TABLE II

Relative Detector Responses of Fatty Methyl Esters

Compound	Detector responses		
	Area/mole, Rm		Area/gram, Rw
	(A)	(B)	
Methyl caproate	(1.000)	142.4	(1.000)
Methyl caprylate	1.160	165.2	0.954
Methyl caprate	1.301	185.3	0.909
Methyl laurate	1.467	208.9	0.891
Methyl myristate	1.619	230.5	0.869
Methyl palmitate[a]	1.773	252.6	0.854
Methyl stearate[a]	1.927	274.6	0.840

[a] Extrapolated.

Acknowledgment

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Extraction of Soybean and Cottonseed Oil by Four Solvents[1]

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VARIOUS SOLVENTS have been both studied and used commercially for the solvent extraction of vegetable oils. Benzol (benzene) has been popular in Europe. Ethyl alcohol has been used in Asia largely because of its ready availability and low cost. In the United States the solvents commonly used are petroleum fractions, mainly the hexane fraction. The common method of determining this fraction by its boiling point does not necessarily establish its chemical composition. The commercial product may contain as low as 60% or as high as 88% n-hexane. Other constituents commonly present are methyl cyclopentane, methyl pentanes, and benzene. Little or no data are available showing the effect of these nonhexane constituents on the solubility of vegetable oils or on the quality of the oil extracted by them.

Studies were made on the rate of extraction of soybeans and cottonseed with pure hexane, pure benzene, and two varieties of commercial hexane commonly used in oil extraction. Composition data on the three hexanes are shown in Table I. The benzene was "reagent" grade. Cracked soybeans containing 7 to 8% moisture were heated to 160°F. and rolled into flakes with an average thickness of 16 mils. The extractions were carried out in laboratory-rate extraction apparatus similar to that used in previous studies in this laboratory (1), except somewhat larger. The extraction chamber was 12 in. high by 2 in. in diameter allowing the use of 100-gram samples. The extraction chamber and the incoming solvent were heated to 135 to 140°F. The solvent passed through the flakes at a rate producing 10 ml. per minute of miscella. Samples were taken at 10-minute intervals. The solvent was evaporated from each fraction under vacuum and the oil weighed. The residual oil content of each sample was determined in Soxhlet apparatus using the solvent drained from the sample in the rate extractor as the extracting solvent. Cottonseed meats were extracted in a similar manner. Results for both are shown in Table II.

TABLE I

Composition of the "Hexanes"[a]—Amounts in Volume Percentages

Constituents	Pure	High purity	Commercial
N-Hexane	99.5	88.4	59.7
Methyl cyclopentane	0.0	7.6	22.4
2-Methyl pentane	0.0	0.0	5.4
3-Methyl pentane	0.0	0.0	11.3
2- and 3-Methyl pentanes	0.0	3.0	0.0
2,2-Dimethyl butane	0.0	0.3	0.0
2,4-Dimethyl pentane	0.0	0.7	0.0
2,3-Dimethyl butane	0.0	0.0	0.3
Cyclo hexane	0.4	0.0	0.9
Benzene	<0.001	<0.001	0.0

[a] Hexanes and their composition data supplied by Phillips Petroleum Company.

Composites of the oil aliquots from each complete extraction were examined for quality with the results

TABLE II

Extraction Data

	Extraction time in min.	Residual extractable oil, % of original[a]			
		"Commercial" hexane	"High purity" hexane	"Pure" hexane	Benzene
Soybean oil	10	69.5	69.5	73.6	64.8
	20	45.2	47.4	53.7	39.9
	30	29.7	32.4	38.9	25.5
	40	19.7	21.4	27.1	15.8
	50	12.8	13.8	20.2	11.2
	60	8.0	8.7	14.2	8.5
	70	4.8	5.0	10.0	7.0
	80	3.0	2.8	7.0	6.2
Cottonseed oil	10	82.8	79.5	80.5	82.8
	20	63.6	63.7	61.4	65.8
	30	48.0	47.4	46.3	50.6
	40	35.4	34.8	34.6	37.1
	50	26.1	25.4	25.3	28.6
	60	19.0	18.8	18.5	20.7
	70	13.6	13.5	13.5	14.8
	80	9.8	9.8	9.9	10.6
	90	7.1	7.2	7.2	7.7
	100	5.1	5.1	5.3	5.7

[a] Original oil content of the flakes, 19.08% (dry basis). A residual extractable oil of 3.0% would be equivalent to 0.7% of the dry extracted meal.

