Acknowledgment

The authors are grateful to J. C. Cowan for his interest and encouragement throughout this work.

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[Received November 23, 1949]

Comparison of the Whole Fruit and Component Methods of Analysis of Tung Fruit

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THE estimation of oil in tung by grinding the whole fruit was described by McKinney, Halbrook, and Agee in 1948 (1, 2). This method was adopted by the Commodity Credit Corporation as the official method of analysis in its price support for the 1947 crop. Further comparisons between the whole fruit and the so-called *component method* in which the hull- and shell-free kernels are analyzed for oil were contained in a report of the Subcommittee on Tung Fruit and Meal Analysis of the American Oil Chemists' Society in 1948 (3). The most recent report of this committee is given elsewhere in this journal. No data have been previously reported for the effects of moisture content and fineness of grinding in the whole fruit method of analysis. The present report contains the results of a comparison of the two methods which were obtained in the same laboratory, using different portions of the same samples of tung fruit. The samples were chosen to cover a wide range in the contents of moisture and oil.

In the whole fruit method the sample was first ground in a Wiley[2] mill equipped with a quarter inch screen, thoroughly mixed, and an aliquot of about a pint was reground in a Bauer laboratory mill with the plates set close enough to give a fine meal. Since moisture is lost during the fine grinding, it was necessary to determine the moisture content of the sample before and after grinding so that the results could be calculated to the original moisture basis. Moisture was determined on five-gram samples by drying them in a vacuum oven for 2½ hours at 104°C. The oil content was determined by extracting five grams of the finely ground sample with petroleum naphtha (Skelly F) in a Butt extraction tube for four hours.

Because of the extrusion of oil, kernels and seeds cannot be ground finely enough in a Bauer laboratory mill for complete extraction of the oil. Samples ground coarsely enough to avoid extrusion of oil can be analyzed after regrinding the partially extracted sample with sand in a mortar, but particles of shell in the sample interfere with the proper regrinding of the sample.

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[2] The specification by firm name of equipment and special reagents used throughout this article is for identification purposes and implies no endorsement of the manufacturer or product mentioned.

The samples analyzed by the component method were weighed; the kernels were separated from all hulls and shells; and all hulls and shells were collected and dried to constant weight; care was taken not to lose any particles. The cleaned kernels were then weighed, ground, and analyzed for oil and moisture content as described for the whole fruit method, except that after extraction for four hours the partially extracted kernels were ground for five minutes with mortar and pestle and then extracted for two additional hours. The percentage of oil in the fruit was calculated from the total weight of sample, and the weight and percentage of oil in the kernels. The percentage of moisture was calculated from the total wet weight and the total dry weight of sample.

In both methods the bulk of the solvent in the micella was removed rapidly on a steam bath. Thirty minutes in a vacuum oven heated to 100°C. served to remove any residual solvent.

TABLE I

Percentage of Oil Extracted From Tung Fruit as a Function of Fineness of Grinding and Moisture Content. Calculated to Dry Basis

Sample No.	Moisture content, %	Distance between plates, inch			
		0.004	0.008	0.012	0.020
1	7.9	25.64	26.22	.....	.....
2	7.8	26.50	26.49	.....	.....
3	9.0	25.56	25.65	25.55	24.81
4	8.4	26.23	25.68	26.10	25.64
5	40.4	24.23	24.16	24.07	23.46
6	9.3	24.60	23.78	24.64	23.56
7	8.4	25.24	24.79	24.62	.....
8	17.0	23.99	23.67	23.56	.....

The complete extraction of oil from ground tung kernels is known to be difficult (5), and in order to be certain that substantially all of the oil is extracted when using the component method it is necessary to interrupt the extraction and thoroughly regrind the partially extracted kernels with mortar and pestle. However presence of hull and shell particles precludes effective regrinding in a mortar after partial extraction of the oil in the whole fruit method. For this reason the effect of fineness of grinding on the amount of petroleum naphtha-extractable material in the ground whole fruit is very important.

