permit the extraction of cottonseed of variable free fatty acid content and to produce a product of controlled uniformity.

Acknowledgment

This work was carried out with funds from the Cotton Research Committee of Texas and the Texas Engineering Experiment Station.

REFERENCES

1. Harris, W. D., Bishop, F. F., Lyman, C. M., Helpert, R., J.A.O.C., *24*, 370-375 (1947).
2. Harris, W. D., Chemurgic Digest, *7*, 9-12 (1948).
3. Wesson, Oil and Fat Ind., *3*, 307 (1926).
4. Lyman, C. M., Holland, B. R., Hale, F., Ind. and Eng. Chem., Anal. Ed., *15*, 489 (1943).
5. A.O.C.S., Methods, Ca-9a-41.
6. A.O.C.S., Methods, Cc-8a-46.
7. National Cottonseed Products Association, Rules, 1947, Rule 137.

Rapid Method of Copra Analysis and Its Application to the Various Oil Seeds

ARTHUR F. PINTO and JOSEPH D. ENAS, El Dorado Oil Works, Oakland, California

THE analysis of copra presents a number of difficulties which must be overcome if dependable results are to be obtained. Most copra is highly variable in quality, such that in a sample sack, representing hundreds of tons, it would be difficult to find two pieces exactly alike. Spoilage, which causes the free fatty acids and color of the oil to be highest where the damage is most pronounced, sets in on the inner surface and progressively works outwardly toward the side which was attached to the shell. To make matters worse the fines, which are highest in free fatty acids and lowest in oil content (1, 2), must be uniformly incorporated in the sample in order that results may be consistent and reliable under any method of analysis. Pieces of coconut shell in the sample too must be reduced to small particles and likewise uniformly distributed.

Because of its high oil content copra cannot be ground by the ordinary laboratory mills without dislodging oil or otherwise rendering the sample unsuitable for all determinations. Nails, bolts, bottle caps, gravel, etc., are also a detriment for even under careful scrutiny these things occasionally slip through, further rendering laboratory mills unfit because they were not designed to handle such a mixture.

Considering the type of material and the tonnage it represents, the selected representative sample to be extracted must be large and its extraction complete, or very nearly so, in order that the results of the analysis may be reproducible and represent the quality of the lot as a whole. Extraction too must be cold and rapid to prevent any possible change in the free fatty acids and color of the oil during an otherwise hot and prolonged extraction. Rapidity, simplicity, and a minimum of manipulation are also desirable for many reasons, such as accuracy, time saving, and the prompt availability of results when they are quickly needed.

All the above problems have been successfully solved by the new method, made possible by the development of new equipment and some improvements on other heretofore in use. Among other things the method employs a specially equipped blender, or mixer for the trituration of the material and extraction of the oil by means of a suitable solvent, and the removal of the solvent from the oil by the application of heat and vacuum simultaneously. Further details will be given later in conjunction with the various steps of the procedure. The use of a mixer for the extraction of oil from oil seeds in the presence of a solvent is not new. We began using the Whiz-Mix blender for this purpose in 1941 and in the intervening time various publications have reported the use of the Waring Blendor in oil and fat extraction (3, 4, 5, 6).

Although the extraction of samples, large or small, is rapid by the new method, total extraction is more nearly complete in 10 minutes than it is with one of the best extractors, now in use, in an eight-hour period. We have run numerous samples by the *Goldfisch Method* and the *Rapid Method* for comparison and in all cases extraction was best by the latter method in spite of the fact that samples 15 times larger were used and time of extraction only 10 minutes instead of eight hours by the former method. Results are shown in Table I.

Comparison with the Soxhlet, Smalley, and Butt extractors was not obtained because these extractors are known to be slower than the Goldfisch extractor. In a 21-hour extraction period however the Soxhlet and the Butt compared favorably with the latter extractor (7).

The better results obtained by the new method of course are based on the fact that no channeling is possible on account of the vigorous agitation and the copra meal becomes so fine by the trituration that, after filtering, washing with solvent, and drying, it can pass through a No. 100 sieve.

The rapidity of the method makes it possible for a chemist, after the copra is chopped, to turn out the complete analysis of a sample, comprising moisture, oil content, color, and free fatty acid determinations in two hours, and if there are several samples to be run together, the time consumed per sample is reduced to about one hour.

The method also makes possible the extraction of large amounts of oil, such as four-ounce bottles, to be sent to interested parties before actual crushing takes place. The method is likewise suitable for the rapid extraction of expeller cake. Results have been compared with those obtained with the Goldfisch extractor and are shown in Table II.

Since the equipment used is in large part new, a brief description thereof will be presented to familiarize the reader before passing on to the analytical procedure.

The Copra Chopper. This chopper, shown in Figures 1 and 2, is a machine similar to a hammer mill, but instead of hammers it has fixed steel blades, the cutting edges of which have been made extremely

TABLE I

Extraction Efficiency of Rapid Method Compared With Goldfisch Method

Copra lots	Rapid Method	Goldfisch Method				Goldfisch Method		
	Determinations on 75-gm. copra samples, 10-min. extractions, continuous triturations	Determinations on 5-gm. copra samples, 8-hr. extractions, samples re-ground after 4 hrs. extraction				Determinations on residues, 4-hr. extractions, after regrinding with sea sand		
	A	B	C	D	E	F	G	H
					Average oil of B, C, D	Residual meal re-extracted	Samples of residues from A	Combined residues B, C, D
	% Oil	% Oil	% Oil	% Oil	% Oil	Grams	% Oil	% Oil
1	65.98	64.40	64.10	65.00	64.50	4.625	.45	1.98
2	66.54	64.70	63.30	64.80	64.90	4.630	.54	2.16
3	65.83	62.80	65.10	64.20	64.03	4.734	.61	1.64
4	63.78	62.90	63.70	63.10	63.23	4.823	.48	2.11
5	66.38	66.60	65.30	65.80	65.90	4.480	.29	.93
6	66.94	64.00	63.50	64.80	64.10	4.732	.44	2.53
7	66.45	64.70	63.30	63.90	63.96	4.793	.47	2.83
8	67.51	66.00	67.30	66.30	66.53	4.406	.43	1.70

