Report of the Literature Review Committee

Annual Review of the Literature on Fats, Oils and Detergents. Part II.

V. MAHADEVAN, University of Minnesota, The Hormel Institute, Austin, Minnesota

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PRODUCTS (EXCEPTING DETERGENTS)

EDIBLE, PHARMACEUTICAL AND COSMETIC PRODUCTS

TLC techniques and lipase hydrolysis were used to study the distribution of fatty acids in milk triglycerides (Blank and Privett, *J. Dairy Sci. 47*, 481). Free fatty acids of milk fat were determined by gas-liquid chromatography using two internal standards (Bills, et al., (*Ibid., 46*, 1342). Milk phospholipids were reported to occur in units of relatively homogeneous composition, despite their complexity (Patton, et al., (*Ibid., 47*, 489). The Alfa-Laval and the Babcock-Gerber methods of determining fat content of milk were compared (Randolph and Gould, *Ind. Aliment. 2*, 60). Indirect estimates of protein and solids-not-fat were compared with determinations of the major components of milk to establish the accuracy of the indirect methods (Erb, et al., *J. Dairy Sci. 46*, 1217). Studies of the rheology of condensed milk and ice cream were reviewed (Tsuchiya, *Yukagaku 13*, 100).

A spectrophotometric method for the determination of tocopherol in milk and milk products was described (Erickson and Dunkley, *Anal. Chem. 36*, 1055). Ascorbic acid was added to protect milk fat tocopherols during saponification of milk fat (Krukovsky, *J. Agr. Food Chem. 12*, 289). The distribution of milk fat tocopherols between the lipid in the fat globule membrane and the milk fat globule was studied (Erickson, et al., *J. Food Sci. 29*, 269). Gamma irradiation of milk fat was shown to produce monocarbonyls at a rate proportional to irradiation dose (Day and Papaioannou, *J. Dairy Sci. 46*, 1201).

Hardness of butter-like products was controlled by rearrangement of milk fat using selected times, temperatures and catalyst concentrations (Mickle, et al., *J. Dairy Sci. 46*, 1357). A process was patented for the manufacture of a free-flowing granular dairy product characterized by high wettability in cold water (McIntire and Loo, *U.S. 3,120,438*).

Butter fats were studied by means of gas chromatographic analysis (Chioffi and Magon, *Riv. Ital. Sostanze Grasse 41*, 243; Kuksis, et al., *JAOCS 41*, 201), isothermal gas chromatography (Daghetta and Jaforte, *Riv. Ital. Sostanze Grasse 40*, 597), and differential thermal analysis (Cantabrana and DeMan, *J. Dairy Sci. 47*, 32). A method was described for continuous determination of moisture in butter by measurement of the dielectric constant (Fexa and Burianec, *Papers Inst. Chem. Tech. Prague 6*, 131).

Adulteration of butter with more than 5–10% of lard or vegetable fat was detected by gas chromatography (Kuksis and McCarthy, *JAOCS 41*, 17). Thin-layer chromatography was used to quantitatively determine fat-soluble dyes in butter and margarine (Janicek, et al., *J. Inst. Chem. Technol. Prague 6-2*, 75). Microcoulometric gas chromatography following a forced volatilization cleanup of butter fat was used to determine organochlorine insecticide residues in butter (Ott and Gunther, *J. Agr. Food Chem. 12*, 239). Heptachlor epoxide residues were found in trace amounts in butter fat from cows that had grazed on pastures treated with granular heptachlor (Rusoff et al., *Ibid. 10*(5), 377).

The volatile constituents of fresh butter were separated by vacuum steam distillation and the carbonyl compounds in the aqueous distillate identified by paper chromatography of the 2,4-dinitrophenylhydrazones (Winter et al., *J. Food Sci. 28*, 554). Precursors of odd-numbered methyl ketones in butter fat were reported to be triglycerides containing one *beta*-keto acid and two fatty acid moieties (Parks et al., *J. Lipid Res. 5*, 232). Keeping quality of refrigerated butter was found to be improved by addition of 0.03% ascorbic acid during the manufacturing process (Budslawski and Zakrzewska, *J. Inst. Chem. Technol. Prague 6-2*, 27).

The manufacture and uses of butter powder were described (Hansen, *Australian J. Dairy Technol. 18*, 79). By control of the manufacturing process, butter powder could be prepared which was satisfactory as the only source of shortening in cake recipes (Hansen, *Ibid. 18*, 86).

Chemical characteristics and the nutritional value of margarine were reviewed (Montefredine, *Riv. Ital. Sostanze Grasse, Sympos. Issue 1962*, 97). Several margarine oils and other hydrogenated fats were evaluated with respect to their fatty acid composition, *trans*-acid content, keeping quality and consistency as determined by melting point and differential thermal analysis (Pokorny, *J. Inst. Chem. Tech. Prague 7-2*, 223). Methods were described for the determination of tocopherols in margarine by column and paper chromatography and spectrophotometric measurement (Lambertsen et al., *J. Food Sci. 29*, 164). Potassium sorbate and sorbic acid were compared as suppressors of the growth of microorganisms in stored margarines (Rutkowski and Holczak, *J. Inst. Chem. Tech. Prague 5-3*, 129).

