the two commercial resins are about equally resistant to water. Only films from resins modified with pure and distilled cyclic acids and with safflower fatty acids remained resistant to 1% NaOH for 14 days. The film from the safflower fatty acid-modified resin retains its resistance (14 days) even with 5% NaOH. Although films from the two resins modified with pure and distilled cyclic acids are less resistant than this one, they are considerably more resistant than any of the others to 5% NaOH.

In summarizing results of evaluations on drying times, hardness, and chemical resistance, each resin was ranked for each test and individual rankings for each resin were added. The order of these sums is shown in Table IV. This summary shows that modification of the alkyd resins with pure cyclic acids definitely improves properties over those obtained with the other modifiers in air-dried films, and gives an almost equal improvement in baked films. Use of distilled cyclic acids also improves resins although not to the same extent as do pure acids. Both the pure and distilled cyclic acids give resins superior to commercial oil-modified resins under conditions of our tests. Crude cyclic acids are good in air-dried films but are poor in baked films.

Tests reported in this paper are preliminary evaluations and are not intended to be exhaustive. They represent a limited number of tests on a single formulation. Modification either of the formulation or of the resin, perhaps by a process such as styrenation (5), might further enhance the good properties imparted by pure and distilled cyclic acids and might also improve the utility of crude acids in alkyd resin modification.

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REFERENCES

REFERENCES 1. Earhart, K. A., and B. Rabin, U. S. Patent 2,308,498, Jan. 19, 1943. 2. Friedrich, J. P., H. M. Teeter, J. C. Cowan, and G. E. McManis, JAOCS, 38, 329 (1961). 3. Kienle, R., U. S. Patent, 1.893.873, Jan. 10, 1933. 4. Monsanto Chemical Co., "The Chemistry and Processing of Alkyd Resins," 1952, p. 52. 5. Norris, W. C., Paint Oil Chem. Rev. 112, No. 7, 16 (March 31, 1949).

- 5. Norris, W. C., Paint Oil Chem. Rev. 114, 100, 1, 20 (1994).
 6. Privett, O. S., M. L. Blank, and W. O. Lundberg, JAOCS, 38, 27 (1961).
 7. Scholfeld, C. R., and J. C. Cowan, *Ibid.*, 36, 631 (1959).
 8. Schwab, A. W., H. M. Tecter, and J. C. Cowan, *Ibid.*, 36, 275 (1959).
 9. Swern, D., Ind. Eng. Chem., 47, 216 (1955).

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Analyses of Pecan, Peanut, and Other Oils by Gas-Liquid Chromatography and Ultra-Violet Spectrophotometry

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Comparative analyses of oils obtained from 12 varieties of pecans, 11 of peanuts, two of avocados and one each of citrus seed, Wesson oil, corn, and of lard by GLC and UV procedures showed good agreement except for Wesson oil, corn oil, and lard. Iodine values computed from GLC results checked Wijs values closely. Oleic acid ranged from 51 to 77% in pecans and from 43 to 64% in the peanut with linoleic acid in complementary percentages.

Level of palmitic acid in the peanut varied directly with level of linoleic acid, in the avocado it apparently varied directly with level of hexadecenoic acid while in the pecan the level of palmitic remained constant even though the level of unsaturated acids varied widely. Both peanuts and pecans were locally grown under similar environmental conditions.

NFORMATION on the composition of certain natural I oils was needed for an experiment designed to test cholesterogenic effect of oils in diets on rat blood serum. Analyses were made on the oils by methods of gas-liquid chromatography (GLC) and by ultraviolet (UV) spectrophotometry. Analyses by the two procedures were compared on oils expressed in the laboratory from 11 varieties of peanuts, 12 of pecans, 2 of avocado, and on market purchased samples of citrus seed oil, Wesson oil, corn oil, and lard.

Apparatus and Methods

Gas-liquid chromatograms were obtained with an A-100 Aerograph using a 4-filament thermal sensitive cell with an oven temperature of 200C and a gas flow rate of 60 ml/min of helium. One to 3 lambda, the volume depending upon the oleic acid content of the sample, of a 20% solution of the fatty acid methylesters (ld) in benzene was placed in the heated injec-

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tion chamber. Passage through a 5-ft. column packed with 10% Lac 446, a glveol adipate polymer, on 30-60 mesh firebrick gave sharply shaped curves for the methyl esters of the fatty acids with 14 to 20 carbons in their chains. The area under each curve was computed by multiplying the height of the peak by width at middle height and ascribed to each fatty acid as indicated by the time it left the column.