The 5-gm. samples, B, C, and D, of each lot were dried in an electric oven at 102°C. for 1½ hours before extraction, as customary. The weight of their combined residues, shown in column F, is the weight of the combined copra samples before drying minus the volatile matter lost in drying and the weight of the oil extracted. After completely drying, an equal weight of residue from A was taken. Both samples were then reground with sea sand and extracted for 4 hours. Their residual oil is compared in columns H and G, respectively. The first four samples of column A were dried 45 minutes in drying jars under vacuum on the water bath at 100°C., before extraction. The other four were dried in the electric oven at 102°C., for the same length of time. Ethyl ether was the solvent used in all cases. The percentages of residual oil in columns G and H are based on the meal of column F. Since the average residual oil in column G is .46, and the extracted meal is about ⅓ of the weight of the copra from which it is taken, the oil left in the meal is only .15% of the weight of the copra samples. This oil can be completely removed as explained in part 2 of the Analytical Procedure.

hard to stand the rough treatment to which they are subjected. It is equipped with two screens, one having ½" and the other ¼" openings. The chopped copra passing through these screens is much finer than indicated by the size of the screen openings.

Although rocks and pieces of steel are avoided and removed when seen, they do not hurt this sturdy chopper very much when they slip through. Should a nail or a rock go down with the copra, the machine is stopped, the impediment removed, and chopping resumed as before. However best results are obtained when the blades are sharp so they must be sharpened on an emery wheel from time to time. The chopping of a sack of copra (80 to 100 pounds) takes only a few minutes.

Two steam jackets under the inner hopper, below the screen, warm the machine gently during cold weather, thus preventing the surface oil of the copra and fines from freezing to the cold steel and making cleaning and flowing of chopped copra difficult or impossible. However too much heat must be avoided in order not to dislodge any oil.

A similar chopper in conjunction with a shredder has been used by Tompkins (2), and a mechanical or hand rasp has also been suggested (8). Another method for the preparation of samples for extraction of seeds containing over 40% oil, when the extractor is of the Butt type, has been presented by Jamieson (9). Because chopping is rapid the copra does not become hot, and therefore no moisture is lost or oil dislodged during the operation.

The Sample Splitter. The sample splitter, here used, is of the riffles type. It is 10" x 18" and has nine chutes on each side. This large size, which has been obtained from the Fisher Scientific Company,

TABLE II

Extraction Efficiency of *Rapid Method* Compared With *Goldfisch Method*

Expeller samples	Rapid Method	Goldfisch Method
	Determinations on 75-gm. samples of expeller cake, 5-min. extraction	Determinations on 5 gm. samples of expeller cake, 3-hr. extraction
	% Oil	% Oil
1	6.45	6.40
2	7.20	7.20
3	5.63	5.50
4	6.20	6.10
5	6.82	6.80
6	7.40	7.30

The copra expeller cake was pulverized in the blender before weighing out samples. The samples run by the Goldfisch Method were dried 1½ hours at 102°C. in the electric oven before extraction. The samples run by the Rapid Method were not dried.

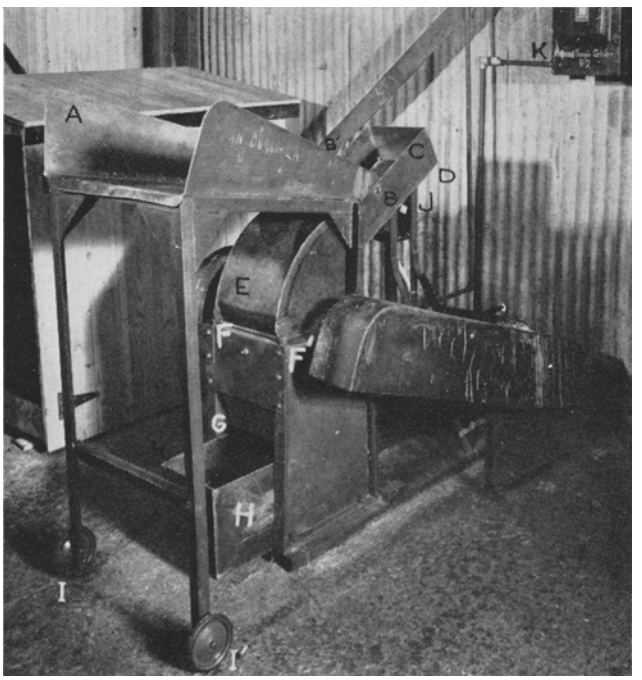

FIG. 1. The copra chopper.

The copra sample is placed on trough A and gradually pushed down into chute C and chopping chamber under hood E. When copra is fine enough to pass through the screen it falls into the receiving drawer H. Trough and chute are pivoted at BB' and hood is pivoted to base at FF'. When chute is lifted at D the wheels II' roll back and chopper holds itself wide open for the changing of screens and cleaning. The front steam jacket is at G, welded to hopper. The rear jacket is not in view. Push button is at J and switch at K.

