found in commercial soybean oil meals. The straight-line arrangement of points on this graph shows the method should give excellent relative values over a 60-fold range in activities.

### Comparison of the Methods

The results for the three methods are compared in Figure 6. Seven commercial samples of soybean oil

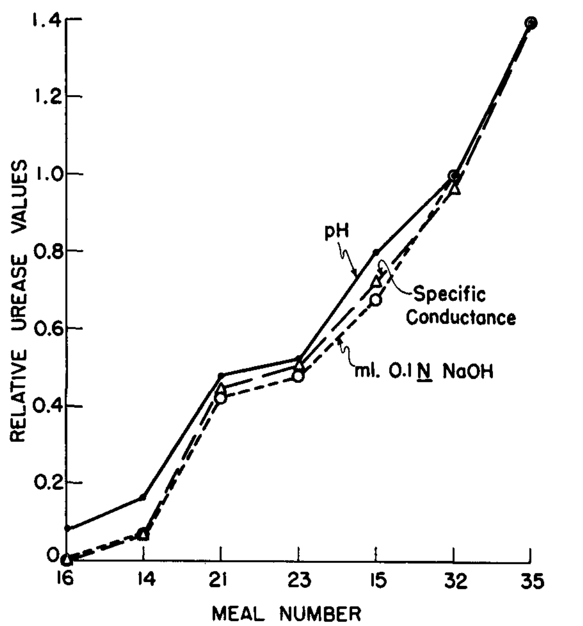


FIG. 6. Comparison of values obtained by the three methods of assay for meals of varying urease activity.

meal having a wide range of activity were used for comparison. The numerical values for the three methods were brought into the same range by multiplying the values obtained by the pH method by 4 and the conductance values by 0.85. The results by the three methods check each other very well.

The differences in the three methods of assay are found principally in the operational techniques. The titrimetric method has the advantage of giving a direct measure of urease activity, but the end-point is not sharp and good results require extreme care in titration. The titration method requires appreciably more time than the others. The pH-change method is simple in operation, but the recorded differences between samples at room temperature are very small and frequent checking of the meter is required to prevent substantial errors. The sensitivity of the titrimetric and pH-change methods can be improved by using 50° or 60°C. reaction temperatures, and a further spread in determined values can be obtained by increasing the size of sample or reaction time. For a final determination of the relative adaptability of the three methods for the control of toasting, they must be compared under practical operating conditions. However from laboratory experience it is the opinion of the authors that maximum precision is obtained with the least skill by the conductimetric method.

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[Received November 5, 1954]

# Materials Balance in a Tung Oil Mill

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TUNG OIL is produced from seed of the tung tree *(Aleurites fordii* and *A. montana)*, a member of the Euphorbia family. The tung fruit is about 2 in. in diameter and normally contains four or five seeds. The entire fruit is covered by a hull about a quarter of an inch thick. Each seed is coated by a hard shell; in it is the kernel, which constitutes about a third of the weight of the fruit and contains about 65% oil. About 39,000,000 pounds of tung oil were produced in the southeastern states from the 1953 crop.

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In the United States tung fruit is processed by pressing in continuous screw presses. A materials balance on a tung mill has never been published, however. Such a balance is needed to show the losses that occur at different stages of the milling process so that more intelligent efforts can be made to lower the losses. It is also needed to check the accuracy of the methods of analysis used for oil in tung products by determining if the weights of oil shown by analysis of the fruit are actually equal to the oil produced plus that left in by-products. This paper reports a materials balance made in a commercial mill under its normal operating conditions.

The filter cake from tung mills contains 40% or more of tung oil. A much-discussed question in the tung industry has been whether this cake should be fed back through the screw presses to try to recover part of this oil, or disposed of otherwise. This experiment was designed to give information also on the feasibility of feeding the cake back through the presses.

### Processing and Sampling

A flow diagram of the milling process as carried out in the mill in which these tests were run is shown in Figure 1. As the process in all tung mills in the tung belt of the southeastern United States differs from this only in detail, the process will be described at considerable length.