Iodine values were determined by the Wijs method (2), and the polyunsaturated acids by the UV spectrophotometric procedure (3).

Samples

The oil samples were chosen from 12 popular varieties of pecans grown in north-central Florida, and from 11 varieties of peanuts which have been or probably will be recommended to growers (4). The pecans were purchased from an Alachua County merchant who handled locally-grown nuts. The peanuts, which included Spanish, runner, and Virginia types, were all grown on the Florida Experiment Station farm in 1958 under the same supervision and under the same environmental conditions. The pecan and peanut oils were cold pressed in a Carver laboratory press. The 2 avocado samples, supplied by a grower in the Miami area, were dried and the oil extracted with hexane. The citrus seed oil was a commercial Florida product. The Wesson oil, corn oil, and leaf lard were purchased in local stores. The lard organoleptically showed a trace of rancidity. All fats were kept frozen under prepurified nitrogen until analyzed.

Results and Discussion

The GLC analyses are given in Table I. The analyses of oils from the 11 peanut and 12 pecan vari-

Item	Pal- mitic	Hexa- dece- noic	Ste- aric	Oleic	Lin- oleic	Lino- lenic	Arach- idic
Peanut ^a Spanish 18-38-47 N. Carolina 2 ^b Va. Runner G-26 Spanette Ga. Bunch 119-20 Va. 56R Early Runner Va. Bunch G2 Florigiant Dixie Runner Fla. 393-47	$\begin{array}{c} \% \\ 12.9 \\ 10.5 \\ 10.3 \\ 10.9 \\ 11.1 \\ 9.6 \\ 9.7 \\ 10.1 \\ 10.3 \\ 9.3 \\ 7.3 \end{array}$	% 0.2 T e 0.5 0.4 T 0.6 T 	$\begin{array}{c} \% \\ 4.5 \\ 4.3 \\ 4.7 \\ 3.7 \\ 4.1 \\ 4.4 \\ 3.1 \\ 3.7 \\ 4.1 \\ 3.1 \\ 3.1 \\ 4.3 \end{array}$	$\begin{array}{c} \% \\ 43.1 \\ 51.9 \\ 52.3 \\ 52.5 \\ 54.4 \\ 54.5 \\ 55.6 \\ 55.7 \\ 55.7 \\ 58.5 \\ 63.5 \end{array}$	$\begin{array}{c} \% \\ 32.5 \\ 26.2 \\ 25.1 \\ 25.7 \\ 23.2 \\ 23.8 \\ 24.7 \\ 23.7 \\ 22.9 \\ 22.4 \\ 17.7 \end{array}$		% 0.7 0.6 0.9 0.7 1.1 0.9 1.0 0.7 0.7 1.1
Pecan Moneymaker Mobile Big Z Randall Teche Frotscher Stuart Seedling 1 Seedling 2 Curtis Van Deman Success	$\begin{array}{c} 6.3 \\ 7.1 \\ 5.9 \\ 6.0 \\ 6.1 \\ 6.5 \\ 6.6 \\ 4.3 \\ 6.1 \\ 5.1 \\ 5.4 \end{array}$	0.2 T T T 0.4 T	$2.6 \\ 2.7 \\ 2.9 \\ 3.0 \\ 2.5 \\ 2.2 \\ 3.9 \\ 3.2 \\ 3.1 \\ 2.9 \\ 2.9 \\ 2.9$	51.0 56.8 59.7 61.3 63.4 63.9 68.8 69.2 71.0 71.8 74.6 76.5	37.8 31.4 30.3 28.2 25.7 25.5 21.0 21.2 18.5 18.1 16.0 13.5	$1.7 \\ 1.8 \\ 1.6 \\ 1.5 \\ 1.3 \\ 1.1 \\ 1.3 \\ 0.9 \\ 0.8 \\ 0.9 \\ 1.3 \\ 1.3 \\ 0.9 \\ 0.9 \\ 1.3 \\ 0.9 $	0.4 0.2 0.4 0.1 0.5 0.2 0.2 0.3 T 0.1 T 0.4
Other Oils Avocado, Taylor Avocado, Lula Cirrus seed oil Wesson oil ^d Corn oil Lard ^e	$19.1 \\ 22.5 \\ 28.0 \\ 22.3 \\ 12.0 \\ 22.8 \\$	7.0 11.0 0.3 1.1 2.9	$0.4 \\ 0.8 \\ 5.4 \\ 2.7 \\ 2.1 \\ 18.5$	55.8 46.6 22.6 17.1 29.7 42.6	$17.0 \\ 17.5 \\ 37.2 \\ 55.6 \\ 54.8 \\ 10.8 $	$\begin{array}{c} 0.8 \\ 1.3 \\ 6.5 \\ 0.2 \\ 1.2 \\ 0.6 \end{array}$	0.3 0.1 0.2 0.4

