiles in castor pomace through the laboratory mill. Return immediately to an air-tight container. Oil and ground moisture are determined on this portion.

D. Procedure

1. Weigh *ca.* 5 g. of the ground sample onto a filter paper and enclose in a second filter paper folded in such a fashion as to prevent escape of pomace. (See illustration in A.O.C.S. Official Method Ba 3-38). 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 on the sample.

2. Place wrapped sample in the Butt extraction tube and assemble the apparatus as shown in the illustration in A.O.C.S. Official Method Ba 3-38. Put *ca.* 25 ml. of carbon tetrachloride into the tared extraction flask before attaching to the tube.
3. Heat on an electric hot plate at such a rate that the solvent will drop from the condenser on the center of the thimble at the rate of at least 150 drops per minute.
4. Keep the volume of solvent fairly constant by adding enough to make up for any that may be lost due to evaporation. Continue the extraction for 4 hours.
5. Cool and disconnect the extraction flask. Evaporate the carbon tetrachloride on a steam bath for an hour after boiling ceases, and then place the flask in a forced draft oven at 101°C. for 2 additional hours. Cool to room temperature, carefully remove any foreign material from the outside of the flask, and weigh. Repeat heating in the forced draft oven for 30-minute intervals until constant weight is obtained.
6. Determine the moisture in the ground sample as directed in the method for moisture and volatiles in castor pomace.

E. Calculation

$$\text{Oil, \%} = \frac{\text{Weight of oil} \times 100}{\text{Weight of sample}}$$

The percentage oil may be calculated to any desired moisture basis with the following formula:

$$\text{Oil, desired moisture basis, \%} = \frac{\text{F}\,(100 - \text{\% moisture desired})}{100 - \text{\% moisture in sample analyzed}}$$

F = % oil in sample analyzed.

The test results do not show as good agreement as desired. However with the exception of two laboratories, no previous experience had been had in the analysis of castor beans and pomace. The variations in oil contents reported may be due to variations in grinding the beans or failure to remove all of the solvent. With more experience both of the difficulties should be overcome, and results checking within reasonable tolerances should be readily obtained.

As the Production and Marketing Administration is in need of the methods in handling the 1952 crop of castor beans, it is unanimously recommended that the methods outlined above be adopted as tentative and that the subcommittee be continued.

T. H. HOPPER, chairman
W. F. BEEDLE
D. S. BOLLEY
EDITH A. CHRISTENSEN
W. T. COLEMAN
G. CONNER HENRY
C. L. MANNING
J. R. MAYS JR.
R. C. POPE
V. B. SHELBURNE
C. R. SOUTHWELL

Specifications for Standardization of Solutions

THE Uniform Methods Committee recommended and the Society approved, at the meeting in Houston, Texas, April 1, 1952, that the stock of Official Standard Sodium Carbonate be discarded and its distribution be discontinued. This acidimetry standard is little used since the National Bureau of Standards can furnish for the purpose acid potassium phthalate (B.S. No. 84c). The committee was directed, in collaboration with the Fat Analysis Committee, to prepare specifications of procedures for the standardization of the most used standard solutions. They are as follows:

Standard Sodium Hydroxide Solution

A. Apparatus

1. Erlenmeyer flasks, 300-ml. capacity.
2. Buret, accurately calibrated to meet National Bureau of Standards specifications. Because alkali will dissolve glass, to avoid volumetric errors it should not be stored in calibrated apparatus. Burets used continuously should be recalibrated periodically.
3. Absorption tower or drying tube of adequate capacity packed with an efficient absorbent for carbon dioxide, such as Ascarite or soda lime.

B. Reagents

1. Carbon dioxide-free distilled water, prepared by one of the following methods: 1. boil water for 20 minutes and cool with absorption tower protection against CO_2 absorption; 2. bubble a vigorous stream of air, freed from CO_2 by passing through a tower filled with Ascarite or soda lime, through water for 12 hours.
2. Sodium hydroxide (1 + 1). To one part by weight of NaOH (containing less than 5% Na_2CO_3) in an alkali resistant flask or bottle add one part of H_2O. Swirl until solution is complete. Close the container tightly with a rubber stopper. Set aside until Na_2CO_3 has settled, leaving perfectly clear supernatant liquid.
3. Acid potassium phthalate, National Bureau of Standards standard sample for acidimetry. Crush sample to a fineness of approximately 100-mesh and dry for 2 hours at 120°C. Allow to cool in an efficient desiccator.
4. Ethyl alcohol, 95% by volume (U.S.S.D. Formulas 30 and 3A are permitted).
5. Phenolphthalein indicator. Dissolve 1 g. of phenolphthalein in 100 ml. of alcohol.