Spattering properties of margarine were reduced by adding up to 0.3% by weight of a partial ester of a high molecular weight alkanol and citric acid (Houben and Wilhelmus, *U.S. 3,124,463*). Hydrogenated edible oils, or mixtures of them, were patented as edible fat sealants for margarine or butter wrappers (Josefowicz and Melnick, *U.S. 3,148,993*).

A process for producing prime steam lard comprised heating, under reduced pressure, with live steam, until equilibrium between condensation and evaporation was reached (Thompson, *U.S. 3,142,576*). To detect tallow adulteration in lard, it was suggested that dilatometric curves, the Bohmer index and the fatty acid composition must all be examined (Jacini et al., *Riv. Ital. Sostanze Grasse 40*, 584). When 10% or more tallow is present in lard, it was reported that a modification of the Bohmer reaction, as well as gas chromatography, will reveal adulteration (Pascucci and Paolini, *Riv. Ital. Sostanze Grasse, Sympos. Issue 1962*, 194). Another method of gas chromatographic detection of tallow in lard was based upon the discovery of two branched fatty acids in tallow which do not occur in lard (Grieco, *Ibid.*, 200).

The effectiveness of phenolic antioxidants in stablizing lard was investigated both with respect to its kind (Pokorny et al., *J. Inst. Chem. Tech. Prague 5-3*, 173) and with respect to the concentration (Porkorny and Vasakova, *Ibid. 5-3*, 11). Copper was found to lower peroxide values of lard independently of the presence of BHT or cystein (Pokorny and Janicek, *Ibid. 7-2*, 193). An extract from rosemary was reported to be a strong antioxidant for lard (Ostric-Matyasevic, *Rev. Franc. Corps Gras 10*, 443).

Determinations of fat consistency by dilatometric methods and by differential thermal analysis were compared (Pokorny, *J. Inst. Chem. Tech. Prague 5-3*, 141). A patent was issued for a plastic shortening comprising a mixture of hydrogenated lard and hydrogenated soybean oil in which the crystalline solids were claimed to be predominantly in the beta phase (Thompson, *U.S. 3,132,951*). A mixture of fatty triglyceride and partial esters of pentitols and hexitols was patented as an antispattering dry shortening composition (Wootton, *U.S. 3,138,463*). A monoester of propylene glycol and a fatty acid was included in a fluid shortening for cake batters (Abbott, *U.S. 3,145,110*). Also included in shortening compositions were: The products of condensation of glycolic and lactic acids with a mixture of fatty acid mono- and diglycerides (Howard, *U.S. 3,145,107*); the products of condensation of fatty acids with polycarboxylic acids containing hydroxyl groups and of fatty alcohols with dicarboxylic acids (Howard, *U.S. 3,145,108*); and mixed diglycerides containing long chain fatty acids and short chain fatty acids (Howard, *U.S.* 3,145,109).

Physical properties of shortenings were claimed to be improved by a quick tempering process in which the shortening triglyceride was held at 90–110F for 6 hr (Hoerr and Vander Wal, *U.S. 3,117,011*). A study of methods for determining shortening stability showed that the oxygen bomb method was twice as effective as the active oxygen method (Bennett, *JAOCS 41*, 505).

Liquid oils were found to be comparable to plastic-type shortenings in producing white bread, provided hard fat or physically hard emulsifiers were added to the oil (Bayfield and Young, *Bakers' Digest 37*(5), 58). When proofing doughs containing liquid shortenings were treated at elevated temperatures, performance was enhanced by addition of more hard fat than normally used at lower temperatures (Bayfield and Young, *Baker's Digest 37*, 59). Sorbitan- or isosorbide fatty acid esters and polyoxyethylene sorbitan- or polyoxyethylene isosorbide-fatty acid esters were included as emulsifiers in a liquid shortening composition (Geisler, *U.S. 3,117,010*).

A review of the edible fat industry in the United Kingdom noted that Britain is limited in its production of shortenings and margarine because oils must be bought on the foreign market (McKerrigan, *JAOCS 41*(4), 56A). Meat fats were reported to account for slightly more than 20% of the total U.S. edible fat production (Woerfel, *Ibid. 41*(1), 4A). Problems in the studies of edible fats were reviewed (Kaneko et al., *Yukagaku 12*, 568). Other reviews discussed the rheology of edible fats (Sone, *Ibid. 13*, 93) and rheometry (Date and Fukada, *Ibid. 13*, 52).

The chief factors in the loss of oil during deep fat frying were reported to be the time of cooking, the temperature of frying and the composition of the food (Ota and Izuyama, *Yukagaku 13*, 328). Foaming tendencies of frying oils were investigated by fractionating thermally oxidized soybean oil and evaluating the foaminess of the fractions by a gas pumping method (Ota, et al., *Ibid. 13*, 210). The foaming tendency of thermally oxidized trilinolein was shown to increase with heating time (Ota, et al., *Ibid. 13*, 264). Surface tension was found to have no relation to the foaming tendency of a frying oil, but viscosity seemed to be correlated with foaming (Ota, *Ibid. 13*, 269). In a study of the steam volatile carbonyls of stored potato chips, diacetyl was positively identified (Dornseifer and Powers, *Food Technol. 17*(10), 118). A procedure was described for preparing fried foods of low oil content by solvent extraction of the residual oil (Goulston, *U.S. 3,127,271*).