TABLE I Oil Analysis by Gas-Liquid Chromatography

^a Included in each peanut value: behenic 3.1%, lignoceric acid 1.1%, and U.S.M. 0.5%. ^b Included 0.5% Clis. ^c Trace.

^d Not included 0.9% myristic. * Not included 1.4% C₁₅.

eties are arranged in order of increasing oleic acid content. Oleic acid in the peanut oil ranges from 43 to 64% and in the pecan oil from 51 to 77%, with linoleic acid in complementary amounts of 33 to 18% and 38 to 14%. Saturated acids are nearly constant in level except for palmitic in the peanut. In the peanut oils palmitic acid ranges from 13 to 7% and varies directly with the linoleic acid content (r=0.92)as indicated by the rank difference method.

The GLC procedure did not determine any acid containing above 20 carbons in its chain or take account of unsaponifiable matter. Iodine values, calculated from GLC data, were higher than the determined ones in the peanut oils. These values suggested the presence of significant quantities of saturated acids with chains longer than 20 carbons, so adjustment was made in the peanut oils percentage for a content of behenic (3.1%) and lignoceric (1.1%)acids, given as average values in Hilditch (1a). The peanut oils analyzed an average 0.5% unsaponifiable matter. These values totaled 4.7% which was applied equally to all peanut analyses.

Oleic acid in the two avocado oils analyzed 47 and 56% and linoleic acid remained at the 17 to 17.5% level. The acid complementary to oleic was hexadecenoic acid at 11 and 7% while palmitic acid varied directly with it from 23 to 19%.

Traces of components other than those mentioned above were found in certain of the oils. Traces of myristic and a 15-carbon acid were found in peanut and avocado oils. Trace of a 15-carbon but no 14carbon acid was found in pecan oil. The rancid lard sample showed 14 pips between the 14-carbon and 20-carbon instead of the expected 7. Peanuts showed consistently slightly more arachidic content than the other oils and, judging from the agreement in the determined and calculated iodine values, these peanuts contained usual amounts of the longer chain saturated fatty acids: behenic and lignoceric.

TABLE II Comparison of Fatty Acid Analysis by GLC and UV Absorption Following Alkaline Isomerization

