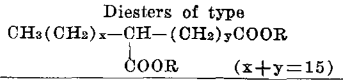
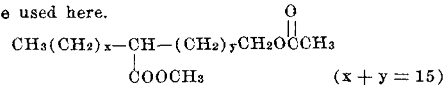
TABLE III

Comparison of Properties of Commercial Poly(Vinyl Chloride) (Geon 101) Plasticized with Diesters and Commercial Plasticizers

Diesters of type  $CH_3(CH_2)_x\text{—}\underset{\displaystyle COOR}{CH}\text{—}(CH_2)_yCOOR$ (x+y=15)	Tensile strength, p.s.i.	Elongation, %	Modulus, 100%, p.s.i.	Clash-Berg		Migration, % wt. loss after 14 days	Volatility wt. loss, %
				$T_{135,000}$, °C.	$T_{10,000}$, °C.		
R[a]							
Methyl	2500	340	1200	—41	—8	13	6.6
Ethyl	2600	310	1100	—48	—6	16	5.6
n-Propyl	2400	310	1100	—50	—6	16	2.7
n-Butyl	1900	220	1200	—57	+2	17	5.0
Isobutyl	2500	330	1300	—47	+9	16	6.7
Carbomethoxyoctadecyl acetate[b]	2400	310	1000	—47	—6	16	3.8
Controls							
Di-2-ethylhexyl phthalate (DOP)	2600	330	1100	—30	0	4	1.6
Di-2-ethylhexyl sebacate (DOS)	2950	370	1400	—59	—19	23	2.0

[a] The same samples used for viscosity characteristics (Table II) were used here.

[b] Note that this ester is different from those above. Its formula is



$CH_3(CH_2)_x\text{—}\underset{\displaystyle COOCH_3}{CH}\text{—}(CH_2)_yCH_2O\overset{\displaystyle O}{\overset{\|}{C}}CH_3$ (x + y = 15)

Certain diesters, however, are compatible with poly (vinyl chloride) at the 35% level. These are the dimethyl, diethyl, and di-*n*-propyl esters of carboxystearic acid and carbomethoxyoctadecyl acetate. These diesters are efficient plasticizers, as shown by the relatively low modulus figures, and they are efficient at low temperatures. The Clash-Berg $T_{135,000}$ values range from —41°C. (dimethyl ester) to —57°C. (di-*n*-butyl ester). Comparison of the $T_{10,000}$ with the $T_{135,000}$ figures indicates a relatively slow increase in modulus with decrease in temperature, a desirable characteristic. The modulus-temperature curves for these diesters are not as shallow as that of di-2-ethylhexyl sebacate.

The dibutyl and diisobutyl esters of carboxystearic acid, at the 35% level in PVC, showed a heavy exudate after 24 hr. Di-2-ethylhexyl carboxystearate and PVC did not even fuse on milling. This is in line with previous results which show a ratio of twelve carbon atoms to one ester linkage is about the maximum for compatibility. In the last case, there is a ratio of 35 to 2.

The migration weight losses for the diesters are slightly lower than that of DOS but higher than that of DOP. Volatility losses are higher in the new diesters than in the controls.

In the heat stability test in air at 160°C., the PVC specimens containing the diesters turned black in 2½ to 3 hr., as did the controls containing DOP.

In the light stability test, samples containing dimethyl and dipropyl esters of carboxystearic acid were equal to the controls with DOP, which showed little or no change to 216 hr. when the test was discontinued. The samples containing the diethyl ester of carboxystearic acid and those containing carbomethoxyoctadecyl acetate showed increased spotting after 144 hr.

No attempts were made to prepare compositions of maximum attainable heat and light stability.

Acknowledgments

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

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• Fats and Oils

PROCESSING OLIVE OILS. II. INFLUENCE OF PROCESSING ON THE STABILITY OF THE OIL. M. Nosti and J. M. R. de la Borbolla (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 139–150 (1960). The effects of processing on the oxidative stability of olive oil (as determined by the A.O.M. test) were studied. The addition of fatty acids to olive oil decreased stability. Lye refining also was detrimental. Vacuum bleaching had no noticeable effect on the stability of refined oils but lowered the stability of crude olive oil. When bleaching was carried out in contact with the air, however, stability was decreased. Deodorization always improved resistance to oxidation. Except for very poor quality oils, processed olive oil always had poorer oxidative stability than the crude oil from which it came.