C. Procedure

1. The following table gives the approximate quantities of NaOH solution (1 + 1) necessary to make 10 liters of standard solution of the indicated normality.

Approximate normality	Sodium hydroxide solution to be diluted to 10 liters	
	milliliters	*grams*
0.1 N	54	82
0.25 N	135	205
0.5 N	270	410
1.0 N	540	820

2. Add the required quantity of NaOH solution (1 + 1) to enough CO_2-free distilled water to give a total volume of 10 liters and mix well. Protect the solution from CO_2 by stoppering tightly or by means of absorption tube or tower.
3. Weigh accurately sufficient dried $KHC_8H_4O_4$ to require about 40 ml. of the sodium hydroxide solution to be standardized and transfer it to a 300-ml. flask that has been swept free from CO_2. Add 50 ml. of cool CO_2-free H_2O. Stopper the flask and swirl gently until the $KHC_8H_4O_4$ is dissolved.
4. When the sample is in solution, add 3 drops of phenolphthalein indicator and titrate to the first persistent faint pink color with the solution to be standardized taking precautions to exclude CO_2.
5. Determine the quantity of sodium hydroxide solution required to produce the same pink phenolphthalein end point in another flask containing the indicator and the same volume of CO_2-free water. Subtract the amount required from that used in the first titration and calculate the normality of the standard solution by the following equation:

$$\text{Normality} = \frac{\text{Grams of } KHC_8H_4O_4}{\text{Ml. of NaOH} \times 0.20422}$$

Notes

1. When the standard sodium hydroxide is prepared as directed, it is essentially free of carbon dioxide. It is therefore possible to correct for the amount of alkali required to produce the end point with phenolphthalein as described above. The resulting solution is expected to have the true normality with respect to the hydrogen ion concentration.

Standard Sulfuric Acid Solution

A. Apparatus

1. Erlenmeyer flasks, 300-ml. capacity.
2. Buret, accurately calibrated to meet National Bureau of Standards specifications.
3. Absorption tower or drying tube of adequate capacity packed with an efficient absorbent for carbon dioxide, such as Ascarite or soda lime.

B. Reagents

1. Carbon dioxide-free distilled water, prepared by one of the following methods: 1. boil water for 20 minutes and cool with absorption tower protection against CO_2 absorption; 2. bubble a vigorous stream of air, freed from CO_2 by passing through an absorption tower filled with Ascarite or soda lime, through water for 12 hours.
2. Sulfuric acid, reagent quality, approximately 95% H_2SO_4.
3. Sodium hydroxide solution, prepared and standardized as directed by A.O.C.S. Specification H 12-52.
4. Ethyl alcohol, 95% by volume (U.S.S.D. Formulas 30 and 3A are permitted).
5. Phenolphthalein indicator. Dissolve 1 g. of phenolphthalein in 100 ml. of alcohol.
6. Methyl red indicator. Dissolve 100 mg. of methyl red in 60 ml. of alcohol and dilute with distilled water to 100 ml.
7. Methyl orange indicator. Dissolve 100 mg. of methyl orange in 100 ml. of distilled water.

C. Procedure

1. The following table gives the approximate quantities of reagent grade H_2SO_4 (95%) necessary to make 10 liters of standard solution of the indicated normality.

Approximate normality	H_2SO_4 to be diluted to 10 liters
	milliliters
0.1 N	28
0.25 N	70
0.5 N	140
1.0 N	280

2. Add required quantity of H_2SO_4 to sufficient CO_2-free distilled water to give a total volume of 10 liters and mix well.
3. Measure accurately, by means of a buret, 40 ml. of the H_2SO_4 solution to be standardized into a 300-ml. flask. Add 3 drops of phenolphthalein indicator and titrate to the first persistent faint pink color with standardized sodium hydroxide solution of about the same concentration as the acid being standardized. Calculate the normality of the acid solution by the following formula:

$$\text{Normality} = \frac{\text{Ml. of standard alkali} \times \text{normality of alkali}}{\text{Ml. of } H_2SO_4}$$

4. If the standard acid is to be used with methyl orange or methyl red as indicators, determine the correction for the volume of acid required to pass from the end point of phenolphthalein to that of methyl orange or methyl red. Add 3 drops of phenolphthalein indicator to 100 ml. of CO_2-free water and then add sufficient sodium hydroxide solution to give the faint pink end point with phenolphthalein. Disregard this quantity of sodium hydroxide solution. Add 3 drops of methyl orange or methyl red indicator and sufficient standard acid to give the red acid color of the indicator. Then titrate back with standard sodium hydroxide of the same strength as the standard acid to the methyl orange or methyl red end point, that is to the disappearance of orange or pink color. Subtract the ml. of sodium hydroxide from the ml. of acid to obtain the correction for the volume of acid required to pass from the end point of phenolphthalein to that of methyl orange, or methyl red. This correction should be added to the ml. of acid used in the standardization for calculating the normality for use with methyl orange or methyl red indicator.