The current status and potential market for confectionery fats were reviewed (Feuge, *JAOCS 41*(4), 4A). A patent described a confectioner's fat comprising 80% of an interesterified tropical fat containing essentially no linoleic acid with the balance of the blend being an unarranged vegetable triglyceride containing enough linoleic acid, so that the final mixture contained up to 15% of linoleic acid (Gooding, *U.S. 3,133,819*). The ester of an aliphatic monohydric alcohol and a saturated fatty acid was included in a confectioners' molding starch composition (Evans, *U.S. 3,130,060*).

The fatty acid compositions and trigylceride structures of cocoa butters from three types of cocoa beans were compared (Chacko and Perkins, *JAOCS 41*, 843). Differential thermal analysis showed cocoa butters obtained by crushing and by solvent extraction differed in glyceride composition (Mathieu et al., *Ind. Aliment. 2*, 57). Only very small changes in cocoa butter were found to occur during normal roasting of cocoa beans (Pokorny et al., *J. Inst. Chem. Technol. Prague 6-2*, 285). Properties of chocolate coatings made with cocoa butter substitutes were reported to be unpredictable on the basis of the properties of the cocoa butter substitutes because the coating is actually a mixture of natural and substitute cocoa butter (Janicek et al., *Ibid. 6-1*, 323). Lauric type synthetic hard butters were shown to form a eutectic mixture with cocoa butter when added to it in even small amounts (Pokorny et al., *Ibid. 7-2*, 239). A study of cocoa butter substitutes prepared by hydrogenation of coconut oil showed their properties were not impaired by the introduction of high melting glycerides from the catalyst slurry (Pokorny and Janicek, *Ibid. 5-3*, 185).

To make a peanut spread which would not stick to the packaging material, the spread composition was heated to melt the fatty materials present, and packaged hot, so that crystallization of the fatty material occurred after packaging (Baker et al., *U.S. 3,127,272*). Hydrogenated rapeseed oil was patented as a stabilizer for peanut butter (Sanders, *U.S. 3,129,102*).

New uses for edible soybean proteins were listed (Machida, *Yukagaku 12*, 461). An extrusion-cooking procedure was described for preparing full-fat soybean flours for human consumption (Mustakas, *JAOCS 41*, 607). A patented protein food composition consisted of an edible oil entrapped as the discontinuous phase in an aerated, film-forming, soy-protein composition (Cheng and Neibling, *U.S. 3,111,408*). A powdered food product containing up to 25% of dispersed fat was prepared by using a grinding action to disperse the fat in the form of fine droplets (Scheidegger, *U.S. 3,142,569*). The action of soy flour lipoxidase was used to develop flavor in continuously mixed bread (Kleinschmidt, *Bakers' Digest 37* (5), 44).

Sagebrush oil was patented as a treatment for ulcers of the alimentary canal (Sherman, *U.S. 3,133,860*). Petroselenic acid and ricinoleic acid, and their derivatives, and derivatives of stearic acid and oleic acid were screened for antimycotic activity (Novak et al., *JAOCS 41*, 503). A number of the compounds exhibited a wide spectrum of activity, and almost all of them were active against at least one organism. Fatty acid polyglycol esters and fatty alcohol polyglycol ethers, fatty acid salts and a solid organic acid were included in a fungicidal composition (Telle and Grewe, *U.S. 3,127,311*).

Blood sugar lowering in animals was accomplished by oral administration of the reaction product of an aldohexose stereoisomer and urea and fatty acid esters of the aldohexose (Clark, *U.S. 3,149,033*). Mannide monooleate, aluminum monostearate and vegetable oil were incorporated in an emulsion vehicle for a parenteral vaccine preparation (Woodhour and Stim, *U.S. 3,149,036*). Sodium proteinate derived from soybean flour was the major component of a buccal tablet containing vitamin A (Wershaw and Kuehns, *U.S. 3,133,862*). A vitamin E-active, polyoxyethylene glycol ester of a tocopheryl ester of succinic acid was patented as a component of a water-dispersible vitamin preparation (Robeson, *U.S. 3,102,078*). A stable vitamin A composition was obtained by iodine isomerizing vitamin A, neovitamin A and their carboxylic esters in an inert solvent (Maxwell, *U.S. 3,136,794*). Polyethylene glycol esters and polyoxyethylene sorbitan esters of fatty acids, glyceryl triesters of short chain fatty acids, benzyl alcohol and antioxidants were included in an emulsifiable solution of fat soluble vitamins (Aiello and Bauernfeind, *U.S. 3,149,037*).