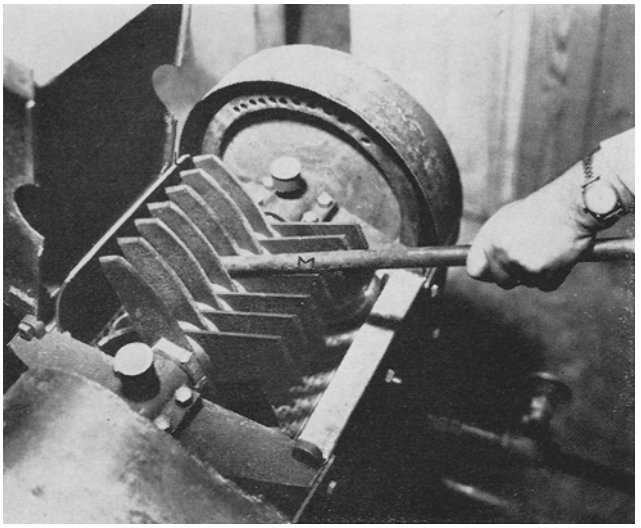

FIG. 2. Chopping chamber and blade assembly.
After stopping motor at J and pulling down switch at K (Fig. 1) the screen L is easily removed by the use of pointed bar M against blade assembly shaft. Cleaning of hopper into receiving drawer is done with long handle brush.
Blades rotate at the speed of 2,100 R.P.M.

Pittsburgh, Pa., is desirable in the handling of large samples. As shown in Figure 3, legs and sides of hopper have been substantially re-enforced for rigidity.

The Waring Blendor. Although it could stand some improvement, the Waring Blendor is a fine machine for laboratory use in the extraction of oil from oil seeds by means of a suitable solvent. It is also useful for the comminution of such seeds for moisture determinations and for pulverizing expeller cake for the determination of moisture and oil content.

The bearing of the container must have no lateral play and as little as possible longitudinal play. The longitudinal play can be corrected by inserting a new washer in the bearing, but when there is lateral play the thing to do is to put on a new bearing. Users of the Waring Blendor should obtain from its manufacturers Bulletin No. 2, Oct. 15, 1946, which contains a description of all replacement parts. From time to time it is advisable to lubricate the bearing with stopcock grease, especially when a bearing is somewhat worn, to insure against leakage.

An important feature of the Waring Blendor is that fires are not likely to occur even though inflammable solvents are used for oil extraction. The only possibility of a fire (not an explosion) is that of an operator allowing the solvent to splash and spill over the top of the container. In such a case the solvent may drain down the outside of the machine and be drawn into the motor. Knowing this however, an operator would be unlikely carelessly to allow any spillage to take place, certainly not enough to cause a fire. When a variable transformer is used in the circuit, the machine is started at a reduced speed and then shifted to high. This prevents the surge from rising too high and possibly splashing over the container. The standard size container is used for both large and small samples.

But the Waring Blendor is not suitable for complete trituration and total oil extraction from oil seeds, such as copra, in the determination of oil content when equipped with its own blades. The blades that come with the machine are too soft, for one thing, and quickly become dull when triturating large samples of copra, which often contain sand, pieces of gravel, and even metals. To overcome this difficulty blades of a special design and construction have been made, and it is with these new blades that total oil extraction on large and small samples is possible. There are on the market today various mixers that would be just as efficient as the Waring Blendor if equipped with the proper blades. A machine with two speeds should be preferable because it would make the use of a variable transformer unnecessary.

The Special Blade Assembly. The special blade assembly, shown in Figure 4, was designed and constructed for rapid comminution of materials, rough treatment, and to maintain the cutting edges sharp for long periods of time in order to insure dependable results. In accord with Hamilton and Gilbert (5) we found the blades supplied with the Waring Blendor to perform rather erratically in maintaining sharp edges, and when it came to the trituration of large samples of copra and complete oil extraction, they all failed with the first sample because they became too dull. Research was undertaken and the new blades developed.

The new blade assembly consists of three blades with six cutting edges (instead of the conventional two, with four cutting edges) and are made of hardened tool steel, so hard that it can not be scratched with a file, yet special and proper annealing prevents them from breaking with normal use. They are easily re-sharpened on a fine grain emery wheel, and each sharpening normally triturates 15 or 20 75-gm. copra samples. Re-sharpening and re-assembling only takes five minutes. They can be re-sharpened many times before needing to be replaced with new ones.

The Surface Cooler. The surface cooler, shown in Figure 5, was developed to prevent vaporization of the solvent in the container and splashing during trituration by stabilizing the surface of the liquid. It consists of a hollow bulb with a tubular neck which serves to carry the cooling water leads and support the cooler in position, clamped to a ringstand post. The bulb, 3″ in diameter and made of copper in this case, can be made in any suitable size to fit the particular container and of any good conductor of heat. The metals are preferable, especially the better conductors, but if copper or brass are used, the cooler should be silver or chrome plated.

Its shape can be varied to fit the particular need for the efficiency of the surface cooler lies mainly in that it cools a liquid where cooling is most effective, namely, its surface. The liquid is first forced up and then down by the vigorous agitation, thus cooling the entire contents of the vessel and preventing their escape through vaporization. The outer ends of the cooler leads are bent down about 95° and are connected to the water supply and drain, respectively, by means of rubber tubing.