The effect of fineness of grinding on the amount of oil extracted from whole fruit is shown in Table I. Reference to this table shows that there are no dif-

TABLE III

Comparison of Percentages of Oil Obtained by Whole Fruit and Component Methods of Analysis of Tung Fruit

Sample No.	Component method					Whole fruit method				
	Replicates				Av.	Replicates				Av.
1	16.66	16.13	15.80	15.92	16.13	16.19	16.01	16.35	16.55	16.28
2	10.87	12.09	12.25	12.15	11.84	12.74	11.77	13.03	11.21	12.19
3	18.83	17.49	17.93	18.60	18.21	18.06	18.99	18.06	18.07	18.30
4	24.21	23.77	23.40	23.93	23.83	21.91	22.72	23.89	22.93	22.86
5	18.86	19.93	20.11	17.74	19.16	19.30	18.91	19.70	19.82	19.43
6	19.53	19.32	19.34	19.49	19.42	19.89	19.32	19.54	19.16	19.48
7	18.46	18.11	19.75	19.50	18.96	19.10	18.53	17.88	18.73	18.56
8	23.90	24.05	23.12	23.94	23.75	23.23	23.57	23.80	23.57	23.54

ferences in the results for the oil contents obtained by grinding with plate settings of 0.004, 0.008, and 0.012 inch, but the results on samples ground with a plate setting of 0.020 inch are significantly lower. It is notable that the moisture content of the samples varied from 7.8% to 40.4% and that irrespective of moisture content all of the samples could be ground and extracted satisfactorily.

Data are given in Table II for replicated analyses of six samples of two series each which were drawn from original samples of tung fruit of high (40.4%) and low (9.2%) moisture, respectively. After the grinding of the original samples of fruit in a Bauer laboratory mill with a plate setting of 0.008 inch and thoroughly mixing, five-gram portions were taken for analysis. The results in Table II show that a five-gram sample of the finely ground whole fruit is large enough to give satisfactory agreement in replicated analyses. It is known from other work (4) that the variation between samples of 200 fruits from the same batch of original fruit is much greater than the variation between replicated five-gram samples of ground fruit found here.

TABLE II

Percentage of Oil Extracted From Five-Gram Replicates of Ground Whole Tung Fruit. Calculated to Dry Basis

Sample A (Moisture 40.4%)	Sample B (Moisture 9.2%)
24.16	23.78
24.04	23.94
24.13	24.04
23.97	23.85
23.97	23.78
24.04	23.87
Av. 24.05	Av. 23.88

Table III shows a comparison between the percentages of oil obtained by the component and the whole fruit method of analysis. Table IV shows the corresponding individual and average figures for the moisture content obtained on four sub-samples of 50 fruits each which were analyzed by each method.

Although the individual values for replicated determinations of oil content show appreciable variation the averaged values obtained by application of the two methods of analysis agree reasonably well in most cases.

In the case of the moisture content the results of replicated analyses show equally divergent values which is also true of the averaged values for the two methods.

When the percentage of oil is calculated to a moisture-free basis, the results reflect the differences resulting from lack of uniformity in the values for the moisture content obtained in the two methods rather than in the extractable oil content.

In the component method of analyses the moisture content does not enter into the calculation of the percentage of oil in the fruit. In the whole fruit method the moisture content enters into the calculation of the oil content only to the extent that the change in moisture occurring during grinding in the Bauer mill is used to calculate the oil content to the as-received basis. However as long as the same method is used for determining the moisture content before and after grinding, any error from this source is negligible, but in calculating the oil content to a moisture-free basis, significant differences may occur as a result of the differences in the values obtained in the moisture determination.

The question has often been raised as to whether or not all of the oil could be extracted from wet samples with any given analytical method of analysis. A series of oil determinations was made on a single batch of fruit over a period of several months during which the moisture content decreased from 46.4% to 9.1%.

Several hundred pounds of fresh, very moist tung fruit were harvested early in October from adjacent trees which had been grafted from the same parent tree. This sample was passed repeatedly through a riffle and ultimately divided into sub-samples of about 50 fruits each, which were stored in the basement of a brick building. At intervals four sub-samples were analyzed by each of the aforementioned methods with the results shown in Table V. Samples analyzed by the whole fruit method are not given for the first three dates since mechanical difficulties involving plate alignment and direction of rotation of

TABLE IV

Comparison of Percentages of Moisture Obtained by Whole Fruit and Component Methods of Analysis of Tung Fruit