Dry, powdered, fat-soluble vitamin compositions were prepared by spray-drying a homogenized mixture of water, an oil solution of the vitamin, edible fat and gelatine and skim milk solids (Rosenberg, *U.S. 3,124,510*). A carotinoid composition of improved color retention was prepared by heating together the carotinoid in solution in edible lipid solvent and an aqueous dispersion of milk, soy or peanut protein to form a complex between the carotinoid and the protein (Wingard and Saperstein, *U.S. 3,125,451*).

The rheology of cosmetics and dentifrices was reviewed (Mataumura, *Yukagaku 13,* 108). Partially acetylated mono- and diglycerides were reported to produce cosmetic creams with better thermal resistance, better emulsion stability and better spreadibility than creams containing completely acetylated derivatives (Pokorny et al., *J. Inst. Chem. Tech. Prague 7-1,* 167). To eliminate interference with the iodometric titration, reducing and oxidizing substances in cosmetic creams were extracted with chloroform and dilute acetic acid solution before determination of peroxide value (Pokorny et al., *Ibid. 6-1,* 291). Lecithin and cosmetically applicable vegetable oils and esters of monocarboxylic and dicarboxylic acids were included in a perfumed bath-oil composition (Emory, *U.S. 3,150,049*).

EMULSIFIERS

The fundamental properties of profoamers and antifoamers were discussed (Schwarz and Reid, *Ind. Eng. Chem. 56,* 26). A GLC procedure for establishing hydrophile-lipophile balance in emulsifiers was described (Becher and Birkmeier, *JAOCS 41,* 169). The analysis of surface active agents was reviewed (Kimura and Izawa, *Yukagaku 13,* 177).

Evaluation of surface activity of symmetrical and unsymmetrical mono- and diglycerides showed the 2-monoglycerides are slightly more effective than 1-monoglycerides in reducing interfacial tension in oil-water systems (Hartman, *JAOCS 41,* 520). Relating emulsifier structure to functionality, it was reported an emulsifier or emulsifier system for continuous bread-mixing must contain high-melting monoglycerides with sufficient monostearin or monopalmitin to act as effective clathrating agents (Birnbaum, *The Baker's Digest 37,* 44). An aqueous emulsion containing glycerol monostearate, glycerine and lecithin was patented for making bread dough (Dalby and Fisher, *U.S. 3,144,399*).

Incorporation of an aqueous emulsion of molecularly distilled monoglyceride into dough in a manner permitting contact between the emulsion and the starch before the starch comes into contact with shortening was described as a means of improving the texture and freshness retention of baked food products (Jackson and Livingston, *U.S. 3,111,409*). A study of the optimum conditions for acetylation of monoglyceride emulsifiers with acetic anhydride showed reaction temperature is the most critical factor (Tomankova and Pokorny, *J. Inst. Chem. Technol. Prague 6-1,* 243). Cereal products containing about 0.3% of edible, saturated monoglyceride were described (Lee, *U.S. 3,113,868* and *U.S. 3,113,869*). Polyglycerol esters were described as new food emulsifiers having applications in margarine, peanut butter, cake mixes, confectionery and ice cream (Nash and Babayan, *Bakers Digest 37* (5), 72).

Stearyl monoglyceridyl citrate was found to serve as an emulsifier enhancer and emulsion stabilizer in shortening systems containing the usual emulsifiers (Geminder, *JAOCS 41,* 92). Stearyl lactoyl lactate and cetyl lactoyl lactate in combination with mono- and diglycerides were used as emulsifiers for edible triglyceride in a shortening composition (Thompson, *U.S. 3,144,341*). A liquid-liquid chromatography procedure was described for determination of lactic acid in shortenings containing lactylated glycerides (Buswell, *JAOCS 41,* 457). To determine sorbitan monostearate in cake and cake mixes, silica gel chromatography was followed by gas or paper chromatography (Wetterau et al., *Ibid. 41,* 791).

Physically stable emulsions of cottonseed oil and soybean oil at 20% concentration in a 2.5% solution of glycerol were prepared using 1% of homogeneous egg lecithin as the sole emulsifier (Zeringue et al., *JAOCS 41,* 688). Sorbitan trioleate was used as a coating for nitrocellulose particles (Coffee, *U.S. 3,118,797*). An emulsifier for use in aqueous emulsion polymerization processes comprised alkali metal soaps of rosin and rosin fatty acid mixtures (Lambert, *U.S. 3,135,698*). The colloidal character of polymeric surfactants derived from alkylphenol-formaldehyde condensates was investigated (Hayano et al., *Yukagaku 13,* 33).

The course of the esterification reaction between sucrose and methyl esters of lauric acid and stearic acid was studied using potassium carbonate as catalyst and dimethylformamide as solvent (Zajic and Bares, *J. Inst. Chem. Technol. Prague 7-1,* 151). The preparation of mono- and diesters of sucrose from the methyl esters of oleic and linoleic acids was also investigated (Zajic and Auerswald, *Ibid. 7-2,* 205). Traces of dimethylformamide in the sucrose esters were determined by IR analysis for the amide group (Zajic and Bares, *Ibid. 7-2,* 215). Separation of sucrose esters of long chain fatty acids was effected by thin layer chromatography and NMR data were obtained for the various components (Mima and Kitamori, *JAOCS 41,* 198). Esters of sucrose were found to lower interfacial tension to a greater degree than glycerol monostearate (Orlova et al., *Trudy Vniiz 23,* 298). Utilization of sucrose stearates as nonionic, edible emulsifiers was found to be limited by their poor solubility in water between 20C and 40C (Orlova et al., *Ibid. 23,* 286).

