the evolution and scrubber flasks should extend nearly to the bottom of the flasks and be bent at right angles and parallel to the sides of the flask.

Condenser, F, a 12-in. or longer spiral condenser of sufficient bore so the condensate will not readily close it.

Measuring Burette, H, a 10 c.c. burette calibrated to 0.1 c.c. and carrying a bulb, I, approximately 100 c.c. capacity, at the lower end.

The stoppers used should be of a good grade of rubber and should have been thoroughly cleaned free from any surface sulfur and should be given a steam distillation in position for several hours before use on a sample.

Insulating the flasks and tubing to reduce condensation aids distillation and its control.

Determination:

Place 150 c.c. NaOH solution (about 46° Be') and several sticks of solid NaOH, to provide against dilution in the scrubber flask. Rinse out the condenser and burette with acetone. Attach a rubber tubing to the lower end of the burette, fill the burette and tubing with water and raise the outer end of the tubing so that the water level in the burette is near the top of the scale when the water is flowing to the drain from the automatic overflow, J. Be sure the connections are tight and that the tubing contains no air bubbles. Place the condenser in position so the lower end extends directly into the upper end of the burette just above the water level or connect to an adapter siphon, G, which discharges into the burette. The cooling water should be 15.5° C. or colder. Ice water may be desirable for low boiling hydrocarbons.

Weigh 100 ± 0.5 grams of the soap (cut into cubes of about 1 cm. edges) or 50 ± 0.3 grams of soap powder and transfer to the evolution flask. Add about 10 grams of gum arabic (commercial) and 100 c.c. of distilled water. Place the flask in position with 100 c.c. of 1:3 H_2SO_4 in a dropping funnel, C, carried in the stopper. Connect the steam, evolution, and wash flasks and condenser into position, making sure that the stoppers are tightly fitting and held in place by wiring. Rubber connections in the lines between the evolution flask and condenser should be avoided.

Add the acid to the sample slowly to avoid excessive frothing. While adding the acid, turn on the steam cautiously, so adjusting the pressure by a bleeder valve that just enough steam flows to prevent any liquid backing into the steam trap flask.

When all the acid has been added, turn on enough steam to cause brisk distillation, taking care that no liquid is carried over from the evolution and wash flasks and that the condenser water does not become warm.

Continue the distillation until there is no increase in the volume of the upper layer for 45 minutes or no small droplets can be noted in the condensate.

When distillation is completed, shut off and drain condenser water and allow the steam to heat up the condenser to drive out the last traces of volatile hydrocarbon. Shut off the steam as soon as vapor begins to issue from the lower end of the condenser. Immediately open the stopcock of the dropping funel to prevent caustic being drawn into the evolution flask.

Stopper the burette and allow its contents to come to room temperature or bring them to a definite temperature by placing the burette in a water bath held at 25° C. for one to two hours.

Read the volume of the upper layer to the nearest 0.01 c.c. The volume times the specific gravity equals the weight of the volatile hydrocarbon. The specific gravity should be determined at the temperature at which the volume is read. A small Sprengel tube made of 3 mm. glass tubing is convenient for this purpose.

Calculation:

$$\frac{\text{c.c. of volatile hydrocarbon} \times \text{specific gravity} \times 100}{\text{Wt. of sample}} = \% \text{ of volatile hydrocarbon}$$

For some samples the volatile hydrocarbon content may be so low that a larger sample than 50 or 100 grams is desirable. The size of the evolution flask may need to be increased if larger samples are used. The amount of water in the evolution flask and acid used should also be correspondingly increased.

THE COMPOSITION OF OITICICA OIL*

By R. S. McKINNEY
and
G. S. JAMIESON

IN the past there has been considerable confusion concerning the identity of the tree, the seeds of which yield oiticica oil, but it has been definitely established that the tree is Licania rigida of the family Rosaceae, which, as is well known, grows in northeastern Brazil. Circular No. 470 (1934) of the National Paint, Varnish and Lacquer Association, by Henry A. Gardner, contains much information in regard to the tree and discusses the commercial production of the oil, its properties and utilization in the manufacture of paint and varnish.

Until recently it was believed that the principal unsaturated constituent of this oil, known as couepic acid, was an isomer of elaeostearic acid. However, W. B. Brown and E. H. Farmer (Biochem. J. 29, p. 631, 1935), have shown that it is a gamma keto $\triangle^{9,11,13}$ octadecatrienoic acid ($C_{18}H_{28}O_3$) which melts at 74-75° C. They have suggested that from now on it be known as licanic acid because the oil is from the seeds of Licania rigida instead of from the tree Couepia grandiflora as formerly believed.

The present investigation was made on oils furnished by Dr. Gardner. One sample was raw oil

*Contribution from the Oil and Fat and Wax Laboratory, Bureau of Chemistry and Soils, United States Department of Agriculture.

and the other was oil which had been heated to about 225° C. for an hour to render it permanently liquid.

It was observed that upon making several determinations of the iodine number by the Hanus method the results were influenced to an unusual degree by the weight of oil taken, the quantity of Hanus reagent used, and the time allowed for the reaction. The results of these and other experiments subsequently made are given in Table I.

TABLE I.
Iodine Numbers by the Hanus Method.