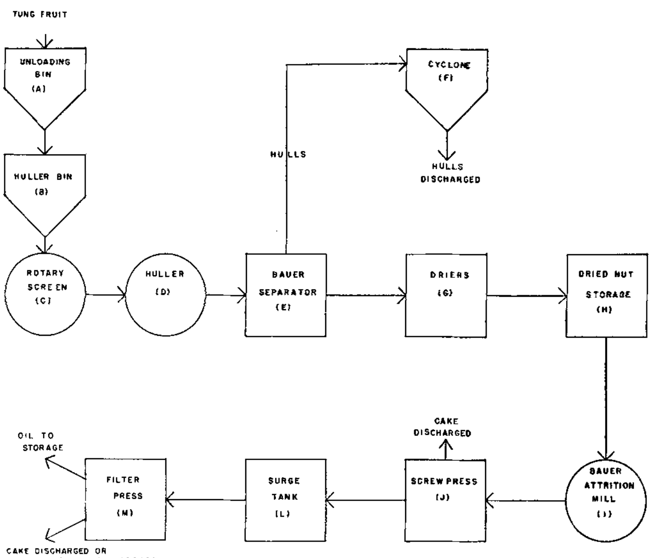


FIG. 1. Flow diagram of tung milling. See text for explanation.

Tung fruit in bags is unloaded from trucks into the unloading bin (A). From this bin it is conveyed to another bin (B) located above the huller, from whence it passes through a rotary screen (C). This screen removes most of the dirt and loose particles of hulls. The fruit is then broken down in a huller (D) of the basket type (7). The huller consists of a drum carrying fluted hard rubber flanges which revolve inside an iron shell with perforations just large enough to allow individual hulled nuts to pass through. (Bauer[4] hullers are used in many mills instead of basket hullers.) It removes practically all of the hulls and part of the shells from the kernels. The mixture of hulled nuts and hulls is discharged to the Bauer[4] separator (E), where the loose hulls are removed by suction and discharged through the cyclone (F). Any unhulled nuts are returned from the separator to the huller. The hulled nuts then pass through driers (G) built to the plans designed by the Florida Engineering Experiment Station (4). The nuts are dried by blowing hot air through them as they descend through louvered columns. The nuts should be dried to 6-10% moisture (2, 3). Occasionally fruit will be encountered dry enough so that the nuts need no further drying. From the driers the nuts are discharged into storage bins (H) where they usually have time to cool before they are further processed.

Dried nuts are taken from the storage bins to a Bauer[4] attrition mill (I), where the nuts are ground to a coarse meal. This mill consists of a rotating, grooved plate revolving past a grooved stationary plate. The clearance between the plates can be varied and controls the particle size of the meal. From the attrition mill the meal passes through a continuous screw press (J), which consists of an interrupted screw that moves the material forward within a cylinder of steel bars set close together against a restricted opening. The cake, containing on the average about 5% oil, is discharged through the constricted opening at the far end of the cylinder, and the oil flows out between the bars of the cylinder. This cake is ground through hammer mills and sold for fertilizer. The cake is hot as it comes from the presses, and it must be allowed to cool before storage to decrease danger from spontaneous ignition. Usually sufficient cooling takes place in conveyors. The crude oil flows into a surge tank (L). At intervals it is filtered through a plate and frame filter press (M). The filtered oil is then pumped to storage. The filter press cake is either discharged from the process and sold to solvent extraction plants, or it is fed slowly back into the stream of ground nuts entering the screw presses.

Two runs, each of about 90 tons of whole fruit, were made in this mill during normal mill operations. In the first run the filter cake was discharged from the process, but in the second run it was fed back through the screw presses.

The fruit used in the experimental runs had been hanging in burlap bags in trees for several weeks with no rain and was very dry. The bags were hauled to the mill in trucks and emptied into the fruit hopper. For the first run the weight of fruit was calculated from the weight of the hulls. For the second run the loaded truck was weighed, and, after unloading, the empty truck plus bags was weighed as a tare. The weights of trash discharged at C, of hulls discharged at F, of screw press cake discharged from J, and of the filter press cake discharged from M were determined either by weighing on platform scales or in trucks. The volume and temperature of the oil discharged at M was measured so that the weight could be calculated.