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[1] Presented at the 51st annual spring meeting, American Oil Chemists' Society, Dallas, Tex., April 3–6, 1960.

TABLE III

Quality of Oil Extracted by the Four Solvents

	"Commercial" hexane	"High purity" hexane	"Pure" hexane	Benzene
Soybean oil				
F.F.A., %	1.4	1.3	0.7	0.9
Neutral oil, %	94.9	94.1	94.7	90.8
Color	46.8	47.1	41.3	58.2
Cottonseed oil				
F.F.A., %	2.8	2.6	1.1	1.0
Neutral oil, %	91.9	91.4	92.3	93.6
Color	319.6	225.8	342.1	477.5

summarized in Table III. Free fatty acid content was determined by the A.O.C.S. official method (2a). Color was determined by the A.O.C.S. tentative photometric method (2b) and neutral oil by the A.O.C.S. tentative chromatographic method (2c).

Extraction of soybeans by pure hexane was slower than by the other two hexanes. During the first 60 minutes extraction by benzene was more rapid than by the other solvents. At the end of 80 minutes the amount of oil extracted by benzene was only slightly more than by pure hexane and definitely less than by the commercial hexanes. The rate of extraction of cottonseed was practically the same for the four solvents.

On the basis of free fatty acids and color the best quality soybean oil was extracted by pure hexane. The benzene-extracted oil had the lowest neutral oil content and highest color. Pure hexane produced a cottonseed oil exceeded in color only by that extracted by benzene. The best colored oil was produced by the high purity hexane. Apparently there is little choice between the two commercial hexanes for soybean oil extraction. A lighter colored cottonseed oil was produced by the "high purity" hexane. These results do not necessarily apply to extraction at other temperatures than 135 to 140°F. range nor to extraction carried out under different conditions.

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ABSTRACTS . . . . R. A. REINERS, Editor

ABSTACTORS: R. R. Allen, S. S. Chang, Sini'tiro Kawamura,
F. A. Kummerow, Louise R. Morrow, and E. G. Perkins

• Fats and Oils

CHROMATOGRAPHIC STUDIES ON THE COMPOSITION OF COMMERCIAL SAMPLES OF TRIOLEIN-I^{131} AND OLEIC ACID-I^{131}, AND THE DISTRIBUTION OF THE LABEL IN HUMAN SERUM LIPIDS FOLLOWING ORAL ADMINISTRATION OF THESE LIPIDS. G. Lakshminarayana, F. A. Kruger, D. G. Cornwell, and J. B. Brown (Ohio State Univ.). *Arch. Biochem. Biophys.* **88**, 318–327 (1960). Triolein-I^{131} and oleic acid-I^{131} ("Raolein" and "Raoleic acid," Abbott) were chromatographed on a standardized silicic acid column. Triolein-I^{131} was found to contain methyl esters and tri-, di-, and monoglycerides of labeled oleic acid. Gas chromatographic, infrared spectrographic, and chemical analyses confirmed the presence of these components. The radioactivity distribution showed a lower proportion of triglycerides than did chemical analysis. Following oral administration of triolein-I^{131} or oleic acid-I^{131} to normal individuals, the chromatographic fractionation of the serum lipids showed that about 90% of the radioactivity was in the triglyceride fraction and the remainder was mostly in the diglyceride and non-esterified fatty acid fractions.