Item	Oleic Acid, %		Linoleic Acid, %		Linolenic Acid, %		Iodine Value	
	GLC	UV	GLC	UV	GLC	UV	GLC ^b	Wijs
Peanut								
Spanish					ĺ			
18-38-47	43.1	40.7	32.5	32.7	1.5	0.2	97.5	101.8
NC 2	51.9	51.4	26.2	25.4	1.3	0.0	93.4	92.3
Va. Runner	Í				,			
G 26	52.3	50.3	25.1	25.6	1.5	0.1	92.9	92.0
Spanette	52.5	50.9	25.7	25.2	1.3	0.1	93.4	92.0
Georgia								
119-20	54.4	54.4	23.2	22.9	1.4	0.1	90.7	90.7
Va. 56 R	54.5	54.8	23.8	22.8	1.4	0.1	92.3	90.8
Early Runner	55.6	54.6	24.7	24.0	1.1	0.1	93.5	92.9
Virg. Bunch G2	55.7	55.3	23.7	22.8	1.3	0.2	92.4	91.3
Florigiant	55.7 55.7	55.1	23.1 22.9	22.8	1.5	0.2	91.8	90.3
Dixie Runner		57.1	22.4	21.8	1.1	0.1	92.0	91.1
Florida 393	63.5	61.6	17.7	17.4	1.4	0.2	88.9	87.4
I fortua 055								
Average	54.5	53.3	24.4	23.9	1.4	0.1	101.9	101.3
Pecan					:			
Moneymaker	51.0	51.3	37.8	37.0	1.7	1.8	114.0	118.0
Mobile	56.8	55.6	31.4	30.6	1.8	2.1	107.9	111.2
Big Z	59.7	62.7	30.3	29.3	1.6	1.4	108.0	110.0
Randall	61.3	59.5	28.2	27.4	1.5	1.5	105.2	107.2
Teche	63.4	62.1	25.7	25.2	1.3	1.4	102.4	105.3
Frotscher	63.9	62.9	25.5	25.4	1.3	1.5	102.5	105.9
Stuart	68.8	65.4	21.0	22.1	1.1	1.3	98.8	101.3
Seedling #1	69.2	69.2	21.2	20.4	1.3	1.4	99.6	100.8
Seedling #2	71.0	71.0	18.5	17.0	0.9	1.1	95.5	97.6
Curtis	71.8	70.5	18.1	17.7	0.8	1.0	95.9	98.3
Van Deman	74.6	73.6	16.0	15.0	0.9	1.1	94.2	96.2
Success	76.5	77.2	13.5	12.7	1.3	0.8	92.6	94.5
Average	65.7	65.1	23.9	23.3	1.3	1.3	101.4	103.8
Other Oils							2	
Citrus seed				1				
oil	22.9	24.7	37.2	35.1	6.5	6.7	100.2	104.1
Avocado,		- 2.1	0	00.1		···	1	
Taylor	62.8*	61.7	17.0	14.2	0.8	1.3	86.2	84.6
Avocado,								
Lula	57.6ª	54.8	17.5	15.5	1.3	1.5	84.3	81.5
Wesson oil	18.2*	22.0	55.6	49.9	0.2	0.4		:111.4
Corn oil	29.7	35.9	54.8	47.7	1.2	1.7	123.6	123.5
Lard	46.7*	61.3	10.8	2.5	0.6	1.1	61.1	62.4

^a Includes hexadecenoic acid.

^b Calculated.

The wide variation in unsaturated acid content of the pecans which were grown in this general area and the peanuts which were grown on the same farm under almost identical environmental temperatures suggested that genetic factors may be of importance equal to that of environmental temperature (1a) in influencing degree of unsaturation. Too, the data showed that in the peanut, possibly in the avocado, but not in the pecan, variation in palmitic acid correlated with variations in unsaturated acid content. These variations suggested that processes of synthesis of unsaturated and saturated fatty acid may not always be as separate in plants as had been hypothesized (1b).

It had been suggested that Spanish, runner, and Virginia types (5) of peanuts would show characteristic differences in concentrations of unsaturated fatty acids. The figures for unsaturated fatty acid concentrations showed little association with type. Perhaps this should be expected since a "pure type" might be hard to find today with geneticists making many crosses between types in order to produce peanuts with desired qualities. But it should be noted that 9 of the peanut oils were quite similar in composition, with one distinctly higher in linoleic acid and the other distinctly lower.

Comparative Analyses

Iodine values, Table II, as calculated from the GLC procedure, agreed well with those obtained in the Wijs procedure, except for the peanut oils. But the iodine values of peanut oils when adjusted for content of longer chain saturated fatty acids and for unsaponifiable matter, fell into line with the others except possibly for Spanish 18, the peanut highest in linoleic acid.

Unsaturated fatty acid analysis by the two procedures shows surprisingly good agreement, Table II, except for Wesson oil, corn oil, and lard. With these samples bought in a local store, both oleic and linolenic acid have shown higher, and linoleic acid lower values, by GLC than by spectral methods. Craig and Murty (6) noted, with oils high in linoleic acid like corn, sunflower, and soybean, a similar lack of agreement in results. Interestingly, the slightly rancid leaf lard fitted into this same pattern. Herb et al. (7) demonstrated close agreement for lard by the GLC and UV methods and their analysis fitted our GLC pattern for lard. It is noteworthy that pecan oils (Table II) showed close agreement in linolenic acid content by the two methods while peanut oils did not, even though the linoleic acid ran through a similar range of values in the two series.