Standard Hydrochloric Acid Solution

A. Apparatus

1. Erlenmeyer flasks, 300-ml. capacity.
2. Buret, accurately calibrated to meet National Bureau of Standards specifications.
3. Absorption tower or drying tube of adequate capacity packed with an efficient absorbent for carbon dioxide, such as Ascarite or soda lime.

B. Reagents

1. Carbon dioxide-free distilled water, prepared by one of the following methods: 1. boil water for 20 minutes and cool with absorption tower protection against CO_2 absorption; 2. bubble a vigorous stream of air, freed from CO_2 by passing through a tower filled with Ascarite or soda lime, through water for 12 hours.
2. Hydrochloric acid, reagent quality, 35-37% HCl.
3. Sodium hydroxide solution, prepared and standardized as directed in A.O.C.S. Specification H 12-52.
4. Ethyl alcohol, 95% by volume (U.S.S.D. Formulas 30 and 3A are permitted).
5. Phenolphthalein indicator. Dissolve 1 g. of phenolphthalein in 100 ml. of alcohol.
6. Methyl red indicator. Dissolve 100 mg. of methyl red in 60 ml. of alcohol and dilute with distilled water to 100 ml.
7. Methyl orange indicator. Dissolve 100 mg. of methyl orange in 100 ml. of distilled water.

C. Procedure

1. The following table gives the approximate quantities of reagent grade HCl (35-37%) necessary to make 10 liters of standard solution of the indicated normality.

Approximate normality	HCl to be diluted to 10 liters
	milliliters
0.1 N	89
0.25 N	222
0.5 N	445
1.0 N	890

2. Add the required quantity of HCl to enough CO_2-free distilled water to give a total volume of 10 liters and mix well.
3. Measure accurately, by means of a buret, 40 ml. of the HCl solution to be standardized into a 300-ml. flask. Add 3 drops of phenolphthalein indicator and titrate to the first persistent faint pink color with standardized sodium hydroxide solution of the same concentration as the acid being standardized. Calculate the normality of the acid solution by the following formula:

$$\text{Normality} = \frac{\text{Ml. of standard alkali} \times \text{normality of alkali}}{\text{Ml. of HCl}}$$

4. If the standard acid is to be used with methyl orange or methyl red as indicators, determine the correction for the volume of acid required to pass from the end point of phenolphthalein to that of methyl orange or methyl red. Add 3 drops of phenolphthalein indicator to 100 ml. of CO_2-free water and then add sufficient sodium hydroxide solution to give the faint pink end point with phenolphthalein. Disregard this quantity of sodium hydroxide solution. Add 3 drops of methyl orange or methyl red indicator and sufficient standard acid to give the red acid color of the indicator used. Then titrate back with standard sodium hydroxide of the same strength as the standard acid to the methyl orange or methyl red end point taken, that is to the disappearance of orange or pink color. Subtract the ml. of sodium hydroxide from the ml. of acid to obtain the correction for the volume of acid required to pass from the end point of phenolphthalein to that of methyl orange or methyl red. This correction should be added to the ml. of acid used in the standardization for calculating the normality for use with methyl orange or methyl red indicator.

Standard Alcoholic Potassium Hydroxide

A. Apparatus

1. Erlenmeyer flasks, 300-ml. capacity.
2. Buret, accurately calibrated to meet National Bureau of Standards specifications. Because alkali will dissolve glass, to avoid volumetric errors it should not be stored in calibrated glass apparatus. Burets used continuously should be recalibrated periodically.
3. Absorption tower or drying tube of adequate capacity packed with an efficient absorbent for carbon dioxide, such as Ascarite or soda lime.

B. Reagents

1. Potassium hydroxide, reagent grade.
2. Ethyl alcohol, 95% by volume (U.S.S.D. Formulas 30 and 3A are permitted).
3. Acid potassium phthalate, National Bureau of Standards standard sample for acidimetry. Crush sample to a fineness of approximately 100-mesh and dry for 2 hours at 120°C. Allow to cool in an efficient desiccator.