Light-colored sulfonated fatty acids and fatty acid derivatives were obtained by bleaching the sulfonated products with inorganic oxidizing agents (Wulff et al., *U.S. 3,142,691*). A process was described for preparation of fatty acid esters of hydroxyalkane sulfonic acids (Koczorowski et al., *U.S. 3,151,136*).

A polyoxyethylene derivative of a higher fatty acid ester of a polyhydric alcohol was used as a liquid emulsifier in combination with a hard partial glyceride to produce a dry, free-flowing emulsifier composition for a frozen confection mix (Knightly and Lensack, *U.S. 3,124,464*). Fatty acid-ethylene oxide adducts were analyzed by counter-current distribution (Wetterau et al., *JAOCS 41,* 383).

ESTERS, ACIDS, ALCOHOLS AND OTHER FAT DERIVATIVES

The influence of lead, calcium, barium and sodium oxides on the rate of alcoholysis of linseed oil with glycerin was examined (Hippe and Mauberg, *Przem. Chem. 42*(6), 309). Transesterification was driven to completion at low temperature by including 2,2-dimethoxypropane in the reaction mixture (Mason and Waller, *Anal. Chem. 36,* 583). Zinc catalyzed esterification of fish oil fatty acids with glycerol was shown to be the most feasible way of preparing triglycerides of these highly unsaturated fatty acids (Lehman and Gauglitz, *JAOCS 41,* 533). Complete esterification of partial glycerides at room temperature was achieved by reacting them with fatty acid anhydrides in the presence of perchloric acid as catalyst (Mattson et al., *J. Lipid Res. 5,* 374). Ortho-substituted quinones having lower aliphatic hydrocarbon substituents were described as catalysts for manufacturing esters of higher fatty acids and ether-alcohols (Johnson, *U.S. 3,138,624*).

Glycerolysis of oleostearine, using sodium hydroxide as catalyst, yielded a product containing 49% of monoglyceride in 2 hr at 205C (Bertoni et al., *Revista Argentina de Grasas y Aceites 5,* 43). Laboratory preparation of monoglycerides was simplified by esterification of isopropylidene glycerol with free fatty acids (Anfinsen and Perkins, *JAOCS 41,* 779). A procedure was also described for preparation of mono- and diglycerides by direct esterification of glycerol in the presence of p-toluenesulfonic acid as catalyst (Gros and Feuge, *Ibid. 41,* 727).

The heat resistance of acetylated mixtures of mono- and diglycerides was found to vary as a function of the degree of acetylation (Pokorny and Tomankova, *Papers Inst. Chem. Tech. Prague 6,* 171). Distribution of acyl groups between primary and secondary hydroxyls in partial esters was studied (Brandner and Birkmeier, *JAOCS 41,* 367). The preparation of methyl hydrogen azelate was investigated to establish optimum reaction conditions (Yasukawa and Abe, *Yukagaku 13,* 317). A procedure was described for purification of triacetin containing acetic acid as a contaminant (Trevoy and Tegg, *U.S. 3,108,133*). In molecular distillation of the glycerides of tall oil fatty acids, the unsaponifiables were found to be concentrated in the first distillate fractions (Antila et al., *Suomen Kemistilehti 35B,* 84).

Tetrahydrofurfuryl alcohol esters of tall oil acids were prepared by first forming the methyl esters of the tall oil acids and then interesterifying these with the tetrahydrofurfuryl alcohol in the presence of an alkaline catalyst (Sumio et al., *Jap. 5,131/63*). Lactic acid esters of fatty acids were prepared by reacting fatty acid halides with lactic acid (Buddemeyer and Moneymaker, *U.S. 3,141,030*). The preparation of glyceride caffeic acid esters was described (King, *U.S. 3,153,659*).

Crystallization of emulsified triglycerides was investigated and the temperature at which crystallization starts in emulsified systems was found to be lower than in nonemulsified systems (Skoda and Van den Tempel, *J. Colloid Sci. 18,* 568). Fractionation of glycerides by crystallization was discussed in terms of phase diagrams representing the balance between solid and liquid phases, taking into account the interconversion of the various polymorphic crystal forms (Sambuc, *Rev. Franc. Corps Gras 11,* 319). Phase behavior of six binary mixtures of palmito-oleo triglycerides was studied by microscopic observation of melting points, X-rays and differential thermal analysis (Moran, *Riv. Ital. Sostanze Grasse 40,* 412). Isologues and geometric isomers of fatty acid methyl esters were separated by countercurrent distribution between hexane and 0.2 N silver nitrate in 90% methanol (Scholfield et al., *Anal. Chem. 35,* 1588). A triglyceride possessing optical activity in the glycerol moiety was separated by column chromatography from the seed oils of the Chinese tallow tree (Maier and Holman, *Biochemistry 3,* 270).