In spite of the fact that much heat is generated by the bearing of the container and the vigorous agitation the surface cooler maintains the contents in the container at one or two degrees warmer than the circulating water. On a hot day when the laboratory temperature was 32° and that of the cooling water 25°, ethyl ether in the container during trituration was maintained at 27°C. Since ethyl ether boils at 34.6°C., there is little tendency for its escape into the

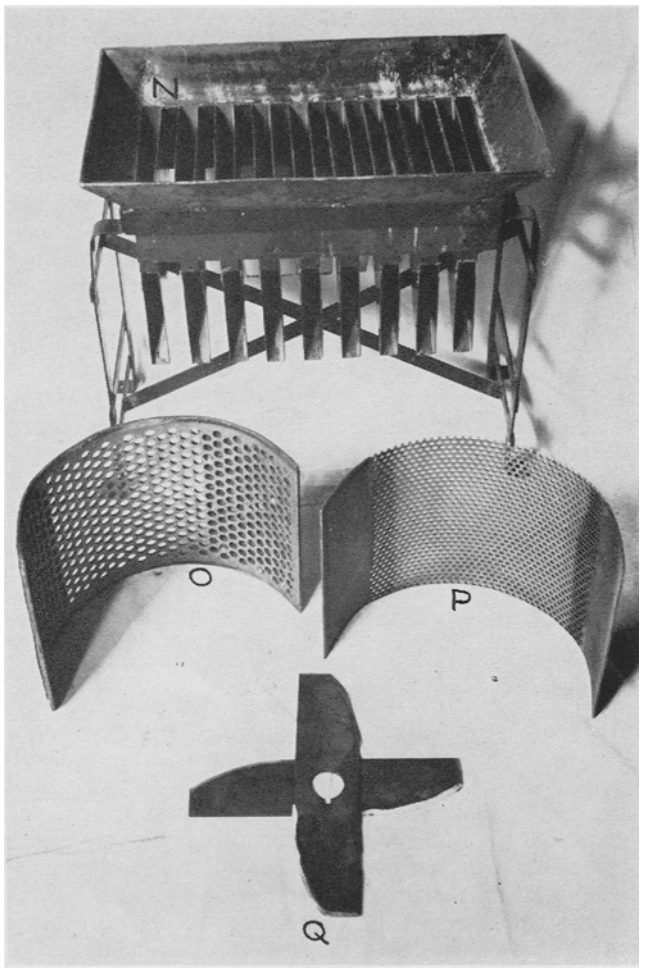

FIG. 3. Sample splitter, screens, and chopper blades.
Sample splitter N, ½" screen O, ¼" screen P, and chopper blades Q are shown.
Blades are 12¾" from end to end. Screens are semicircular, 13" in diameter.

air at this low temperature. Without the cooler the ether readily boils off and fills the room, making prolonged trituration and complete extraction impossible. In the extraction of tung kernels with hexane in a Waring Blendor, without a cooler, temperatures as high as 45°C. at the end of 5- and 10-gm. sample runs have been registered (5).

The Water Bath and Vacuum Manifold. A constant temperature electric water bath 19¾" x 17" x 8¼" is used to heat the samples under vacuum. It has four 6" openings with concentric rings which serve as lids. It is the so-called "Model A" water bath sold by the Central Scientific Company.

Since the tops of water baths are invariably made of thin material, which bends easily, this one has been re-enforced with copper bars, soldered underneath, to support the vacuum manifold, soldered on the top, equidistant from the four openings, as shown in Figure 5. The vacuum manifold in this case consists of a cylindrical vacuum chamber 1½" in diameter and 8" long, four stopcocks and a vacuum gauge at the top, and a horizontal connector tube at its base. The connector tube is attached to the vacuum hose leading from the glass condensers, which provide the manifold with vacuum. Each of the four stopcocks is provided with a short length of tubing for connecting with the filter flasks and the drying jars on the bath. This arrangement makes possible simultaneous operations. The vacuum gauge is absolutely necessary in the operation of the manifold. The water bath has also been provided with an extra tray inside for holding the flasks only 1" deep in the water.

Although developed exclusively for the analysis of copra by the Rapid Method, the vacuum manifold in conjunction with the water bath has found many other uses in the laboratory. In addition to the uses for which it was developed it has been used to remove quickly solvents and other volatile matter from materials, dry oil samples to make color reading possible when the moisture content is high, break up oil-water-meal emulsions, carry out filtrations under suction and heat when heat is necessary to maintain materials in a fluid state, determine oil content in miscella rapidly, etc.

The Drying Jar and the Drying Dish. The drying jar was developed to dry samples under heat and vacuum on the water bath. It consists of a thick wall Pyrex cylindrical vessel 3" in height and 4" in diameter. A metal cover, having a nipple at the top for the vacuum hose and a rubber gasket underneath, fits tightly over the mouth of the jar. Placed in the water bath at 100°C. under the reduced pressure of the vacuum manifold, it is an efficient device for drying the copra samples before oil extraction and can be used instead of an electric oven. It can also be used to determine the moisture content of copra by placing within moisture dishes with 5- or 10-gm. samples.

Instead of the drying jar, Pyrex dishes 5 cm. high and 12 cm. wide at the top, fitted with covers like that of the drying jar, are also used. If the copra is dried in the oven instead of on the water bath before oil extraction, these dishes are better suited than the drying jars because they are lighter and their shape permits the heat to penetrate the samples better.

The Condensers for Recovering the Solvent. A 40-cm. reflux condenser is set upright, connected with a 500-ml. boiling flask at the lower end. The flask, which has a stopcock at the bottom, serves as a receptacle to receive the solvent condensate, which is in turn drained through the stopcock when necessary. A smaller condenser, 20 cm. long, is set up at about 45° to the vertical, the lower end of which leads to a connecting spout on the neck of the flask. The flask spout and the condenser are fastened together by means of a small piece of rubber tubing.

To prevent any oil from passing from a sample flask into the condensate receiving flask and contaminating the recovered solvent, should a sample accidentally boil over, a trap is set below the upper end of the small condenser and is attached to the latter by means of an upright piece of glass tubing and to the connector tube of the vacuum manifold by means of rubber tubing. The lead from the filter pump (also called aspirator, air-ejector, etc.) is then attached to the top of the vertical condenser, thus subjecting the entire system to the vacuum action of the pump. The vacuum is controlled by hand by means of a lever-handle stopcock placed between the filter pump and another trap for the pump.

In operation the solvent vapors pass first through the small condenser, the condensate runs down into the receiving flask, and any solvent still in the vapor state is likewise cooled and brought down by the upright condenser. In addition to the fact that about 200 ml. of solvent are thus recovered from each sample, their disposal through the drain is avoided.