4. Phenolphthalein indicator. Dissolve 1 g. of phenolphthalein in 100 ml. of alcohol.

C. Procedure

1. Dissolve the appropriate amount of KOH (35 g. for 0.5 N; 14 g. for 0.2 N) in 20 ml. of distilled water and dilute to a liter with alcohol in an alkali resistant bottle. Mix well and allow to stand overnight. Decant the clear supernatant solution into a brown glass bottle with a rubber stopper without exposure to carbon dioxide.
2. Weigh accurately sufficient dried $KHC_8H_4O_4$ to require about 40 ml. of the potassium hydroxide solution to be standardized and transfer it to a 300-ml. flask that has been swept free of CO_2. Add 50 ml. of cool CO_2-free water. Stopper the flask and swirl gently until the $KHC_8H_4O_4$ is dissolved.
3. When the sample is in solution, add 3 drops of phenolphthalein indicator and titrate to the first persistent faint pink color with the solution to be standardized, taking precautions to exclude CO_2.
4. Determine the quantity of the potassium hydroxide solution required to produce the same pink phenolphthalein end point in another flask containing the indicator and the same volume of CO_2-free water. Subtract the amount from that used in the first titration.
5. Calculate the normality of the standard solution by the following equation:

$$\text{Normality} = \frac{\text{Grams of } KHC_8H_4O_4}{\text{Ml. of KOH} \times 0.20422}$$

Recommendations

THE reports of the subcommittees and the specifications for the preparation of the most used standard solutions have been reviewed by the Seed and Meal Analysis Committee. Its recommendations are:

1. That the recommendations of the Subcommittee on the Analysis of Tung Fruit and Meal be adopted. They are
   a) That method Ad 1-48 for sampling tung fruit be revised to include sampling of hulled tung fruit and be continued tentative.
   b) That method Ad 2-48 for the determination of moisture in tung fruit be revised to include the determination of moisture in hulled tung fruit, made official, and designated as Ad 2-52.
   c) That method Ad 3-48 for the determination of oil in whole tung fruit be made official and designated as Ad 3-52.
   d) That method Ad 4-48 for the physical analysis of tung fruit be made official and designated as Ad 4-52.
   e) That method Ad 5-48 for the determination of oil in tung kernels be made official and designated as Ad 5-52.
   f) That the proposed method for the determination of oil in hulled tung fruit be adopted as tentative and designated as Ad 6-52.
2. That the recommendations of the Subcommittee for the Analysis of Castor Beans and Pomace for the adoption of methods for the analysis of castor beans and pomace as tentative be accepted. The methods involved are
   a) Determination of moisture and volatile matter in castor beans.
   b) Determination of oil in castor beans.
   c) Determination of free fatty acids in castor beans.
   d) Determination of moisture and volatile matter in castor pomace.
   e) Determination of oil in castor pomace.
3. That the proposed specifications for the preparation of standard solutions for volumetric analysis be adopted. They are those for the standardization of
   a) Sodium hydroxide solution,
   b) Sulfuric acid solution,
   c) Hydrochloric acid solution, and
   d) Alcoholic potassium hydroxide.

These recommendations have received the unanimous approval of the committee with the exception that two members voted against the specifications for the standardization of the acid solutions by use of acid potassium phthalate. They have a strong preference for the use of sodium borate.

T. H. HOPPER, chairman
E. C. AINSLIE
L. R. BROWN
F. R. EARLE
E. B. FREYER
G. CONNER HENRY
J. C. KONEN
R. S. MCKINNEY
D. F. MASKEY
V. C. MEHLENBACHER
T. C. POTTS
T. L. RETTGER
T. C. SMITH

[Received October 29, 1952]

Effects of Heat on Crude Peat Wax

A. SUNDGRÉN and VEIKKO T. RAUHALA, Peat Technical Laboratory,
The State Institute for Technical Research, Helsinki, Finland

IT has been observed that the yield of bitumen extracted from peat depends on the temperature at which the peat has been dried, and Sundgrén (6) has reported that drying at a temperature of only 100-105° reduces the yield. In this paper bitumen refers to the material extracted from peat with a hot neutral solvent under ordinary pressure, which contains mainly waxes and resins. Crude wax is the part of bitumen left when the crude resins are separated from it.

Stadnikoff and coworkers (4, 5) reported that the solubility of peat bitumen in certain solvents decreases when the material is heated or stored for some time. They expressed the opinion that this phenomenon is a polymerization process and mentioned especially that the crude resin in peat bitumen is easily polymerized. Zetzsche (8) calls the part of peat bitumen which is difficult to dissolve "Polymer-Bitumen." Cawley and Carlile (1) use the term "asphalt" for the part of peat bitumen insoluble in light petroleum (B. Pt. 80-100°), and the term crude wax for the product extracted directly from peat.

The workers in the present research have reported (7) that the crude fatty acids separated from the crude wax of peat bitumen behave, when heated, in the same way as the bitumen.

As far as is known, none of the investigators mentioned has studied closely the formation of this insoluble or difficultly soluble part. The question is of interest not only as a scientific problem but also as an important factor in the production of these materials from peat on a commercial scale. The Peat