INDUSTRIAL UTILIZATION OF WASTE LIQUOR FROM OLIVE OIL EXTRACTION ("ALPECHIN"). POSSIBLE USE AS FERTILIZER. M. A. Albi and J. A. Fiestas Ros (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 123–124 (1960). The waste liquor from olive oil extraction ("alpechin") was evaluated as a fertilizer since it was found to contain small amounts of potassium (0.94% as K_2O), phosphorus (0.27% as P_2O_5), and traces of Na, Ca, S, Cl, and Fe. In tests growing corn and wheat, no differences were observed between fertilized and unfertilized plants.

CHARACTERISTICS OF SPANISH OLIVE OILS. III. BELLIER'S TEST AS A METHOD OF CHARACTERIZATION. J. Gracian and G. Arevalo (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 261–269 (1960). Analysis of Spanish olive oils with Bellier's test (amount of alcohol-insoluble fatty acids) showed wide variations from year to year. Variations were attributed to climatic conditions and agricultural practices. Data indicated that Bellier's test must be carefully interpreted when used as a test of olive oil adulteration.

REFINING LOSS AND OTHER PROPERTIES OF SPANISH SOYBEAN OILS. R. de Castro (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 248–255 (1960). Laboratory refining of Spanish soybean oils gave losses under 7%. Most refined oils could be bleached to a 3.5 Lovibond red color, but some oils of high chlorophyll content remained darker.

THIN LAYER CHROMATOGRAPHY AND ITS APPLICATIONS. E. Vioque (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 223–228 (1960). A review.

STUDIES ON THE RANCIDIFICATION OF LARD. VI. THE INFLUENCE OF RENDERING AND STORAGE ON OXIDATIVE STABILITY. A. Vargas, J. M. R. de la Borbolla, and R. Gutierrez (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 243–247 (1960). Inadequate filtration after rendering, exposure to ultraviolet radiation, and contamination with Fe and Cu were found to decrease the A.O.M. stability of lard. Antioxidants helped overcome the detrimental effect of Fe.

THE INFLUENCE OF LIGHT ON CHEMICAL CHANGES IN OLIVE OIL. I. Miss R. Vazquez and J. M. R. de la Borbolla (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 163–172 (1960). The influence of different wave lengths of light on the development of oxidative rancidity in olive oils was studied. Crude olive oils were found to be very sensitive to light frequencies between 320 and 720 millimicrons, either with or without added antioxidants. This sensitivity was partly attributed to their chlorophyll content. Refined and bleached olive oils were only found sensitive to radiation between 320 and 450 millimicrons. Their stability (determined by peroxide number) was improved by the addition of antioxidants.

REFINING SOYBEAN OIL. R. de Castro and M. Nosti (Instituto de la Grasa y sus Derivados, Seville, Spain. *Grasas y Aceites* 11, 213–219 (1960). Laboratory test refining of U. S. soybean oil showed no relationship between crude FFA and refining loss. The use of excess lye tended to increase refining losses without improving refined oil color.

SEPARATION, IDENTIFICATION, AND QUANTITATIVE ANALYSIS OF FATTY ACIDS BY THIN-LAYER CHROMATOGRAPHY AND GAS-LIQUID CHROMATOGRAPHY. H. K. Mangold and R. Kammereck (Hormel Institute). *Chem. & Ind. (London)* **1961**, 1032–4. The use of thin-layer adsorption chromatography (TLC) for the fractionation of methyl esters of fatty acids is presented as a method complementary to gas-liquid partition chromatography (GLC). Since the esters of some hydroxylated acids may interfere in GLC, they are first removed by TLC. Then, the purified esters are reacted with mercuric acetate, and the acetoxymercuri-methoxy-derivatives of unsaturated esters are separated from saturated components and from each other, as classes, by TLC on silicic acid. The separation depends only on the degree of unsaturation and is independent of chain lengths. TLC supplements GLC in two ways. Minute amounts of esters in GLC fractions can be identified by TLC. Conversely, large amounts of the acetoxymercuri-methoxy-adducts of esters of unsaturated fatty acids were fractionated by TLC. Each group of esters was recovered and after cleavage of the addition compounds, it was resolved by GLC according to chain length.

TREATMENT OF VEGETABLE OIL WASH WATERS. E. E. Allen and G. J. Wack (A. E. Staley Mfg. Co.). *U. S. 2,993,006.* A method is described for treating wash water obtained during the refining of vegetable oils so as to recover the fatty materials and lower the B.O.D. value. Lignin, at a concentration of 0.05% to 0.10% by weight, is dissolved in the wash water at a pH of about 11–12 and a temperature in the range of 140–170°F. The solution is acidified to a pH of 1–3 and the resultant supernatant fatty layer is separated from the lower water layer. Improvement is effected by the addition of sodium aluminate to the wash water prior to the acidification in an amount of 25–100% by weight of the lignin.

