

# Studies on the Refractive Index and Dispersion of American Tung Oil<sup>1</sup>

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BECAUSE tung oil has a much higher index of refraction than any other common oil, the refractive index is included in its specifications. The refractive dispersion of tung oil is also much higher than that of any other common oil and has been used as a measure of purity. With modern precision refractometers, designed for use with sodium and mercury-vapor lights, the determination of the dispersion requires very little more time and effort than does the determination of the refractive index alone. Considerable work has been done on the dispersion of Chinese tung oil for the H and C lines of hydrogen (1, 4, 5, 10, 12, 16, 19, 21), but little work has been reported on the dispersion of American tung oil. Chinese tung oil usually consists of mixtures of oil from the two species, *Aleurites montana* and *Aleurites fordii*, while the American tung oil comes from the latter exclusively. This study of the refractive index and dispersion of American tung oil, for the D line of sodium and the g line of mercury, was undertaken to see if additional worth-while information could be obtained from a measurement of dispersion.

## Equipment and Materials

The refractometer used for this work was a Bausch and Lomb Precision Refractometer introduced in 1938 (3), which was equipped with sodium and mercury-vapor lamps. The mercury lamp was used with Wratten Filter No. 50 to isolate the mercury g line (4358 Å), and all dispersions were measured between this line and the sodium D line (5890 Å). In this particular refractometer the refractive index can be measured either by transmitted or reflected light. In practice it was found desirable to set the equipment up with the sodium light in position for reading the refractive index by transmitted light and the mercury light in a position for reading the refractive index by reflected light. Both lights were left on, and, after reading one index, the other was read simply by flipping the shutter, thus avoiding the trouble of having to readjust the lights for each separate reading. Before taking a reading, water was circulated through the refractometer until the temperature became constant within .1°C. The temperature correction factors for tung oil were determined to be .000386 for the sodium line and .000415 for the mercury line, and these were used to correct the refractive indices to 25°C. A correction factor of .00038 was used for both lines on other oils. Corrections were small, since the temperature was never more than one or two degrees from 25°C.

Specific dispersion is calculated from the formula

$$\frac{(N_g - N_D) \times 10^4}{d} \text{ where } N_g \text{ and } N_D \text{ are the refractive}$$

indices for the g and D lines respectively and d is the density. In this study dispersion is reported as  $(N_g - N_D) \times 10^4$  to simplify the work and because changes in dispersion are so great relative to the changes in

density. In routine work it would be much simpler if the refractive index and dispersion could be used as determined without the necessity for determining the density to calculate the specific values.

The tung oil used in these studies was American, obtained by mechanical pressing in commercial screw presses. The other oils were commercial products bought in the open market or obtained directly from a company handling them. The glycerol and glycerides were Eastman C. P. chemicals.

## Refractive Indices and Dispersions of Tung Oil and Related Substances

In Table I are given the refractive indices and dispersions of samples of tung, oiticica, and several other oils, and also of several chemicals of interest in connection with a study of tung oil. An examination of the table shows that the refractive index and dispersion of tung oil are far above those of any other oil listed with the exception of oiticica oil. While the refractive index of the samples of oiticica oil given here are below the minimum of the A.S.T.M. specifications for tung oil, the history of these samples is not well known, and it is possible that some samples of oiticica oil will have an index as high as the minimum specified for tung oil.

In none of the references consulted was an explanation of the high dispersion of tung oil suggested. During the last 10 years much work has been done on the specific dispersion of hydrocarbons (2, 6, 8, 9, 13, 17, 18, 20), and this work has brought out very clearly that the value of the specific dispersion of hydrocarbons depends largely on their structure. The value for a saturated hydrocarbon is very nearly constant regardless of its molecular weight. It has long been known that formation of double bonds increases both the dispersion and the refractive index, and that conjugation exalts the increase in both. Tung oil contains about 80% eleostearic acid which has three conjugated double bonds. Both the high refractive index and the high dispersion of tung oil are obviously caused by the conjugated double bonds of the eleostearic acid.