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REFERENCES

- Hilditch, T. P. The Chemical Constitution of Natural Fats, 3rd ed. rev. 1956. A-p. 234; b-p. 171; c-p. 445; d-p. 575.
 Official and Tentative Methods of the American Oil Chemists' Society, 2nd ed., rev. to 1957, Cd. 1-25.
 Ibid. Cd. 7-48.
 Hammons, Ray. Quoted from letter 10/3/58.
 Freeman, A. J., Nelle J. Morris, and Robert K. Willich. AIC. 370, March 1954, p. 22.
 G. Graig, B. M., and N. L. Murty. JAOCS, 36, 549-52 (1959).
 Herb, S. F., Paul Magidman, and R. W. Riemenschneider. JAOCS, 37, 127-9 (1960).

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Some Differences in Composition of Covering Fat, Intermuscular Fat, and Intramuscular Fat of Meat Animals¹

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A method is described for rapid, reproducible extraction of total lipids from adipose and muscle tissues. The distribution of polyunsaturated fatty acids in the depot fats and intramuscular lipids in loin or rib roasts of pork, beef, lamb, and veal has been determined. The phos-phatides of the muscle were found to contain a greater amount of polyunsaturated fatty acids than the neutral fats of the intramuscular lipids.

TT HAS long been recognized that the lipids of an aniand are not uniform in composition throughout the carcass. Banks and Hilditch (1), Bhattacharya and Hilditch (2), and Dean and Hilditch (6) showed that the inner fat of pigs contained a higher proportion of saturated fatty acids than were found in the outer fats. Hilditch and Zaky (9) studied the body fats of sheep and found a similar relation. They also reviewed other work and noted that a similar distribution occurred in cow fat. Dugan, et al. (7) studied the brisket fat of cattle and found that the outer layers were more unsaturated than the inner layers. They showed further that the oleic acid percentage was higher and the saturated acid percentage was lower in brisket fat than in back fats of cattle. Shorland (15) noted that sheep depot fats were higher in stearic acid content and correspondingly lower in long chain polyunsaturated acids than were the muscle fats. Hartman and Shorland (8) reported that the muscle lipids from loin mutton chops contained a somewhat higher proportion of C_{20} unsaturated fatty acids. They found approximately 3% as compared with 1% or less reported by other observers on sheep fats. Callow (4) noted that the iodine value of the fats from fatty tissues of lambs ranged from 43.6 to 56.1 while the iodine values of the fats from muscle ranged from 51.5 to 61.7.

The compositional variations of fat within the carcass have been noted but not systematically studied for all species of meat animals. The observation of Hartman that muscle lipids showed a greater proportion of C₂₀ unsaturated fatty acids in one location in one species prompted an investigation of the possible difference between the covering, intermuscular, and intramuscular fat of three species of meat animals.

Methods and Materials

The problem of fat extraction from adipose tissues is fairly commonplace, but the muscle tissues are more difficult to extract due to the lower total lipid, and higher protein and water content. Therefore, the lipid material was extracted from the muscle by a variety of methods. These included:

- 1. Digestion with sulfuric acid and extraction with petroleum ether b.r. 36-60C (12).
- 2. Extraction in a Soxhlet extractor using a 50/50mixture of ethanol (absolute) and petroleum ether.
- 3. Maceration with hot methanol (absolute) in a Waring Blendor, followed by addition of petroleum ether and blending, then adding a distilled water solution of zinc acetate to precipitate the protein and aid in separation into two phases.
- 4. A modification of the method of Bligh and Dyer (3) for total lipid from fish.

Since this last method (14) was chosen for subsequent analyses, it is described in detail.

Preparation of the Sample

- 1. Separate the fat and muscle by dissection. Grind the various samples through a meat grinder with a ¹/₈-in disk. Grind the adipose tissues two times and the muscle samples three times. Mincing into small pieces with a scalpel serves equally well.
- 2. Weigh samples for extraction on aluminum foil on an analytical balance. If they are not to be extracted within one day, they are stored at -20Cuntil used.

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