AQUEOUS COMPOSITION COMPRISING WERNER COMPLEX, A STARCH, HYDROGENATED VEGETABLE OIL, AND EMULSIFYING AGENT AND METHOD OF PREPARING SAME. R. K. Gagnon and G. E. Rammel (Owens-Corning Fiberglas Corp.). *U. S. 2,993,872.* An aqueous composition for sizing glass fibers contains from 1–10% by weight of a Werner complex compound in which an acido group coordinated with a trivalent nuclear chromium atom contains from 10–18 carbon atoms in combination with 3–10% of a partially dextrinized starch, 3–10% of a hydrogenated vegetable oil lubricant, from 0.1–10% of an emulsifying agent, from 0.4–4% of a fatty acid cationic amine softening agent, from 0.03–2.0% gelatin, and from 0.05–1.0% polyvinyl alcohol.

MARGARINE OIL MANUFACTURE. D. S. Lindsay (Procter & Gamble Co.). *U. S. 2,996,388.* An oil suitable for margarine manufacture is prepared by corandomizing a mixture consisting of 40 to 65 parts of palm oil or a hydrogenated oil resembling palm oil in solid fat content (tallow, hydrogenated whale, fish or vegetable oil) and from 60 to 35 parts of a coconut oil type. Then about 1%–30%, by weight of the total oil, of an unrandomized oil of the coconut type is added to the corandomized mixture.

METHOD OF MAKING OLEOMARGARINE. G. C. North, P. P. Noznick, and B. A. Stiritz (Beatrice Food Co.). *U. S. 2,997,396.* A process of making margarine including an edible fat consists of incorporating in the melted fat an emulsion of fat and liquid milk with the fat in the dispersed phase, chilling the mixture, and working the mixture to produce margarine having dispersed in it the fat from the emulsion as discrete globules coated with non-fat milk solids.

• Fatty Acid Derivatives

GAS CHROMATOGRAPHIC ANALYSIS OF SUCROSE MONOSTEARATE. Mildred Gee and H. G. Walker, Jr. (Western Regional Res. Lab., Albany, Calif.). *Chem. & Ind. (London)* **1961**, 829–30. Commercial sucrose monostearate was methylated, saponified, deionized, and molecularly distilled. All material boiling through the hepta-*O*-methyl-sucrose range was collected and subjected to methanolysis to give a mixture of methyl-*O*-methyl fructosides and glucosides. The mixture was chromatographed at 162° on an aluminum column packed with 15% diethylene glycol succinate on Chromosorb W (Johns Manville). The labeling pattern of the methyl groups indicated that substitution of stearate was predominantly on C-6 of glucose but that appreciable substitution also occurred on C-4 of glucose and C-1 of fructose. About 3 times as much acyl substitution occurred on glucose as on fructose.

SUGAR ESTERS IN COSMETICS. L. Nobile (Ledoga-Le-Petit, Milan). *Drug and Cosmetic Ind.* **89**, 34, 92 (1961). Sugar esters and sucroglycerides have been prepared in Italy by a new process which uses natural fats instead of fatty acids. These products are solid or waxy, with melting points ranging from 80° to 130° for the sugar esters. Sucroglycerides have lower melting points and are semi-solid in consistency. They are potentially very useful raw materials in cosmetics because of their high dispersion and detergent power, their lack of toxicity, and their moderate surfactant activity, and because they do not interfere with the action of preservatives and bioactive products.

ANTI-SLUDGING HYDROCARBON FUEL OILS. J. C. Ownby and H. E. Davis (Eastman Kodak Co.). *U. S. 2,992,082.* A hydrocarbon oil is treated with 0.001% to 0.1% by weight of a sucrose mono- or diester in which the sucrose is esterified with an aliphatic carboxylic acid having at least 8 carbon atoms. The acid may be either an alkyl or alkenyl carboxylic acid.