Sample	Reagent cc.	Weight of oil, Grams	Time of Reaction, Hours	Iodine Numbers
Raw oil	25	0.1090	1	146.9
Raw oil	25	0.1383	1	122.0
Raw oil	25	0.1048	2	157.0
Raw oil	25	0.1103	2	150.0
Raw oil	50	0.1000	0.5	196.0
Raw oil	50	0.1074	0.5	192.0
Raw oil	50	0.1010	2	225.0
Raw oil	50	0.1008	2	227.0
Heated oil..	25	0.1000	1	144.5
Heated oil..	25	0.1500	1	109.6
Heated oil..	25	0.1068	2	144.5
Heated oil..	25	0.1160	2	136.2
Heated oil..	50	0.1049	0.5	170.4
Heated oil..	50	0.1168	0.5	170.0
Heated oil..	50	0.1029	2	195.0
Heated oil..	50	0.1069	2	195.0

For comparative purposes similar experiments were made with linseed and perilla oils using 25 and 50 cc. of the Hanus reagent. Doubling the quantity of the Hanus reagent gave an increase of 4.8 in the iodine number of linseed oil and 1.6 for the perilla oil.

Using Kaufmann's method thiocyanogen values of 76.2 for the raw oil and 80.1 for the heated product were obtained. On the other hand it will be seen in Table I that the heated oil gave iodine numbers below those for the raw oil. These results are interesting because they indicate that although the total unsaturation of the oil was lowered by the heat treatment a small portion of the licanic acid was affected in such a manner as to make it more reactive with the thiocyanogen radical than it was in the original unheated oil, in which but one of the three double bonds of the licanic acid took part in the reaction.

In order to determine the true iodine number of the oil, which could be used for calculating the proportions of the unsaturated acids, unsuccessful attempts were made to determine by analysis the extent of substitution, if any, of halogen after the oil was allowed to react for two hours with a large excess of Hanus solution. The cause for this difficulty was not investigated. As no evidence was obtained of the presence in the oil of any unsaturated constituent other than oleic and licanic acids, it was evident that if the quantity of oleic acid could be determined, the result, together with the thiocyanogen values, could be used for calculating the quantity of licanic acid in the oil. With this in mind the controlled oxidation of the saponified fatty acids by alkaline permanganate was made according to the method of Lapworth and Mottram (J. Am. Chem. Soc. 127, p. 1628, 1925). The quantity of dihydroxystearic acid finally isolated was equivalent to 6.2 per cent of oleic acid in the total fatty acids.

Considerable difficulty was experienced with the separation of the dihydroxystearic acid owing to the formation of sticky substances during the alkaline permanganate oxidation which were probably due to the incomplete oxidation of the licanic acid. Experiments were then undertaken using much larger quantities of the one per cent solution of permanganate than that recommended by Lapworth and Mottram. This treatment prevented the formation of the sticky partial oxidation products. In cases in which known amounts of oleic acid were taken the yield of dihydroxystearic acid corresponded only to 78 per cent of the oleic acid. Applying this modified procedure to the saponified oiticica fatty acids and using the indicated correction gave 5.9 per cent of oleic acid as compared with 6.2 per cent obtained by the original method.

The quantity of licanic acid as glyceride (81.7 per cent) in the oil was calculated using the following equation: $86.8x + 86.04y = 7620$ (or $100 \times$ SCN value of oil), in which $x =$ the per cent of licanic acid glyceride and y that of oleic acid glyceride. 86.8 is the theoretical thiocyanogen value of licanic acid glyceride and 86.05 that of oleic acid glyceride.

From the percentages of licanic and oleic acid glycerides present, it was also calculated that the true iodine number of the sample of raw oiticica oil was 218. J. Van Loon and A. Steger (Rec. trav. chim. 50, 936, 1931) gave 231 as the iodine number of the oil which they investigated. It should be noted, however, that they were unable to detect the presence of any oleic acid in the oil. Apparently they considered that the 82.43 per cent of unsaturated acids which they found in the oil consisted only of what they called couepic acid.

The characteristics and composition of raw oiticica oil are given in Table II.

TABLE II.
Oiticica Oil.

Refractive index at 25° C.....	1.5145
Saponification value	192.6
Iodine number (calculated).....	218.0
Thiocyanogen value	76.2
Characteristics and Composition of	
Unsaponifiable matter, per cent..	0.57
Iodine number of unsaponifiable.	111.0
Saturated acids (Bertram method), per cent	10.7
Oleic acid, per cent	5.9
Licanic acid, per cent	78.2
Glycerides of saturated acids, per cent	11.2
Glycerides of oleic acid, per cent	6.2
Glycerides of licanic acid, per cent	81.7

REPORT OF THE OLIVE OIL COMMITTEE

By M. F. LAURO, Chairman

THIS committee reports progress. A questionnaire has been sent out to sound out each member as to the advisability of including certain values in the list of specifications for olive oil and olive oil foots.

We have about completed a tentative classification for oils of the Italian and Spanish type, but work on the exceptional oils like those from Tunis and Dalmatia, which fall outside that category, depends to a large extent on our securing authentic samples from those countries. This is necessarily a slow process. A few have been received and analyzed in advance by the chairman. Since they have shown no material variations from the constants of the group already listed, these samples have not been distributed to the members.

We are of course going ahead with our schedule of drafting the proper specifications and trust to have something substantial to report at the Spring Meeting in 1936.