Analytical Procedure

1. *Preparation of Sample*

The copra chopper is fitted with the ½" screen and the sample (usually a sack containing 80 or 100 lb.), sent for analysis by the official sampler at the point of discharge, is placed on the copra trough over the chopper. The chopper is started, the sack is cut open, and the copra is pushed along the trough and down into the chopping chamber with a stick. Rocks, pieces of steel, bottle caps, etc., should be removed while the copra is being pushed down along the trough. After this operation, which takes only a few minutes, the chopper is stopped, the screen is removed, and the copra that might have adhered to the sides is cleaned down into the drawer below.

The chopped copra is then quartered through the sample splitter. Three of these portions are discarded, and the other is again split into two equal portions. One of these portions is also discarded and the other, which for convenience is here designated as portion A, is sifted by hand through a 12" No. 6 sieve into a large pan. The copra chopper is then fitted with the ¼" screen, and the portion of A which did not pass through the sieve is chopped again. The ¼" screen is then removed and the chopper is cleaned once more into the receiving drawer containing the chopped copra.

The sifted and chopped portions of A are then put together in the receiving drawer and mixed well. This copra, nearly all of which now could pass through the No. 6 sieve, is split again into two parts, B and C, and either of the two equal parts may be used for the analysis.

When the copra sample consists of not more than three pounds, as is the case when shipped by air from long distances, neither copra chopper nor sample splitter is needed. The entire sample is first chopped with a knife into pieces, not larger than ½", then comminuted in the blender to a suitable fineness and finally treated just as either B or C below.

In the preparation of the sample for trituration in the blender the copra from either B or C is spread out in a large flat pan and mixed well and then with a tablespoon 75 gm. of it are weighed into a drying dish or drying jar from the various parts of the pan. This amount of copra yields about 50 gm. of oil, sufficient for color determination on a 5¼" standard tube. A torsion balance of 500-gm. capacity and 15-mg. sensitivity is used in all weighing throughout the analysis except in moisture determinations.

The dish is then placed in an electric oven at 102°C., and the moisture is reduced to about 1.5%. With copra containing in the neighborhood of 4% moisture 45 minutes of drying is sufficient. If copra is extracted without previous drying, complete extraction is difficult, if not impossible, and the excess moisture tends to dissolve coloring and colloidal matter which affects color reading of the oil. If the moisture content exceeds 4% the oil extraction is progressively decreased with an increase in moisture, the colloidal matter and turbidity are increased, and the acidity also rises. Prolonged drying in the oven, on the other hand, may not only cause a loss in free fatty acids, but also a darkening of the oil. If the copra is dried and about 1.5% of the total moisture is still left in the sample, a thing easily done because the last 1% moisture takes rather long to remove, the free fatty acids and color of the oil are not affected, extraction is complete, and filtration is much easier.

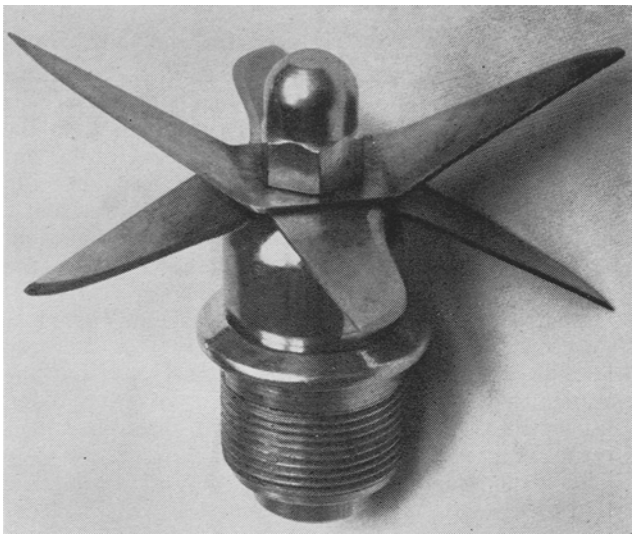

FIG. 4. The special blade assembly.
Six cutting edges instead of the conventional four. Designed and constructed for rapid comminution of materials and rough treatment.

Instead of drying the copra in the electric oven, it may be efficiently dried in a drying jar or drying dish, under vacuum, on the water bath at 100°C. Here too a drying time of about 45 minutes is sufficient, but again from 1 to 2% of moisture must be left in the copra. Prolonged drying on the water bath under vacuum does not darken the oil but with some copra sufficient free fatty acids are removed to cause low results in acid and color determinations. Therefore be the copra dried in the oven or on the water bath, from 1 to 2% moisture must be left in the sample.

Once copra is chopped, the analysis must be completed without delay because the acidity begins rising and is appreciably higher even on the next day (2), and the color of the oil is also progressively increased.

2. *Trituration and Filtration*

These operations consist of comminuting the copra with a solvent in a suitable blender or mixer and separating the solution from the fine meal by filtration under suction. The Waring Blendor, equipped with the special blades and cooler described, is a good mixer for this purpose, and it is the one we use at present. A good grade of ethyl ether (10) or petroleum ether of 35°-60°C. boiling range (11) is used.

The blender is fastened to the base of a large ringstand by means of two small screws. The cooler is clamped by the neck to the ringstand post and adjusted so that it can be raised or lowered into the container without touching the sides. A variable transformer in the circuit with the blender controls its speed.

A 500-ml. tared filtration flask is fitted with a 350-ml. medium porosity fritted glass disc funnel (Buchner type), and a circle of 7½-cm. Whatman's No. 1 filter paper, or a similar smooth surface qualitative paper, is placed inside of funnel.