PROCESS FOR THE PRODUCTION OF COMPOUNDS CONTAINING EPOXIDE GROUPS. G. Dieckelmann (Dehydag, Deutsche Hydrierwerke G.m.b.H., Dusseldorf). *U. S. 2,992,237.* An olefinically unsaturated compound (vegetable or animal oil, aliphatic hydrocarbon, natural wax ester, etc.) is mixed with (a) an organic polyhydroxy compound such as aliphatic hydrocarbon polyols containing from 3 to 10 carbons, monosaccharides, sugar polysaccharides, amylose, amylopectin, dextrins, glycogens, pectins, etc., and (b) an acid activator capable of accelerating peracid formation (phosphoric, nitric, sulfuric, or perchloric acid, boron trifluoride, *p*-toluenesulfonic acid, or high molecular weight synthetic cation exchange resins containing sulfonic acid groups). Then from 1.1 to 2.0 moles per molar equivalent of the olefinic bonds of an aqueous hydrogen peroxide solution containing 30% to 60% by weight of hydrogen peroxide is added. The mixture is maintained at a temperature between 0° and 100°

for 1 to 4 hours while stirring vigorously. The epoxidized olefinically-unsaturated compounds are then isolated.

Preparing glycidyl esters of higher fatty acids. C. S. Nevin and J. H. Fletcher (American Cyanamid Co.). *U. S. 2,992,239.* A molten fatty acid containing at least 10 carbon atoms is mixed with an alkali-metal carbonate and a quaternary ammonium halide catalyst in the mol ratios of 1.0:1.0–1.5:0.0025–0.01, respectively. About 9 to 13 moles of epichlorohydrin per mol of fatty acid is added and the resultant mixture is maintained at a temperature above the melting point of the fatty acid until reaction substantially ceases. The glycidyl ester of the fatty acid is then recovered from the resultant solution.

Stable lead alkyl compositions and a method for preparing the same. H. Shapiro and H. R. Neal (Ethyl Corp.). *U. S. 2,992,251.* A method of inhibiting the decomposition of an alkyllead compound at temperatures from 100° to 160° consists of incorporating a small amount of a saturated aliphatic monocarboxylic acid containing from 1 to 12 carbon atoms.

Cocoa butter substitute and process of making same. W. G. Alsop and A. C. Bell (Colgate-Palmolive Co.). *U. S. 2,993,063.* The cocoa butter substitute is a mixed higher fatty acid diester of propylene in which the acyl radicals are a mixture of oleyl and palmitoyl or stearoyl radicals in a molar ratio from 0.06 to 0.66 of oleyl to the saturated fatty acyl radical. The mixed diester has a melting point of about 30 to 35°.

Sustained release tablets. H. A. Nash and S. F. Jeffries (Dow Chemical Co.). *U. S. 2,993,836.* The described pharmaceutical preparation is a compressed tablet containing as the essential carrier ingredients approximately equal parts of glyceryl monostearate and gum karaya and a solid, nontoxic, particulate, water insoluble material in an amount equal to the combined quantities of the first two ingredients. In the carrier is incorporated up to 15% by weight of the desired therapeutic agent. The tablet is slowly disintegratable at a uniform rate in an aqueous medium independent of the pH of the medium.

Method for the production of water-insoluble fatty acid salts of multivalent-metals. H. Meyer. *U. S. 2,993,921.* High-molecular saturated fatty acids are saponified with an excess of alkali carbonate of between 2 and 10%. A precipitate is formed by reacting the mixture of saponified fatty acid and excess alkali carbonate with an acidified aqueous multivalent metal salt solution having a pH of 3 to 5. The precipitate is then separated.

Cutting oil containing calcium stearate. H. M. Stine and E. C. Hughes (Standard Oil Co.). *U. S. 2,995,516.* A liquid cutting oil consists of an anhydrous mineral oil of cutting oil viscosity, benzyl polysulfate in an amount to produce a corrosive active sulfur content of about 0.8%, and about 5% of calcium stearate.

Hexitol and hexitan partial esters and shortening agents containing same. S. B. Radlove (Glidden Co.). *U. S. 2,996,387.* One molar equivalent of a hexitol or hexitan is reacted with 1 to 2 molar equivalents of an even numbered, unsubstituted fatty acid having 12–22 carbon atoms and from 0.5–2.0 molar equivalents of edible, lactic acid. The reaction products contain, on an average, at least one fatty acid esterified with each molecule of polyhydric alcohol and are mixed esters composed in part of partial esters having at least 1 and not more than 3 unesterified hydroxyl groups per ester molecule.

Textile finishing agents. E. B. Lawler and K. D. Ballou (American Cyanamid Co.). *U. S. 2,997,407.* A method for applying a water-repellent composition to textiles comprises wetting out the textiles in an aqueous dispersion of the water-repellent composition by the wetting action of ammonium soaps of a mixture from 20% to 80% by weight of abietic acid and from 80% to 20% of fatty acids having 8 to 22 carbon atoms. The textiles are then dried by heating them at temperatures of from 220° to 400° F.