Oiticica oil contains about the same amount of licanic acid that tung oil contains of eleostearic acid. The only difference between licanic and eleostearic acid is that in the former the two hydrogens or carbon atom No. 4 are replaced by an oxygen atom. Thus the conjugated double bonds of licanic acid would also explain the high refractive index and dispersion of oiticica oil.

The specific dispersion of hydrocarbons as measured between the D and the g lines is 122.5, corresponding to a dispersion of  $.935 \times 122.5$  or 114.5 as measured for a saturated hydrocarbon of the same density as tung oil. Grosse and Wackher (9) have shown that the introduction of oxygen into a hydrocarbon lowers the dispersion. The triglycerides are hydrocarbons except for the oxygen at the ester linkages. The presence of the oxygen lowers the dispersion of triacetin to 94, for the D and g lines, and, as

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the length of the saturated acid radical increases, the depression resulting from the oxygen decreases, the dispersion of tristearin being 101. (The dispersion of the long chain saturated triglycerides could not be determined with a high degree of accuracy because it was necessary to determine the refractive indices slightly above their melting points and correct the

TABLE I  
Refractive Indices and Dispersions of Tung Oil and Related Substances

Substance	$n_D$	$n_g$	Dispersion ( $\times 10^4$ )
Tung Oil, Sample No. 1.....	1.5173	1.5453	280
Tung Oil, Sample No. 2.....	1.5180	1.5462	282
Tung Oil, Sample No. 3.....	1.5178	1.5452	274
Tung Oil, Sample No. 4.....	1.5186	1.5466	280
Tung Oil, Sample No. 5.....	1.5170	1.5448	278
Tung Oil, Sample No. 6.....	1.5188	1.5471	283
Oiticica oil, No. 1, heat treated.....	1.5118	1.5344	226
Oiticica oil, No. 2, heat treated.....	1.5110	1.5334	224
Oiticica oil, No. 3, not heat treated.....	1.5130	1.5375	245
Linseed oil.....	1.4786	1.4917	131
Cottonseed oil.....	1.4702	1.4820	118
Olive oil.....	1.4670	1.4786	116
Mineral oil.....	1.4803	1.4909	106
Glycerol.....	1.4668	1.4762	94
Monoacetin.....	1.4500	1.4596	96
Diacetin.....	1.4423	1.4519	96
Triacetin.....	1.4288	1.4382	94
Tributyrin.....	1.4338	1.4432	94
Tricaproin.....	1.4405	1.4501	96
Tricaprylin.....	1.4459	1.4558	99
Trilaurin.....	1.4531	1.4630	99
Trimyristin.....	1.4555	1.4654	99
Tripalmitin.....	1.4572	1.4672	100
Tristearin.....	1.4587	1.4688	101

readings to 25°C., and there is a slight difference in the correction factors for the sodium and mercury lines. The factors determined for trimyristin were also used for tripalmitin and tristearin.) The dispersions of mineral, olive, cottonseed, and linseed oils were 106, 116, 118, and 131, respectively, which values are in fair agreement with a dispersion of 114.5 for saturated hydrocarbons and a value of about 100 for the glyceride of the saturated 18-carbon stearic acid. The elevation of the dispersions of the three

TABLE II

Per Cent Tung Oil in Tung Oil-Linseed Oil Mixtures Calculated From the Refractive Indices and Dispersions

Actual % of Tung Oil	5890 Å Line		4358 Å Line		Dispersion Disp. % Oil ( $\times 10^4$ ) Cal.
	R.I. at 25°C.	% Oil Cal.	R.I. at 25°C.	% Oil Cal.	
0.....	1.4786	0	1.4917	0	131 0
20.....	1.4864	20.2	1.5023	19.8	159 18.8
40.....	1.4941	40.1	1.5131	40.0	190 39.7
60.....	1.5018	60.0	1.5238	60.0	200 59.8
80.....	1.5096	80.1	1.5345	79.9	249 79.2
85.....	1.5115	85.0	1.5372	84.9	257 84.6
90.....	1.5134	89.9	1.5398	89.8	264 89.3
100.....	1.5173	100.0	1.5453	100.0	280 100.0

vegetable oils above that of the glyceride of the saturated 18-carbon acid can be accounted for by known double bonds in these oils. Linseed oil has almost as much unsaturation as tung oil; yet the dispersion of linseed oil is only 131 as compared to 280 for tung oil, the difference being that the unsaturation is conjugated in the tung oil but not in the linseed oil.