A wash bottle with solvent is also used. This is a 500-ml. Smith sanitary bottle (Arthur H. Thomas Company, Philadelphia, Pa. See Oil & Soap, July, 1946, p. 9), the nozzle of which has been constricted

on the flame to deliver a fine jet. No other wash bottle has been found better suited for this work.

Having all the equipment in readiness, the dish of dried copra is emptied into the container of the blender, and any oil and copra still adhering to the dish are washed into the container by a jet of solvent from the wash bottle. To the container is added 100 ml. of solvent and the cooler is brought down into the container within about one inch from the contents. The variable transformer is set at 70, the blender is started, and the transformer is then increased to 120 volts. If the blender is not started at a reduced speed the surge is likely to run over the container and, as said before, the solvent may be drawn into the motor and a fire may result. When the machine is started at a reduced speed however, no trouble is encountered.

When the machine is running at full speed, the cooler is adjusted on the surface of the whirling contents, where running is best, as determined by the sound of the motor and the smoothness of the surface of the liquid. An occasional squirt from the bottle will bring down any particles of copra that adhere to the sides of the container and cooler during trituration, thus bringing them into contact with the blades.

When trituration has been carried out for 10 minutes, the contents are filtered into the flask under suction making sure that the vacuum has been turned on and the filter paper is well stuck to the bottom of the funnel before pouring. If this precaution is not observed, the fine meal will clog the funnel disc, making cleaning necessary before another sample can be filtered. After pouring, the container is thoroughly washed with solvent from the bottle and emptied into the funnel.

As soon as all the solution disappears through the filter cake, the oil adhering to the wall of the funnel is washed down with solvent from the wash bottle. Then to wash down the oil still in the filter cake, 100 ml. of solvent are poured in, 50 ml. at a time, and the funnel is allowed to drain well. Before all the solvent has passed through however, the flask and funnel are tilted and rotated gently to wash down any oil that may be sticking to the inside wall of the funnel below the disc. The oil left in the cake, which is about .15% of the weight of the copra, can be washed down with another 50 ml. of solvent, if desired.

When protected by a circle of filter paper, many samples can be filtered through the same funnel without clogging. When filtering becomes difficult, the disc of the funnel is cleaned by reverse washing with solvent or with chromic acid cleaning solution.

Should the analysis consist of only free fatty acid and color determinations, a fritted glass disc funnel is not necessary nor the complete washing down of the oil from the filter cake. The contents may be filtered through a porcelain 4" Buchner funnel, fitted with a 9-cm. circle of Whatman's No. 1 filter paper, or any other similar paper, and then the filtrate is filtered once more through the same funnel and cake into a new flask. Since the oil adhering to the cake is not washed down, about 100 gm. of copra are required to give enough oil for color reading in a standard tube. About 120 ml. of solvent are required for a 100-gm. sample.

Instead of using filtering flasks for filtration and the boiling off of the solvent, low-neck, wide-mouth extraction flasks may be used, especially when dealing with small samples. They are lighter than filtering flasks and boiling seems to be more easily controlled.

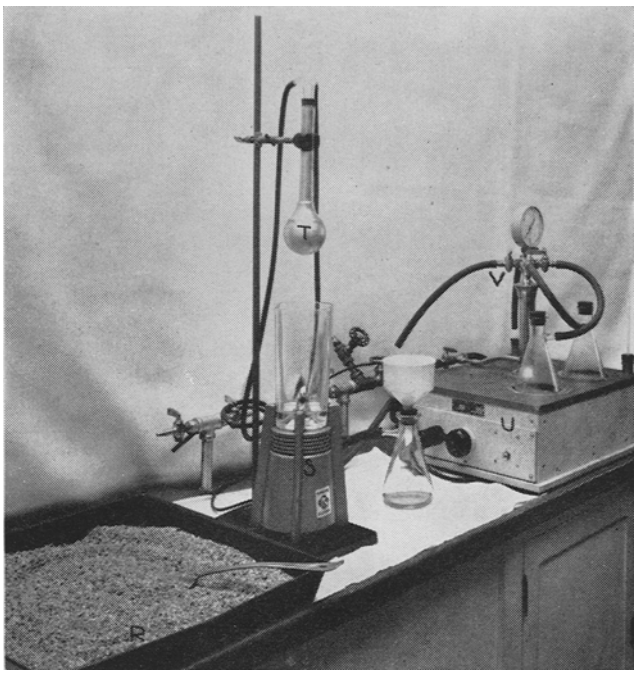

FIG. 5. Laboratory set-up.

The figure shows chopped copra and spoon in pan R, blender S, surface cooler T, water bath U, vacuum manifold V, filter flasks and Buchner funnel. Variable transformer, condenser system for recovery of solvent, and filter pump are not in view.

3. *Removal of the Solvent From the Oil*

When filtration is completed, a small pinch of sea sand (about 7 mg.) is put into the flask by means of a scoopula or small spoon and the flask is stoppered. The sand prevents bumping and hastens boiling and, since the sensitivity of the balance is 15 mg., it has no effect upon the tare of the flask. Ignition of the sand before use renders it more active.

The filter pump is started, the cooling water of the condenser is turned on, and the stoppered flask is placed into one of the openings of the water bath, which is at 100°C., and connected to one of the leads of the vacuum manifold.

Since the volume of the solution is rather large for a 500-ml. flask, care must be exercised for a few minutes at the beginning in order to prevent any loss of oil due to a too vigorous boiling. This is easily accomplished by controlling the vacuum to within about two inches on the gauge by means of the lever-handle stopcock between the filter pump and trap. It is also advisable to press down lightly on the flask stopper until the condensation of solvent vapors begins to appear in the receiving flask. At this point the operation consists merely of volatilizing the solvent on one side and condensing it on the other and therefore the vacuum can be increased occasionally, as boiling permits, and then shut off completely, thus preventing solvent vapors from escaping through the filter pump. The vacuum is gradually increased to 15 inches and then the controlling stopcock is shut off, leaving the system under the vacuum until no more visible solvent is being recovered. One of the stopcocks of the manifold is then opened to allow the atmospheric pressure into the system, and

the recovered solvent is drawn off at the bottom of the flask under the vertical condenser.