The whole fruit was sampled as it dropped from a conveyor belt into huller bin (B). Each hour a gallon of fruit was caught in a bucket. The composited fruit from each 8-hr. shift was handled as a separate sample. Grab samples of the trash discharged from the rotary screen (C) were taken each hour and composited into one sample for each run. Hourly samples of wet hulled nuts were collected at the discharge from the Bauer[4] separator (E) and composited into a single sample for each run. Once each 8-hr. shift a 50-lb. lard can was filled with hulls at the discharge from the cyclone (F). Each of these cans was handled as a separate sample. Samples of dried nuts were taken every few minutes, while the driers (G) were being emptied, and were composited into a single sample for each run. The screw press cake was sampled each hour as it was discharged from the press (J) and composited into a single sample for each 8-hr. shift. Small samples of oil were caught each hour from the filter press (M) while filtration was in progress, and these were composited into a single sample

[4] Trade names have been used only for the purpose of identifying equipment or materials actually used in conducting the work and such use does not imply endorsement or recommendation by the U. S. Department of Agriculture over other firms or similar products not mentioned.

for each run. A representative gallon sample of filter press cake was also obtained during each discharge of the filter press.

### Results and Discussion

Samples of all materials were analyzed for moisture, oil, and nitrogen at the Bogalusa Tung Oil Laboratory, or at the Southern Regional Research Laboratory. The moisture content was determined by drying in a vacuum oven at 100°C. for 2.5 hours. The oil and nitrogen contents were determined by procedures of the American Oil Chemists' Society (1) except that in the nitrogen determination the ammonia was absorbed in boric acid solution. Previous experience had indicated that the extractable oil in the screw-press cake might decrease rapidly after extrusion because of polymerization. For this reason a laboratory was set up at the mill to determine the oil content of the screw-press cake by a rapid method (5) as soon as samples were taken. These samples were also analyzed several days later by the usual methods of analysis.

TABLE I
Comparison of Percentage of Oil in Screw Press Cake at Time of Sampling and a Week Later

| Sample No. | % oil by rapid method at time of sampling | % oil by A.O.C.S. method a week later |
|---|---|---|
| | Run No. 1 | |
| 1 | 5.7 | 4.1 |
| 2 | 6.6 | 4.0 |
| 3 | 5.6 | 4.2 |
| 4 | 6.3 | 4.2 |
| 5 | 6.3 | 3.7 |
| 6 | 7.0 | 4.8 |
| 7 | 7.0 | 4.5 |
| Averages | 6.4 | 4.2 |
| | Run No. 2 | |
| 8 | 5.5 | 4.4 |
| 9 | 6.5 | 4.3 |
| 10 | 6.0 | 4.4 |
| 11 | 5.3 | 5.0 |
| 12 | 6.6 | 5.3 |
| 13 | 6.5 | 6.2 |
| 14 | 8.9 | 6.3 |
| 15 | 8.9 | 5.2 |
| 16 | 7.5 | 6.0 |
| Averages | 6.9 | 5.2 |

In Table I are given the oil contents of the screw press cake as determined by the rapid method of analysis at the time of sampling and by the method of the American Oil Chemists' Society, about eight days later for Run No. 1 and about 4 days later for Run No. 2. In the interval the extractable oil had decreased by 2.2% for the first run and 1.7% for the second run. Since less opportunity for polymerization of oil had occurred in the samples at the time they were analyzed by the rapid method, the values by this method are considered more reliable and were used in calculating the oil balance.

The weights and analyses of all incoming and outgoing materials for the first run are given in Table 2. The weight of fruit in this run was calculated from the weight of the hulls (including trash) and the analyses of the whole fruit, hulled nuts, and hulls (6). The ratio of the weight of fruit to hulls was calculated from the oil contents of the fruit, hulled nuts, and hulls; and the weight of fruit was obtained by multiplying the weight of the hulls by this ratio.

In this run the weights of all materials were satisfactorily accounted for, considering the difficulty of sampling such materials as tung products and of getting accurate weights. Ninety-eight per cent of the dry matter, 101% of the nitrogen, and 101.7% of the oil were accounted for.

The loss of oil in the hulls is excessively high, amounting to 8.9% of the oil in fruit. The fruit was very dry and was hulled in a basket type of huller which was designed to handle comparatively wet fruit (7). Broken kernels carried over with the hulls from this huller were responsible for this high loss. It should be pointed out that several mills have installed gravity separators which recover part of the kernel particles and are said to reduce considerably the loss of oil in the hulls. A separator of this type was installed in the mill in which these tests were made, but it was out of order when these tests were made. Losses of oil in the screw-press cake and filter cake were 7.1% and 5.9% of the total oil, respectively. The loss of oil in the screw-press cake is quite high as shown by an oil content of 6.4%. The latter loss is probably caused by too much drying of the hulled nuts in the driers as the dried nuts contained only 2.6% moisture. As a result of these losses the recovery of filtered oil was quite low, only 78.2%.