#### Analysis of Mixtures of Tung Oil with Other Oils By Refractive Indices and Dispersions

If two liquids differ appreciably in refractive index the composition of a mixture of them can be calculated from the indices of the mixture and of the two

components, provided there is no reaction or volume change upon mixing.

By the law of mixtures (7),

$$(1.) (N^0 - 1)V^0 = (N^1 - 1)V^1 + (N^2 - 1)V^2$$

where  $N$  is the refractive index,  $V$  is volume and the superscripts 0, 1, and 2 refer to the mixture and to the pure first and second components respectively. This law can be simplified to

$$(2.) V\% = \frac{100(N^0 - N^2)}{N^1 - N^2}, \text{ where } V\% \text{ is the volume per cent of the first component.}$$

If formula 2 is true for refractive indices, it can also be proven that it is true for dispersion, that is,

$$(3.) V\% = \frac{100(D^0 - D^2)}{D^1 - D^2}$$

where  $D$  refers to dispersion (i.e., the difference between the refractive indices measured at two different wave lengths), and the superscripts have the same meanings as under (1.).

Formulas 2 and 3 give the volume percentages, but it is simpler to work with weight percentages ( $P$ ). Volume percentages can be converted to weight percentages by the following formula:

$$(4.) P = \frac{V^1 G^1}{V^1 G^1 + V^2 G^2} \text{ where } G^1 \text{ and } G^2 \text{ refer to the specific gravities of the first and second components respectively.}$$

TABLE III

Per Cent Tung Oil in Tung Oil-Cottonseed Oil Mixtures Calculated From the Refractive Indices and Dispersions

Actual % of Tung Oil	5890 Å Line		4358 Å Line		Dispersion Disp. % Oil ( $\times 10^4$ ) Cal.
	R.I. at 25°C.	% Oil Cal.	R.I. at 25°C.	% Oil Cal.	
0.....	1.4702	0	1.4820	0	118 0
20.....	1.4796	20.0	1.4947	20.1	151 20.4
40.....	1.4890	39.8	1.5073	39.9	183 40.1
60.....	1.4985	59.7	1.5198	59.4	213 58.4
80.....	1.5081	79.6	1.5331	79.9	250 80.8
85.....	1.5105	84.6	1.5362	84.7	257 85.0
90.....	1.5130	89.7	1.5394	89.6	264 89.2
100.....	1.5180	100.00	1.5462	100.00	282 100.00

Various mixtures of tung oil were made with linseed, olive, cottonseed, and mineral oils. The refractive indices of the mixtures and of the pure components were determined for the 5890 and the 4358 lines. The compositions calculated from the refractive indices and the dispersions are tabulated in Tables II to V. The compositions were calculated by formulas 2, 3, and 4.

An inspection of the tables shows that any appreciable adulteration of tung oil by any of the four oils studied could easily be detected from either one of the refractive indices or from the dispersion. The values for the calculated compositions are close to the actual compositions except for the mixtures of tung and mineral oils. Apparently something takes place upon mixing tung and mineral oils that causes a slight error in analyses by the refractive indices.

For the mixtures of tung oil with other vegetable oils, the slight differences of the calculated values from the actual values are probably caused either by incomplete mixing of the components, or by slight errors in reading the refractive indices, as the errors

seem to be random. The fact that the composition of these mixtures can be calculated so closely from the refractive indices indicates that there is little change in volume and little reaction when the components are mixed.

Because of the greater difference between the refractive index of tung oil and that of any other oil when both are measured by the mercury line, theoretically the analyses should be more accurate for the mercury line than for the sodium line.