Sample No.	Component method					Whole fruit method				
	Replicates				Av.	Replicates				Av.
1	28.68	29.91	30.15	30.60	29.84	29.68	29.24	28.08	29.14	29.04
2	33.60	32.48	33.26	34.00	33.34	31.14	32.52	30.32	33.80	31.94
3	28.21	28.27	27.34	26.78	27.65	25.76	25.30	25.60	26.70	25.84
4	9.14	9.09	8.89	9.32	9.11	9.12	8.86	8.86	9.96	8.95
5	20.31	18.59	18.83	20.79	19.63	18.50	18.58	17.90	18.32	18.32
6	14.76	14.49	14.82	14.47	14.64	12.40	12.70	13.02	12.90	12.76
7	20.87	21.62	19.91	19.52	20.48	19.10	18.53	17.88	18.73	18.56
8	9.03	8.92	9.03	8.94	8.98	9.14	9.18	8.94	8.74	9.00

TABLE V

Percentage of Oil Obtained by the Component and Whole Fruit Methods of Analysis on Same Sample of Tung Fruit [a]

Date	Moisture [b] %	Component method					Whole fruit method				
		Replicates				Av.	Replicates				Av.
Oct. 14	46.4	26.76	25.76	26.82	26.47	26.45	.....	.....	.....	.....	.....
Oct. 19	31.6	26.44	27.33	26.67	26.49	26.73	.....	.....	.....	.....	.....
Oct. 25	14.2	26.27	26.10	25.28	26.48	25.78	.....	.....	.....	.....	.....
Nov. 3	11.4	26.53	25.97	26.31	25.92	26.18	25.47	24.00	25.48	25.77	25.18
Nov. 16	9.6	26.03	26.79	24.96	26.43	26.05	25.64	26.50	25.56	26.23	25.98
Dec. 13	8.7	25.84	25.05	26.59	25.79	25.82	25.22	24.52	24.02	24.47	24.56
Feb. 15	9.1	26.65	26.15	25.81	26.26	26.22	24.11	24.93	26.21	25.19	25.11
Average	....					26.18					25.21

[a] Percentage of oil calculated to moisture-free basis.
[b] Determined by the component method.

the mill were encountered, and the results were not considered to be reliable.

In the whole fruit procedure the moisture content was determined only on the finely ground material. Since some moisture is lost during this operation, it is not feasible to calculate the results to the original basis. Therefore comparisons can be made between the methods only on the moisture-free basis. It can be seen from the figures in Table III that the average percentages of oil obtained by any one method are quite consistent, but those for the component method are higher than those for the whole fruit method. However the values for the moisture determinations by the two methods differ significantly and probably account for the differences in the apparent oil content calculated to a moisture-free basis.

The data in Table V show that results by the component procedure are reproducible over a wide range of moisture contents (9% to 46%). In this method the sample is reground after partial extraction, and in the case of very moist samples (kernels containing more than 10% moisture) it is necessary partially to dry the partially extracted sample before regrinding, but it is apparent that with careful work all of the oil can be extracted by this method irrespective of the moisture content of the original sample.

The data in Table V also show that the results obtained by the whole fruit method are reproducible over the limited moisture range of 8% to 11%. Moreover the data in Tables III and IV show that samples containing up to 33% moisture can be successfully analyzed by this method.

Summary

The effect of moisture content and fineness of grinding on the percentages of oil extracted by the whole fruit method were investigated and the results compared with those obtained by the component method.

The spacing of the plates on the Bauer laboratory mill used for grinding whole tung fruit for oil analysis was found not to be critical within certain narrow limits. No differences were found in the oil content when samples of fruit were ground with plate spacings from 0.004 inch to 0.012 inch, but the results were lower with plate spacings of 0.020 inch.

No difference was found in the percentages of oil obtained by the component and the whole fruit methods when the results were calculated on the basis of the original moist sample and no correction had to be applied in the calculation of the results by the whole fruit method.

The average percentage of moisture obtained by the two methods differ, consequently care must be used in comparing oil contents calculated to a moisture-free basis since the differences in moisture content will be reflected in the values for the apparent oil content.

Careful analyses of tung fruit by either the whole fruit or the component method yield reliable results over a wide range of moisture content although in the case of the component method unusually wet kernels (above 10% moisture) must be partially dried before regrinding in a mortar and pestle during the extraction operation.

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[Received January 23, 1950]