The sample is then subjected to the full vacuum of the filter pump until all the solvent and perhaps a small amount of moisture present are removed. Under an absolute pressure of 2 cm. and a temperature of 100°C. this is fully accomplished in about 45 minutes when ethyl ether is used, but with petroleum ether one hour should be allowed.

When several samples are to be run on the same day, the solvent from each flask is first recovered and the sample set aside until it has been recovered from all of them. A new pinch of sand is then added to each flask for the first sand added has been rendered inactive, and all the samples are put back under the full vacuum of the pump for about one hour.

While the solvent from one sample is being recovered however, the next sample can be triturated in the blender or filtered in order to save time.

4. *The Oil Content*

The percentage of oil in a 75-gm. sample of copra is the weight of solvent-free oil in the flask multiplied by 4/3, or be it 1.333. After the amount of oil is determined the flask is stoppered again and shaken to bring into solution any free fatty acids that may be adhering to the sides.

5. *Color Reading*

A 5¼" column of the oil is read for color in a standard tube against yellow or red Lovibond color glasses by the Wesson's method (9). The same oil is then used for the free fatty acid determination.

6. *The Strength of the NaOH Solution*

In plant control a large amount of NaOH solution is usually prepared at a time and its strength adjusted to a normality which will make routine determinations easy. Since per cent free fatty acid, expressed as oleic, is

$$\frac{28.2 \times \text{ml. of NaOH} \times \text{Normality}}{\text{weight of sample}}$$

the normality of the solution is adjusted to .3546 so that, when a 10-gm. oil sample is titrated, its per cent free fatty acid corresponds to the number of ml. of NaOH used in titration. If a 20-gm. sample is taken, the number of ml. of solution used in titration is divided by 2. We use 20 grams of oil in our free fatty acid determinations on crude oil and 40 on refined.

Evidently the use of this normality simplifies the work considerably, and the end point is reached sooner and is sharper than it is when a weaker solution is used. With this normality no special weights need be prepared for routine determinations. If a weight is lost, another is readily available.

7. *Titration of the Oil Sample*

A 250-ml. Erlenmeyer flask is balanced on the torsion balance and 20 grams of the oil used for color reading are poured into it. Fifty ml. of neutral ethyl alcohol are added. The alcohol is neutralized by the addition of NaOH solution until a faint pink color of phenolphthalein persists. The flask is heated to light boiling on an electric hot plate, with occasional shaking, and immediately titrated to the same pink end-point. The sample is agitated with a swirling motion while being titrated until the end-point will be able to persist for 30 seconds after titration. The percentage of free fatty acids is equal to the number of ml. of the NaOH solution used, divided by 2, when the .3546 N solution is used.

8. *The Preparation of Samples for Moisture Determinations*

The Waring Blendor, equipped with the special blades described, is likewise a good machine for this work. In a matter of seconds it comminutes samples to a fineness difficult to obtain in any other way without appreciable loss of oil and moisture.

From part B or part C approximately 100 grams of chopped copra are placed in the container, and the machine is operated for a few seconds at reduced speed. The copra adhering to the wall of the container is then loosened with a small spatula, the machine is run for a few seconds longer, and the sample is ready for weighing. If a sample, thus prepared, is washed with ether and dried, 50% of it usually passes through a No. 30 sieve.

Five-gm. samples are then weighed into moisture dishes and placed in an electric oven at 102°C. for 1½ hours, or they may be placed in a drying jar and dried under vacuum on the water bath at 100°C. for the same length of time.

9. *Expeller Cake*

In the determination of the oil content in expeller cake by the Rapid Method no laboratory flour mill is required to grind the sample. Seventy-five gm. of broken-up cake are weighed and put into the container of the blender, the cover is put on it, and the blender is run for a few seconds to pulverize the cake. One hundred ml. of solvent is then added and the contents are triturated for five minutes. The rest of the operation is the same as in the copra analysis but in this case only 10 minutes are required for complete removal of the solvent from the oil after solvent recovery because of the small amount of oil present.

If it is desired to determine color and free fatty acids of the oil in expeller cake, this method is likewise at a decided advantage because of the rapidity with which complete extraction of large amounts of oil is accomplished.

The Rapid Method in the Analyses of the Various Oil Seeds

Although the method was developed exclusively for the analysis of copra, it can also be used successfully for the analyses of the various oil seeds. Cotton seed, soy beans, peanuts, flaxseed, castor beans, sesame, palm kernel, coquito kernel, safflower seed, hemp seed, mustard seed, apricot kernel, peach kernel, and walnuts have been run for oil content and the results were good.

With cotton seed for instance the seeds were first treated by the oven and HCl method, as is customary, and then run with petroleum ether in the blender for 10 minutes. The filter cake was then dried in the oven, ground with sand, and extracted on the Goldfisch extractor for four hours, giving a residual extract of only .15%, based on the weight of the cake taken.

Some seeds were first comminuted in the blender and then dried before oil extraction and others were not. The castor beans were put into the blender and extracted with ethyl ether without previous comminution or drying in the oven. In a few seconds

these materials can generally be chopped fine and rendered fluffy, a thing which is not possible with laboratory mills when the oil content is high, and are then used in moisture and oil content determinations.

It was found that filtration is improved when some Polycel-RCB-80 or Super-cel filter aids are added to the contents in the container after trituration and mixed for a few seconds before pouring into the funnel. In some cases, it was also found, a funnel of coarse porosity worked better than the medium porosity funnel used for copra.