TABLE IV

Per Cent Tung Oil in Tung Oil-Olive Oil Mixtures Calculated From the Refractive Indices and Dispersions

Actual % of Tung Oil	5890 Å Line		4358 Å Line		Dispersion	
	R.I. at 25° C.	% Oil Cal.	R.I. at 25° C.	% Oil Cal.	Disp. (× 10 <sup>4</sup> )	% Oil Cal.
0.....	1.4670	0	1.4786	0	116	0
20.....	1.4768	19.9	1.4916	19.9	148	19.9
40.....	1.4868	39.9	1.5048	39.8	180	39.6
60.....	1.4969	60.0	1.5182	59.9	213	59.7
80.....	1.5071	80.1	1.5315	79.7	244	78.4
85.....	1.5096	85.0	1.5351	85.0	255	85.1
90.....	1.5122	90.1	1.5384	89.9	262	89.2
100.....	1.5173	100.00	1.5453	100.00	280	100.00

Any error in refractive index is magnified when it is used in calculating the composition from the dispersion. An error of 0.0001 in reading the refractive index would cause an error of about 0.6% in calculating the composition of the mixture from the dispersions, but the error would be less than half this amount if the composition is calculated from the refractive indices. This emphasizes the necessity for using a refractometer of high accuracy when analyzing mixtures by dispersion. An accurate control of temperature while taking the readings is also necessary.

TABLE V

Per Cent Tung Oil in Tung Oil-Mineral Oil Mixtures Calculated From the Refractive Indices and Dispersions

Actual % of Tung Oil	5890 Å Line		4358 Å Line		Dispersion	
	R.I. at 25° C.	% Oil Cal.	R.I. at 25° C.	% Oil Cal.	Disp. (× 10 <sup>4</sup> )	% Oil Cal.
0.....	1.4803	0	1.4909	0	106	0
20.....	1.4870	19.2	1.5010	18.6	140	20.3
40.....	1.4940	38.5	1.5114	37.7	174	40.0
60.....	1.5016	58.4	1.5224	58.0	208	59.4
80.....	1.5091	77.5	1.5332	77.4	241	77.7
85.....	1.5115	83.8	1.5368	83.6	253	84.3
90.....	1.5135	88.8	1.5397	88.7	262	89.2
100.....	1.5180	100.00	1.5462	100.00	282	100.00

Although the composition of a mixture of tung oil with another oil can be calculated more accurately from the depression of the refractive index if the kind and refractive index of the other oil are known, when the other oil is not known the composition can be calculated more accurately from the dispersion, because the dispersions of non-conjugated oils are less variable than their refractive indices (see Table VI). The dispersion of tung oil is about 280 as compared to an average of 115 for oils that would be used for adulteration. Thus, dividing the depression of the dispersion by the difference between 280 and 115 gives approximately the proportion of adulteration on the assumption that the adulterating oil has a dispersion higher than 115 the calculated value will be too low, and vice versa.

TABLE VI

Changes in Refractive Index and Dispersion of Tung Oil for Each 1% Dilution With Various Oils

Diluting Oil	5890 Å Line Ref. Index	4358 Å Line Ref. Index	Dispersion
Linseed.....	.000387	.000536	.000149
Cottonseed.....	.000478	.000642	.000164
Olive.....	.000503	.000667	.000164
Mineral.....	.000377	.000553	.000176

Partially polymerized tung oil will also give lower refractive index and dispersion values than fresh oil, but in any case the oil would not be classified as pure, fresh tung oil.

### Effect of Heating Tung Oil on Its Refractive Index and Dispersion

Batches of tung oil were heated at various temperatures controlled to within 0.5°. At regular intervals samples were withdrawn on which the refractive indices for the mercury and sodium lines, and in some cases the viscosities, were determined. Batches were heated at 200°, 210°, and 232°C. Results at the different temperatures were very similar except that the higher the temperature the more rapidly the changes took place. Only the data and curves for one batch of tung oil (heated at 210°C.) are given in Table VII and in Figures 1 and 2. For comparison, a batch of linseed oil was heated at 200°C., and changes in the refractive index were followed. Data for the linseed oil are given in Table VIII.