In Table III are given the weights and analyses of materials for Run 2. All materials in this run are also fairly well accounted for. The oil accounted for is within less than 2% of that in the fruit, but the dry matter and nitrogen are too high by 3% and 11%, respectively. The high proportion of nitrogen indicates that the weight of the screw-press cake may have been somewhat too high.

The loss of oil in the hulls is satisfactorily low, being only 2.8% of the oil in the fruit, but the loss of oil in the screw-press cake is very high, amounting to 10.1% of the oil in the fruit. This high loss was also probably caused by excessive drying of the hulled nuts as the dried nuts contained only 2.1% moisture.

The oil content of the filter cake and the time of filtering and blowing for each of the filtration cycles in the two runs are given in Table IV. The data in

TABLE II
Materials Balance for Run No. 1 at Leon Tung Mill
(Filter Press Cake Not Fed Back Through Screw Presses)

| Material | Wt. lbs. | Percentage comp. | | | Weight in pounds | | | Oil Recovery, % |
|---|---|---|---|---|---|---|---|---|
| | | Moisture | Nitrogen | Oil | Dry matter | Nitrogen | Oil | |
| Whole fruit | 180,342[a] | 10.70 | 1.15 | 21.30 | 161,045 | 2,074 | 38,413 | ...... |
| Hulls | | | | | | | | |
| (a) from cyclone | 80,934 | 13.23 | 0.63 | 4.13 | 70,225 | 516 | 3,341 | 8.7 |
| (b) trash | 11,870 | 11.96 | 0.66 | 0.55 | 10,450 | 78 | 65 | 0.2 |
| Wet hulled nuts | ........ | 6.85 | 1.79 | 40.32 | ........ | ...... | ........ | ...... |
| Dry hulled nuts | .... | 2.55 | 1.95 | 42.82 | .... | ...... | ........ | ...... |
| Screw-press cake | 43,135 | 2.52 | 2.99 | 6.36 | 42,048 | 1,291 | 2,743 | 7.1 |
| Filter cake | 5,235 | 3.07 | 3.87 | 43.44 | 5,074 | 203 | 2,274 | 5.9 |
| Screw-press "foots" | 336 | 1.00 | 2.68 | 64.44 | 333 | 9 | 217 | 0.6 |
| Filtered oil | 30,058 | 0.00 | 0.00 | 100.00 | 30,058 | 0 | 30,058 | 78.2 |
| Total accounted for | 171,568 | ...... | ...... | ........ | 158,188 | 2,097 | 38,698 | ..... |
| % accounted for | | | | | 98.2 | 101.1 | 100.7 | 100.7 |

[a] Weight of fruit was calculated from weight of hulls plus trash and from the analyses of whole fruit, hulled nuts, and hulls.

TABLE III

Materials Balance for Run No. 2 at Leon Tung Mill (Filter Press Cake Fed Back Through Screw Presses)

| Material | Wt. lbs. | Percentage comp. | | | Weight in pounds | | | Oil Recovery, % |
|---|---|---|---|---|---|---|---|---|
| | | Moisture | Nitrogen | Oil | Dry matter | Nitrogen | Oil | |
| Whole fruit | 169,920 | 10.89 | 1.15 | 21.26 | 151,416 | 1,954 | 36,125 | ...... |
| Hulls | | | | | | | | |
| (a) from cyclone | 71,702 | 15.43 | 0.51 | 1.28 | 60,634 | 364 | 918 | 2.5 |
| (b) trash | 10,955 | 12.05 | 0.59 | 0.75 | 9,635 | 65 | 82 | 0.2 |
| Wet hulled nuts | ........ | 6.51 | 1.84 | 40.71 | ........ | ...... | ...... | ...... |
| Dry hulled nuts | ........ | 2.09 | 1.93 | 43.94 | ........ | ...... | ...... | ...... |
| Screw-press cake | 53,338 | 2.17 | 2.92 | 6.82 | 52,181 | 1,557 | 3,638 | 10.1 |
| Filter cake | 4,784 | 2.60 | 3.70 | 44.62 | 4,660 | 177 | 2,135 | 5.9 |
| Screw press "foots" | 464 | 0.65 | 0.86 | 77.84 | 461 | 4 | 361 | 1.0 |
| Filtered oil | 29,507 | 0.00 | 0.00 | 100.00 | 29,507 | 0 | 29,507 | 81.7 |
| Total accounted for | 170,750 | ...... | ...... | ...... | 157,078 | 2,167 | 36,641 | ...... |
| % accounted for | ........ | ...... | ...... | ...... | 103.7 | 110.9 | 101.4 | 101.4 |

this table show that the oil content of the filter cake was quite uniform, within the ranges observed, regardless of the time of blowing.