Method for preparing fatty esters. W. F. Huber (Procter & Gamble Co.). *U. S. 2,997,490.* Inositol is reacted with a fatty acid ester selected from the group consisting of the fatty acid esters of aliphatic primary mono-hydroxy alcohols having from 1 to 8 carbon atoms and fatty acid esters of polyhydric alcohols having not more than 3 carbon atoms in the presence of an interesterification catalyst at a temperature of from 50° to 150°. The reaction is run in dimethylsulfoxide.

Method for preparing partial fatty esters of inositol. W. F. Huber and J. B. Martin (Procter & Gamble Co.). *U. S. 2,997,491.* Inositol is reacted with a fatty acid ester selected from the group consisting of the fatty acid esters of aliphatic primary mono-hydroxy alcohols having from 1 to 8 carbon atoms and completely and incompletely esterified fatty acid esters of polyhydric alcohols having from 2 to 6 hydroxyl groups, the molar ratio of the fatty acid esters to inositol being less than that required for complete esterification of the inositol. The reaction is conducted in the presence of an interesterification catalyst at a temperature or from 80° to 150° and in the presence of an amount of dimethylacetamide at least sufficient to dissolve the inositol.

Method for preparing fatty esters of straight chain hexitols. J. B. Martin (Procter & Gamble Co.). *U. S. 2,997,492.* A straight-chain hexitol is reacted with a fatty acid ester selected from the group consisting of fatty acid esters of aliphatic primary monohydroxy alcohols having from 1 to 16 carbon atoms and fatty acid esters of aliphatic alcoholic polyhydroxy substances, in the presence of an interesterification catalyst at a temperature of from 90 to 115° and in the presence of pyridine. The catalyst is inactivated and the pyridine is distilled from the reaction mixture. The residue is taken up in a solvent and the resultant solution is water washed to obtain fatty esters of straight chain hexitols which are substantially free from anhydro groups.

Method for preparing straight chain hexitol fatty esters. W. F. Huber (Procter & Gamble Co.). *U. S. 2,997,493.* A straight chain hexitol is reacted with a fatty acid ester selected from the group consisting of fatty acid esters of aliphatic primary monohydroxy alcohols and completely and incompletely esterified fatty acid esters of polyhydric alcohols having from 2 to 6 hydroxyl groups, in the presence of an interesterification catalyst at a temperature from 50° to 150° in the presence of sulfoxide or an amide solvent.

• Biology and Nutrition

Olive seed proteins. II. Amino acids from acid hydrolysis. M. J. Fernandez (Instituto de la Grasa y sus Derivados, Seville, Spain). *Grasas y Aceites* 11, 173–179 (1960). The amino acid composition of acid hydrolyzed olive seed proteins were studied by means of electrophoresis and paper chromatography. The types and amounts of amino acids found in three different varieties of olive seeds were nearly identical. All the essential amino acids were present, indicating that the extraction of olive seed proteins could produce a food product of commercial importance.

III. New extraction methods. *Ibid.*, 220–22 (1960). Olive seed proteins were fractionally extracted from olive seeds by successive treatments with three solvents: water, brine, and 0.2 *N* NaOH.

Lipid transport in the laying hen and the incubating egg. P. Budowski, N. R. Bottino, and R. Reiser (Texas Agr. Expt. Sta.). *Arch. Biochem. Biophys.* 93, 483–90 (1961). Tracer doses of tripalmitin, C^{14}-labeled in both the glycerol and fatty acid moieties, were fed to laying hens over a 3-week period. Only 1.3% of the dietary tripalmitin glycerol, but 13.4% of its fatty acids, were transferred to the eggs. Triglyceride transport through the intestinal mucosa and ovary was accompanied by replacement of an appreciable fraction of its glycerol. Phospholipids passed from ovary to eggs without change. The relatively high isotope concentration in the glycerol of carcass glycerides indicated that considerable accumulation of dietary glycerol occurred in adipose tissue. New evidence is presented for the ability of the hen's ovary to synthesize sterols. During incubation, triglycerides were transported from yolk to embryo without change in activity, while phospholipids underwent hydrolysis with loss of glycerol activity. Evidence is also presented indicating synthesis of fatty acids and sterols in the embryo.

Stable solution of vitamin A—ascorbic acid complex in alcohol and process of preparing said solutions. G. Pancrazio and M. Vitali (Orma Instituto Terapeutico Romano I.T.R. S.r.l.). *U. S. 2,995,495.* An equimolecular mixture of ascorbic acid and vitamin A alcohol, acetate, or palmitate is heated in a nitrogen atmosphere under agitation in absolute ethyl alcohol. The solution has a titre from 1,000 to 500,000 I.U.