As shown in Table VIII, the refractive index of linseed oil increases upon heating. It was thought possible that the dispersion would also increase on heating because of the conversion of non-conjugated unsaturation to conjugated unsaturation. However,

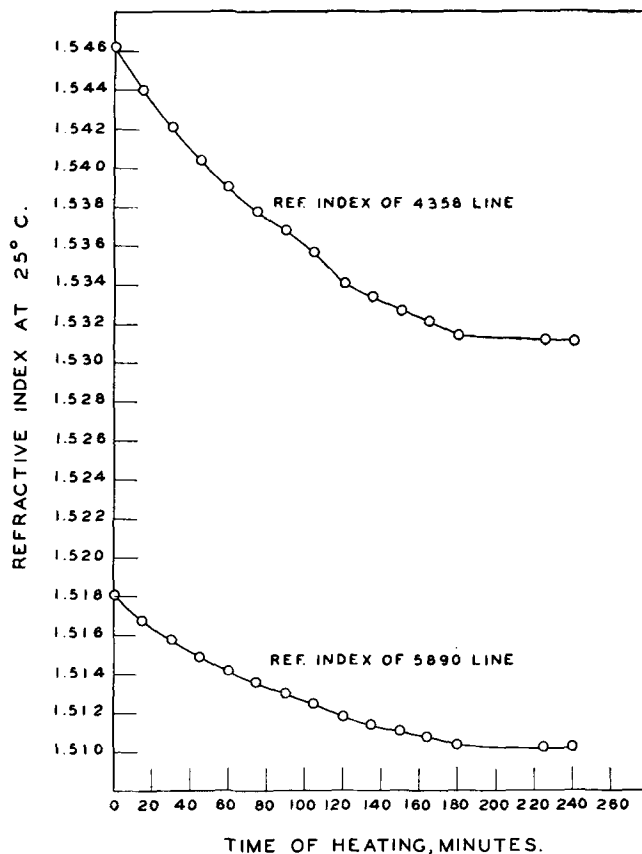


FIG. 1. Changes in refractive index of tung oil on heating at 210°C.

TABLE VII  
Effect of Heating Tung Oil at 210°C. on Its Refractive Index,  
Dispersion, and Viscosity

Minutes Heated	5890 Å (D Line) Ref. Index, 25°C.	4358 Å (g Line) Ref. Index, 25°C.	Dispersion ( $\times 10^4$ )	Viscosity, Poise
0.....	1.5181	1.5462	281	2.00
15.....	1.5168	1.5440	272	2.75
30.....	1.5158	1.5421	263	3.70
45.....	1.5149	1.5404	255	5.50
60.....	1.5142	1.5390	248	6.27
75.....	1.5136	1.5378	242	8.84
90.....	1.5130	1.5368	238	10.70
105.....	1.5125	1.5357	232	12.90
120.....	1.5118	1.5341	223	27.00
135.....	1.5114	1.5334	220	36.20
150.....	1.5111	1.5327	216	46.30
165.....	1.5108	1.5322	214	63.40
180.....	1.5104	1.5315	211	148.00
225.....	1.5103	1.5312	209	*
240.....	1.5103	1.5311	208	*
255.....	gelled	.....	.....	.....

\* No facilities for determining such high viscosities.

according to these data, there were no significant changes in the dispersion with the heating. The history of this sample of linseed oil is not well known so the possibility that there is an increase in dispersion upon heating raw linseed oil is not precluded by the data on this single sample.

An examination of Table VII and the curves in Figure 1 shows that, contrary to the case of linseed oil, both the refractive index and dispersion of tung oil fall rapidly and continuously up to the point of gelation. The decrease in the refractive index for the mercury line during heating is much greater than that for the sodium line. This is necessarily true because the dispersion is decreasing and the change in the refractive index for the shorter wave length is equal to that for the longer wave length plus any change in dispersion. For this reason changes in refractive index that are accompanied by changes in dispersion can be determined more accurately by using the mercury line.