In the first run 90 tons of fruit were milled. The oil was filtered in three filtration cycles, and none of the filter cake was fed back through the screw presses. In this run 5,235 pounds of filter cake were produced. During the second run in which 86 tons of fruit were milled and part of the filter cake was fed back through the screw presses, a total of six filtration cycles were required. Since cake from the second run was not available for feed-back at the start of the second run, 1,600 pounds of filter cake from the first run was used at the start of the second run. (Data in Tables II and III have been corrected for this transfer of materials from the first to the second run.) If cake is fed back to the screw presses too fast, they fail to produce a satisfactory cake. For this reason only the 1,600 pounds of cake plus that from the first two filtration cycles of the second run could be fed back during the second run, leaving the cake from the last four filtration cycles, 6,615 pounds, left over at the end of the run. Assuming that all the 1,600 pounds of cake added from the first run were finally caught in the filter press and again weighed as cake, this leaves 6,615 minus 1,600 or 5,015 lb. of cake on hand at the end of the second run, which was produced from the fruit of the second run.

TABLE IV

Effect of Filtering and Blowing Times on Apparent Oil Content of Filter Cake

| Cycle No. | Time of filtering hours | Time of blowing hours | % oil |
|---|---|---|---|
| Run No. 1 | | | |
| 1 | 3.00 | 8.78 | 43.35 |
| 2 | 4.08 | 9.18 | 43.56 |
| 3 | 1.33 | 4.00 | 43.42 |
| Run No. 2 | | | |
| 1 | 2.00 | 8.67 | 48.50 |
| 2 | 1.58 | 6.08 | 43.26 |
| 3 | 1.42 | 4.00 | 43.51 |
| 4 | 1.33 | 4.08 | 47.44 |
| 5 | 1.42 | 4.25 | 42.34 |
| 6 | 1.12 | 6.00 | 43.78 |

Without feed-back 4,932 pounds of filter cake should have been produced in the second run, assuming the same ratio of cake to fruit as in the first run. With feed-back of the filter cake, as much cake was still on hand at the end of the run as would have been obtained without feed-back, and this cake was just as high in oil content. Hence no additional oil was recovered because of the feed-back, and with feed-back only about half as much oil was filtered per cycle with the resulting increase in labor costs and lowering of the filtration capacity. Apparently the fine material of the filter cake, when recycled through the screw press, simply passes through the slits of the press barrel and is again caught in the filter press.

## Summary

Weights were determined and analyses made of tung fruit milled and of all products leaving the mill for two runs of about 90 tons each in a commercial mill under normal operating conditions. Dry matter, oil, and nitrogen in the fruit were satisfactorily accounted for in products leaving the mill, 101% of the oil being accounted for in each run. This showed that the methods of analysis and sampling were accurate.

Losses occurred principally in particles of kernels occluded with the hulls and in the screw-press cake. Seventy-eight and 82% of the oil in the fruit was recovered as filtered oil.

Repressing the filter-press cake by adding it back to the stream of ground nuts just before they entered the screw-presses was not proven to be economical as at the end of the run just as much cake was on hand, and it had as high an oil content as if no filter cake had been fed back through the screw presses. Only about half as much oil could be filtered per filtration cycle, resulting in an increase in cost of labor and a decrease in filtering capacity.

The apparent oil content of the screw-press cake decreases by about 2% after four to eight days as compared to its apparent oil content at the time of pressing because of polymerization. Thus, screw-press cake samples should be analyzed for oil as soon as possible after extrusion.

## Acknowledgment

We wish to express our grateful appreciation for the cooperation of E. V. Dunbar and Leon Tung Oil Inc. in making available facilities and operating personnel which made it possible to carry out this investigation.

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[Received November 8, 1954]