A new, beta-prime, crystal form of 2-palmitoyldistearin was reported (Hugenberg and Lutton, *J. Chem. Eng. Data 8,* 606). The crystal structures of alkyl stearates were discussed (Aleby, *Acta Cryst. (suppl.) 16, Pt. 13,* A 55). Crystal structures of the stable forms of single triglycerides and 2-monoglycerides were determined by replacing the terminating methyl group in the hydrocarbon chain by a bromine atom (Larson, *Acta Cryst. (suppl.) 16, Pt. 13,* A 57). To explain the consistent pattern for long spacings observed in studies of the crystals of fatty acids and their derivatives, it was proposed that among normal-saturated compounds there are certain preferred angles of tilt of the long polymethylene chains and the various polymorpic modifications assume one of these (Gunstone, *Chem. Ind. (London) 1964,* 84). Investigation of the infrared spectra of single crystals of hexadecanoic, octadecanoic and docosanoic acids provided evidence for a new crystal form (Holland and Nielsen, *Acta Cryst. 16, Pt. 9,* 902).

Solid fatty acids were reported to be separated almost quantitatively from liquid fatty acids by solvent fractionation in a mixture of 60% symm. dichloroethane and 40% acetonitrile (Martinenghi, *Olearia 17,* 99). Optimum separation of stearic and oleic acids by urea inclusion was obtained when a 10–20x molar excess of urea was used (Zajic and Capova, *J. Inst. Chem. Tech. Prague 6-2,* 275). Separation of critical pairs of fatty acids by paper chromatography was facilitated by bromethoxylation of the fatty acids before chromatographing (Jobtscheff et al., *Fette Seifen Anstrichmittel 65,* 913). A procedure was described for preparation of isomer-free fatty acids by stereospecific hydroxylation of the methyl esters, chromatographic purification of the dihydroxystearate and bromination and debromination (Fedeli et al., *Riv. Ital. Sostanze Grasse 40,* 459).

Methods of isolation of erucic acid from the fatty acids of rapeseed oil were compared (Grynberg and Cyganska, *Tluszeze Srodki Pior. 7,* 319). Because of the potential commercial value of erucic acid from oriental mustard seed oil, the by-product essential oil was studied by GLC analysis (Kirk et al., *JAOCS 41,* 599). Brassidic acid was prepared by selenium-catalyzed isomerization of colza oil (Jakobowski *Tluszeze Srodki Pior. 7,* 260). The isomerization of polyunsaturated fatty acids was studied by gas chromatographic methods (Cartoni et al., *Riv. Ital. Sostanze Grasse 60,* 482). A procedure was proposed for systematic identification of unsaturated fatty acid esters by GLC on polyester substrates (Ackman and Burgher, *J. Chromatog. 11,* 185). Abietic, oleic, linoleic and linolenic acids were reacted with a mixture of glycerine and excess ethylene oxide to form a water-soluble, nonionic material (Altscher and Groll, *U.S. 3,124,602*).

Methyl-8-hydroxycaprylate was prepared by electrolytic oxidation of methyl hydrogen azelate or by bromination of the silver salt of the acid, acetylation and hydrolysis. The dimeric lactone of the acid was obtained by transesterification of the monoglyceride (Yasukawa and Abe, *Yukagaku 13,* 360). *Trans, trans*-9,11-octa-decadienoic acid was prepared in 35% yield by crystallization of the products of pyrolysis of the polymer formed by heating ricinelaidic at 235C under vacuum for 3–4 hr (Schneider et al., *JAOCS 41,* 605). To form *cis*- unsaturated fatty acids having an odd number of C atoms, a triple bonded compound was hydrogenated in methanol-pyridine using Raney nickel as catalyst (Grimmer and Kracht, *Chem. Ber. 96,* 3370). Polyenoic acids having a labeled carbon in the carboxyl group were prepared by a nitrile synthesis and subsequent acid catalyzed hydrolysis of the nitrile in dimethylsulfoxide (Stoffel and Vierwirth, *Angew. Chem. Internat. Edit. 2,* 94). In the reaction of phosphorus trichloride with fatty acids, shorter chain fatty acids were found to require a larger excess than the long chain fatty acids (Galbraith et al., *JAOCS 41,* 104).

A study of the monolayer properties of stearic acid on subsolutions ranging in pH from 2 to 11.1 showed that marked changes in the electrical characteristics and cohesion of the monolayer occur at pH 9 (Goddard and Ackilli, *J. Colloid Sci. 18,* 585). Changes in the force-area and potential-area characteristics of monolayers of saturated fatty acids were related to changes in the degree of ionization induced by changes in the pH of the subsolution (Spink, *Ibid. 18,* 512). When a monolayer of stearic acid was formed on a buffered calcium-containing substrate, complete conversion to calcium stearate occurred when the pH was greater than 9, no calcium stearate formed at a pH below 4, and in the pH range 5–7 the amount of calcium stearate formed in the monolayer depended on the buffer present (Bagg et al., *J. Am. Chem. Soc. 86,* 2759).