In general, the changes in the two refractive indices and the dispersion appear very similar. Correlations of the two indices with each other and with the dispersion were calculated from many other data, in addition to those in Table VIII, with the following results: The correlation coefficient ( $r$ ) for the two indices with each other was .994; that for the 5890 index with the dispersion was .973; and that for the 4358 index with the dispersion was .993. These correlations are so high that no useful additional information can be obtained by determining a second index of refraction and the dispersion on samples of tung oil subjected to heat treatment, if the sample

TABLE VIII  
Effect of Heating Linseed Oil at 200°C. on Its  
Refractive Index and Dispersion

Minutes Heated	5890 Å Line Ref. Index, 25°C.	4358 Å Line Ref. Index, 25°C.	Dispersion ( $\times 10^4$ )
0.....	1.4789	1.4918	129
15.....	1.4789	1.4920	131
25.....	1.4790	1.4922	132
45.....	1.4791	1.4922	131
60.....	1.4791	1.4923	132
100.....	1.4792	1.4923	131
140.....	1.4793	1.4924	131
180.....	1.4793	1.4925	132
220.....	1.4794	1.4925	131
320.....	1.4795	1.4926	131
420.....	1.4796	1.4928	132
540.....	1.4797	1.4929	132
660.....	1.4798	1.4930	132
780.....	1.4799	1.4932	133

is known to be pure tung oil. Under these conditions it would be possible to calculate the other index and the dispersion quite accurately if either index is known.

That there is not a high correlation between the refractive index and dispersion in general is shown by the fact that, in the case of the saturated hydrocarbon series, as the molecular weight increases the dispersion remains practically constant, while the refractive index increases (20). No doubt there is a high correlation between increase in refractive index and increase in dispersion due to unsaturation, but the relative values of these increases would vary, depending upon whether the unsaturation is conjugated or non-conjugated.

Figure 2 shows that during the heating of tung oil viscosity increases as refractive index drops and that plotting viscosity against refractive index gives a

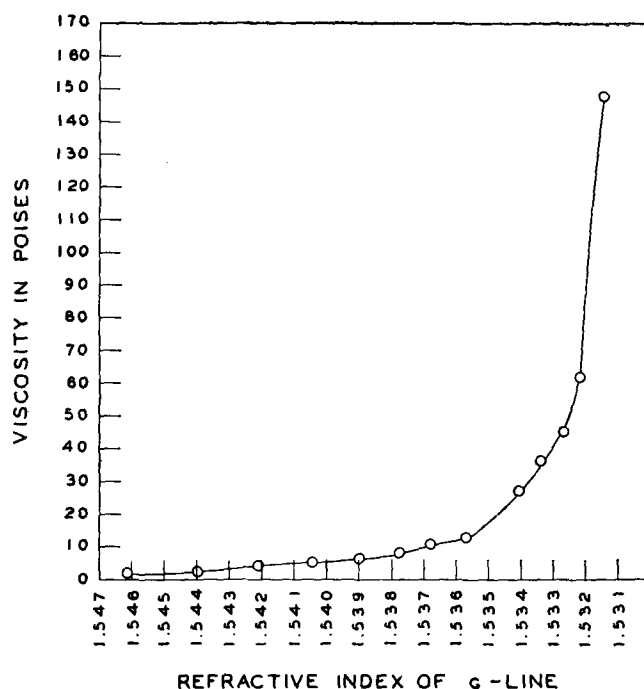


FIG. 2. Changes of viscosity and refractive index during heating of tung oil at 210°C.

smooth curve. This suggests the possibility of using the refractive index as a guide in controlling the bodying of tung oil, but whether the control by refractive index would have any advantage over cooking to "stringing" would have to be determined by trial under factory conditions.

Gelation of the tung oil tested took place after the refractive index (5890 Line) had dropped to 1.5097-1.5103 and the dispersion had dropped to 204-210. These values are so high as to indicate that much of the conjugated unsaturation in tung oil is still unreacted at the point of gelation.