NEUTRON ACTIVATION PAPER CHROMATOGRAPHIC ANALYSIS OF PHOSPHATIDES IN MAMMALIAN CELL FRACTIONS. E. H. Strickland and A. A. Benson (Pennsylvania State Univ.). *Arch. Biochem. Biophys.* **88**, 344–348 (1960). Diphosphatidylglycerol (cardiolipin) was found in the mitochondria of a number of cell fractions. Its concentration was estimated by neutron activation chromatographic analysis of the deacylated derivative, 1,3-diglycerophosphorylglycerol. Microsomes contained little or none of this lipid. The distributions of the other glycerol phosphatides in mitochondria and microsomes were similar. A possible functional role for diphosphatidylglycerol is discussed.

EDIBLE FATS AND PROCESS FOR MAKING SAME. H. Wissebach (Lever Bros. Co.). *U. S. 2,942,984.* A natural fat such as palm oil, shea butter, lard, beef tallow, or mutton tallow is fractionally crystallized so as to obtain a liquid fraction having a softening point below 30°C. The liquid is then hydrogenated to give a fat having an iodine value in the range of 50 to 65, a softening point in the range of 30°C. to 45°C., and a dilatation at 20°C. of not less than 1200.

DYEING SOLUTION FOR COLORING CITRUS FRUITS AND METHOD OF DYEING CITRUS FRUITS. H. J. Keller (Clearwater, Fla.). *U. S. 2,943,943.* An annatto derivative is dissolved in a hydrocarbon solvent, oleic acid, and an amine (the amine being present in at least equal amounts in relation to the oleic acid). The mixture is emulsified with water, maintained at a pH between 7 and 13, and applied to the skin of citrus fruits to dye them.

PROCESS FOR NEUTRALIZING VEGETABLE OR ANIMAL OILS OR FATS IN A WATER-SOLUBLE SOLVENT. C. Vaccarino and G. Vaccarino (Messina, Italy). *U. S. 2,944,072.* Vegetable and animal oils and fats are dissolved in a solvent miscible in all proportions with both water and the fats and oils. The solution is treated with sodium or potassium hydroxide or carbonate until neutralization of the oils and fats is complete. Hot water is added and the two immiscible liquid phases thus obtained are separated. The solvent is removed and the neutralized oils and fats are collected. The solvent-free aqueous phase, which is a solution of soap and water-soluble impurities, is discarded.

MAYONNAISE PRODUCT AND METHOD OF MANUFACTURE. J. G. Spitzer, L. S. Nasarevich, J. L. Lange, and H. S. Bondi (Carter Products, Inc.). *U. S. 2,944,906.* A mayonnaise-like product of relatively low caloric content is an oil in water emulsion in which the oil phase dispersed throughout the continuous water phase consists of edible oil droplets at a concentration of 5.0 to 30% by weight of the total composition. The emulsion also contains an egg yolk containing material (1 to 7% by weight), an acidifying agent (0.1 to 1.5% by weight based on pure acid content), a stiff gum (0.5 to 2.5% by weight), and a predominant amount of water.

SOLVENTS FOR FAT AND OIL ANTIOXIDANTS. M. B. Knowles and H. S. Pridgen (Eastman Kodak Co.). *U. S. 2,944,908.* A liquid antioxidant for addition to fats and fatty oils contains as the active constituent 5 to 50% of a phenolic antioxidant. The solvent consists of from 10 to 95 parts by weight of an acetic acid ester of glycerine out of a total of 10 to 164 parts by weight of solvent.

PROCESS FOR LUBRICATING AND PROMOTING THE COHESION OF TEXTILE FIBERS. W. H. Shields and H. T. Buckley (Emery Ind., Inc.). *U. S. 2,944,920.* A textile dressing consists of 5% to 50% polymerized fatty acid compound (polymerized fatty acids, polymerized esters of fatty acids and monohydric alcohols of up to 8 carbon atoms, or polymers of polymerized fatty acids and dihydric alcohols of up to 8 carbon atoms chain length) and 95% to 50% of an oily diluent. This diluent may be an animal, vegetable, or mineral oil, a liquid fatty acid or a liquid ester of a fatty acid.