Stabilisation of unsaturated fatty acids by conversion to urea inclusion compounds was found to be effective only for oleic and linoleic acids, and then only if storage was at low temperature. Oxidation occurred if the compounds were stored at temperatures above 40–60C. Esters were protected less effectively than the free acids (Zalud et al., *Prum. Potravin 13,* 660).

The effects of aliphatic acids and their salts on the flame spectrometric emission of calcium were investigated (West, *Anal. Chem. 36,* 310). Linoleic acid was shown to affect both viscosity and the size of the granules of starch when present during the gelatinization of starch (Yasumatsu and Moritaka, *J. Food Sci. 29,* 198).

The hydrolysis of fatty allyl bromides was reported to be a good method for the preparation of unsaturated hydroxy fatty acids (Naudet et al., *Rev. Franc. Corps Gras 11,* 247). Procedures were evaluated for the preparation of the hydroxy unsaturated oils, methyl dimorphecolate and methyl lesquerolate, from *Dimorphtheca* and *Lesquerella* oils (Diamond et al., *JAOCS 41,* 430). Esters of 9,10-epoxystearic acid were converted to 9,10-dihydroxystearates in high yield by dissolving them in 1,4-dioxane and treating the solution at 15C with aqueous acid and then water. Fluoboric acid was more effective than sulfuric acid as catalyst for the reaction (Maerker et al., *JAOCS 41,* 585). Using gas-liquid chromatography, carbon numbers were determined for all 17 isomeric hydroxy- and acetoxy-stearates and for 15 of the 16 isomeric methyl oxosterates (Tulloch, *Ibid. 41,* 833). Infrared absorption spectra were determined for a number of the hydroxyl and carbonyl derivatives of octadecanoic acid (Guillaumin, *Rev. Franc. Corps Gras 10,* 541).

Palmitaldehyde was prepared by catalytic reduction of palmitoin or the corresponding diketone and by lithium aluminum hydride reduction of the diketone, and the mechanisms of the reactions were investigated by tritium labeling (Weiss, *Biochemistry 3,* 584). Unsaturated C-18 aldehydes were prepared by oxidizing oleyl and elaidyl tosylates with a mixture of sodium bicarbonate and dimethyl sulfoxide (Mahadevan, *JAOCS 41,* 520).

Unsaturated long chain alcohols were prepared by reduction of methyl esters in the presence of copper-chromium oxide and a cadmium soap (Ikeda and Komori, *Yukagaku 13,* 313). Both copper and cadmium soaps were used as catalysts for the hydrogenolysis of fatty acids to alcohols. Saturated fatty acids were reduced in the presence of 1% copper as soap; the unsaturated fatty acids, oleic and ricinoleic acid, were reduced in the presence of 2% copper and 1% cadmium as soaps (Pantulu and Achaya, *JAOCS 41,* 511). Primary alcohols from nonadecyl alcohol to triacintanol were prepared from behenic and palmitic acids, stearyl alcohol and n-octanol using standard methods of chain extension and degradation (Schilknecht and Renner, *Fette Seifen Anstrichmittel 66,* 176). The first ten members of a series of para-t-octylphenoxypoly(ethoxy)ethanols were prepared by the base-catalyzed addition of ethylene oxide to alkyl phenols (Mansfield and Locke, *JAOCS 41,* 267).

Long-chain hydroxy compounds, including alcohols, hydroxy esters, glycerides and glyceryl ethers, were converted to nitrates by treatment with acetyl nitrate to facilitate their analysis by thin-layer chromatography and infrared spectrometry (Malins et al., *Anal. Chem. 36,* 658). Fatty alcohols and glyceryl ethers were converted to alkyl iodides for separation by gas-liquid chromatography (Hoffman et al., *J. Lipid Res. 4,* 385). In still another approach to this same problem, methyl esters and naturally occurring glycerides were reduced to alcohols with lithium aluminum hydride and the alcohols were converted to p-phenylbenzoates which were then separated by reversed-phase thin-layer and reversed-phase column chromatography (Katz and Keeney, *Anal. Chem. 36,* 231). Separation of even-numbered n-aliphatic alcohols was achieved by reversed-phase paper chromatography using kerosine as the stationary phase and 80–95% acetic acid as the developing agent (Mukai, *Yukagaku 12,* 505). Long chain aliphatic alcohols were separated from hydrocarbons by silica gel thin-layer chromatography (Hashimoto and Mukai, *Yukagaku 12,* 613). A method for the determination of the amount of hydroxyethylation of various materials was developed from infrared spectroscopic measurements on ethylene oxide adducts (Glassmann and

Maennchen, *Fette Seifen Anstrichmittel 65*, 741).

Linolenic acid was reduced with hydrazine to form 9,15-octadecadienoate isomers which were then separated by countercurrent distribution and analyzed by gas-liquid chromatography (Butterfield et al., *JAOCS 41*, 397). In the transformation of methyl oleate to methyl elaidate catalyzed by mercaptans, an equilibrium mixture containing 77% elaidate was obtained from either direction (Kircher, *Ibid.* 41, 351).