#### Correlation of the Refractive Index with the Diene and Iodine Numbers of Tung Oil

Since increase in the eleostearic acid content of tung oil increases the diene number, the iodine number, and the refractive index, a positive correlation would be expected for the refractive index and the diene number, and for the refractive index and iodine

number, as shown by Frahm and Koolhaas (4) for the oil of *Aleurites montana*.

The correlation coefficient (4) was calculated for refractive index (sodium line) with the diene number (14) and was found to be .83, a value which is highly significant since a value of only .28 is required for significance at a probability of 99 in 100. This means that .70 ( $r^2$ ) of the variation in diene number can be accounted for by variation in the refractive index, and vice versa.

Calculation of the correlation coefficient ( $r$ ) for refractive index (sodium line) with the iodine number (Wijs) for 180 samples gave a value of only .357. While this correlation is highly significant, it is not high enough to predict the iodine number from the refractive index. The iodine number of these 180 samples varied from 161.1 to 166.3, and the refractive index from 1.5144 to 1.5190. An inspection of the data showed that the relation between the refractive index and iodine number was not the same for two groups separated on a date basis. When the correlation coefficient was worked out for the two groups separately, it was found to be .49 for one and .52 for the other. The iodine number of tung oil is notoriously difficult to determine since only two of the three double bonds react readily with iodine. The extent to which the third reacts depends upon the conditions under which the determination is made so it is difficult to get reproducible results. Some slight change in conditions, such as temperature or strength of solutions, probably accounts for the differences in these two groups separated on a date basis.

The refractive index can be used for the determination of the iodine numbers of linseed and soybean oils (11, 12, 15, 22, 23), but there are theoretical reasons why such a close correlation between refractive index and iodine number should not be found in tung oil as in linseed oil. In the latter case, the increase in the refractive index above that of the completely hydrogenated oil is almost proportional to the amount of unsaturation, because the amount of conjugated unsaturation is negligible. In addition to eleostearic acid, which contains three conjugated double bonds, tung oil contains a small proportion of non-conjugated unsaturation. The ratio of increase in refractive index with increase in unsaturation to the increase in iodine number is much greater for conjugated than for non-conjugated saturation (because of the exalting effect of conjugation on the refractive index and because the third double bond in eleostearic acid does not readily take up iodine). Hence if there is a variation in the proportion of conjugated to non-conjugated unsaturation in tung oil, a lower correlation between refractive index and iodine number would be expected than for linseed oil. However, the proportion of eleostearic acid in tung oil is so high that the iodine number is probably closely correlated with the refractive index if conditions are controlled carefully enough during determination of iodine number.

### Conclusions

1. Tung oil has a refractive index and a dispersion so far above those of any other common oil that both are valuable criteria for identification purposes. With proper equipment the dispersion, in addition to the refractive index, can be determined with little extra

effort and would confirm the conclusions drawn from the refractive index.

2. Mixtures of tung oil with another vegetable oil (except oiticica and other rare conjugated oils) can be analyzed to within 0.5% from the refractive index for either the sodium or the mercury line if the refractive indices of the separate oils are known. The mixtures can be analyzed from the dispersion to within about 1% of the correct composition if the dispersions of the separate oils are known. If the adulterating oil is not known the adulteration can be more closely estimated from the depression of the dispersion than from the depression of the refractive index.

3. When tung oil is bodied by heat the refractive indices for the sodium and mercury lines and the dispersion fall rapidly and continuously to the point of gelation, but the changes are so similar that no worth-while additional information is obtained by determining more than one refractive index. The fact that refractive index decreases as viscosity increases suggests the use of the refractive index in controlling the bodying of tung oil.

4. Other things being equal, the refractive index for the mercury line should give more accurate information on tung oil than that for the sodium line because of the greater changes in the refractive index for the mercury line upon adulteration or heating.

5. A correlation coefficient of 0.83 was found for refractive index with the diene number of tung oil. A lower correlation coefficient was found for refractive index with the iodine number, but the latter would probably be higher if a more accurate method for the determination of the iodine number of tung oil were available.

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