The removal of the solvent from the oil of 75-gm. samples of most of these seeds was accomplished in only ½ hour because these materials have generally a lower oil content than copra does.

Detailed procedures for the analyses of the various seeds have not been worked out because in our mill we seldom crush anything other than copra. Such details will be left to those who will find the method advantageous. Recently the Soybean and Flaxseed Advisory Committee voiced the need for quick and accurate determinations of oil content and quality of oil in oil seeds, stating that "such quick tests would facilitate trading and be a boon to sound production and marketing practices" (12). The authors hope that the present method will assist in fulfilling that need.

REFERENCES

1. P. W. Tompkins. "Copra Sampling, Inspection and Analysis," Oil and Fat Industries, *4*, No. 1 (Jan., 1927).
2. P. W. Tompkins. "Copra Quality Under New Rules," Oil and Soap, *XVIII*, No. 5 (May, 1941).
3. John P. Nielsen and G. S. Bohart. "Determination of Crude Lipid in Vegetable Matter," Industrial and Engineering Chemistry, Analytical Edition, *16*, No. 11 (Nov., 1944).
4. R. B. Oesting and I. P. Kaufman. "Rapid Determination of Fat in Meat and Meat Products," Industrial and Engineering Chemistry, Analytical Edition, *17*, No. 2 (Feb., 1945).
5. Joseph Hamilton and Seymour G. Gilbert. "Rapid Method for Determining Oil Content of Tung Kernels," Industrial and Engineering Chemistry, Analytical Edition, *19*, No. 7 (July, 1947).
6. A. L. Ayers and J. J. Doodly. "Laboratory Extraction of Cotton Seed With Various Petroleum Hydrocarbons," The Journal of the American Oil Chemists' Society, *XXV*, No. 10 (Oct., 1948).
7. Lawrence Zeleny and D. A. Coleman. Rapid Determination of Oil Content and Oil Quality of Flaxseed, Technical Bulletin No. 554, United States Department of Agriculture, Washington, D. C., Feb., 1937.
8. International Chemical Union. "Minutes of Meeting of International Commission on Fats and Oils," Part II, The Journal of the American Oil Chemists' Society, *XXVI*, No. 3 (March, 1949). (London, England, July, 1947; translated by C. L. Hoffpauir.)
9. George S. Jamieson. Vegetable Fats and Oils (1943), p. 375.
10. Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists (1945).
11. Official and Tentative Methods of the American Oil Chemists' Society (1947).
12. "Soybeans and Flaxseed Advisory Committee," The Cotton Gin and Oil Mill Press, August 21, 1948, p. A-3.

[Received for publication May 6, 1949]

Oil From the Kernels of Lalob Fruit, Balanites Aegyptiaca[1]

S. A. HUSSAIN, State of Hyderabad (India), and F. G. DOLLEAR and R. T. O'CONNOR, Southern Regional Research Laboratory,[2] New Orleans 19, Louisiana

Balanites aegyptiaca is a tree 20 to 30 feet high which is found principally in West Africa, Sudan, Uganda, Tanganyika, Palestine, and Arabia. The fruit of this tree is known locally by such names as "betu," "lugba," "heglig," and lalob. The kernel of the fruit, which contains about 46% oil, amounts to 9 to 10% by weight of the whole fruit.

The fruit, which usually falls to the ground on ripening, is often parasitized by the larvae of an unidentified noctuid moth. The caterpillar develops inside of the fruit and when ready to pupate, eats its way to near the surface, leaving a membraneous window through which the adult moth emerges. This parasitism is responsible for considerable loss of kernels of wind-fallen fruit.[3]

Except for two anonymous reports (1, 2) from the Imperial Institute of London in 1908 and 1935, respectively, practically no information appears to have been published on the characteristics and composition of lalob kernel oil. Because of the current interest in exporting lalob kernels for crushing and the reported similarity of the oil to cottonseed oil, a comprehensive investigation was made of the fruit and oil by modern chemical and physical methods (3).

Experimental

Material: Lalob fruit used in this investigation was grown in the Fung district of Sudan.[4] The fruit resembles the date in size and appearance and consists of a loose, thin brownish-yellow skin, covering a brown sticky pulp, embedded in which is a hard woody shell enclosing a light yellowish kernel.

Components: The percentage distribution of the component parts of lalob fruit together with the contents of moisture and lipids made on 10 selected fruits weighing an average of 7 grams each is given in Table I.

TABLE I

Distribution of Principal Components of Lalob Fruit With Their Moisture and Lipid Content

Component	Proportion of whole fruit	Moisture	Lipids
	%	%	%
Outer covering	21.6	16.2	.....
Sticky pulp	34.8	26.8	.....
Shell	34.1	10.5	.....
Kernel	9.5	5.7	46.5

Extraction of Oil: A large lot (8.8 kg.) of lalob fruit were dried in a vacuum oven at 158°F. for 20 hours to facilitate cracking and separation of kernels. Only 2.2% moisture could be removed by drying, and the pulp was still very sticky. The incompletely dried fruit was cracked and the kernels separated by hand. All worm-damaged fruit (12% by weight) were rejected, and only sound kernels were used for extracting the oil. The original lot of dried lalob fruit yielded 10.2% by weight of sound kernels.

The kernels (908 g.) were ground in a food grinder and extracted with commercial hexane (b.p. 146°F.) in a laboratory all-glass extractor. At the completion of extraction the bulk of the solvent was removed

[1] Presented at the 40th Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 10-12, 1949.
[2] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.
[3] Information supplied by A. W. M. Disney, Department of Economics and Trade, Sudan Government, Khartoum.
[4] The lalob fruit used in this investigation was supplied by the Greene Trading Company inc., New York, *via* the Sudan Trading Company Ltd., Khartoum, Sudan, Africa.