Yields of cyclic fatty acids from thermal treatment of 95% pure linolenic acid in ethylene glycol were increased by increasing solvent ration, catalyst concentration or temperature. Highest yields were obtained in the presence of ethylene, which apparently added to the double bond to form a C-20 acid (Eisenhauer et al., *JAOCS 41*, 60). The feasibility of a process for continuous production of cyclic fatty acids from linseed oil was investigated (Beal et al., *Ibid. 41*, 683).

Fatty dichlorocyclopropanes were prepared in yields of 75–88% by adding dichlorocarbene to unsaturated fatty esters and fatty olefins. The dichlorocyclopropanes were converted to ethers by heating them with alcohols, and to alcohols by heating them with water (Kenney et al., *JAOCS 41*, 82). Undecano-lactan and a lauryl lactan were prepared from cycloundecanoic acid by nitrosation with a mixture of nitrosil sulfuric acid and oleum in chloroform (Ziegenbein and Lang, *Angew. Chem. Internat. Edit. 2*, 149). Cyclopropene-containing acids in cottonseed oil triglycerides were effectively decreased by treating the triglycerides at high temperature with aluminum silicate catalyst (Deutschman et al., *JAOCS 41*, 175).

Alpha-phosphono fatty acids, salts and esters were prepared from pelargonic, capric, lauric, myristic, palmitic and stearic acids and their properties investigated (Maurer et al., *JAOCS 41*, 205). Commercially available alpha-olefins were converted to organic phosphorus compounds by treating them with phosphorus trichloride and aluminum chloride in cold methylene chloride (Jungermann and Reich, *Ind. Eng. Chem. Prod. Res. Develop 2*, 315).

Silicon-containing heat-resistant oils were prepared in good yield from fatty derivatives and functional silanes (Ismail, *Fette Seifen Anstrichmittel 66*, 356). Trichlorosilane and methyldichlorosilane were added to the double bonds of fatty acids, including oleic, linoleic and 10-undecenoic acids, their methyl esters and methyl acetyl ricinoleate, in the presence of diacetyl and benzoyl peroxides as catalysts (Takaoka and Mihara, *J. Jap. Soc. Col. Mat. 35*(10), 439).

Patents were issued for a composition containing 99.5% maleic anhydride and at least 0.07% of an aliphatic monocarboxylic acid (Gans and Russell, *U.S. 3,140,300*); and for a monoester formed by reacting polyethylene glycol with the anhydride group of a maleic anhydride adduct to an unsaturated fatty acid (Crecelius et al., *U.S. 3,141,897*). Aromatic compounds were added to methyl linoleate, using methanesulfonic acid as catalyst, but the recovery of mono-adducts was complicated by the presence of di-adducts and polymeric material (Eisner et al., *JAOCS 41*, 557).

Epoxides of oleic acid, butyl oleate and the butyl ester of tall oil fatty acid were obtained in high yield by carrying out the epoxidation in benzene as a diluent and immediately extracting the product (Suhara, *Yukagaku 13*, 279). In a patented process for epoxidation of unsaturated fatty materials, formic acid was substituted for the acetic acid used in the conventional epoxidizing mixture, thus permitting the use of only catalytic amounts of sulfuric acid (Murata and Higo, *U.S. 3,112,325*).

A series of 8-keto fatty acids were prepared by condensation of suberic acid half methyl ester chloride with n-alkyliodides in the presence of a copper-zinc catalyst (Breusch and Kirkarli, *Fette Seifen Anstrichmittel 65*, 995). The glyceride of 12,13-epoxyoleic acid was isolated in 22–28% yield from *Veronica anthelmintica* seed (Krewson and Scott, *JAOCS 41*, 422). The heat stability and odor of epoxidized oils was improved by oxidizing the oils and then neutralizing them with an aqueous alkaline material (Harrison and Simmerman, *U.S. 3,150,153*). Dioxolated fatty acid esters were patented (Kuester, *U.S. 3,127,418*). The polarography of organic peroxides was reviewed (Hayano, *Yukagaku 12*, 605).

A variety of nitriles were added to alpha-olefins to form N-substituted amides, but the reaction failed using diolefins containing the vinyl group (Clarke et al., *JAOCS 41*, 78). Investigation of the reaction of fatty acids with diethanolamine showed that a complex equilibrium involving diethanolamide, monoesteramide and diesteramide results when two moles of fatty acid are reacted with one mole of ethanolamine. This equilibrium was affected by changes in reaction conditions as well as by changes in the ratios of reactants (Rigamonti and Peirano, *Olearia 17*, 91).

Fatty derivatives of aminoalkyl and hydroxyalkyl heteorocyclic amine bases (Katz, *U.S. 3,114,649*), and the fatty acid amide of dialkylaminoalkoxyalkyl amine (Katz, *U.S. 3,129,106*) were patented as compositions to enhance the bonding, wetting and anti-stripping properties of asphalt, tar and bituminous coatings. The preparation and plasticizing properties of N,N-bis(2-alkoxyethyl) amides of long chain fatty acids were investigated (Mod et al., *JAOCS 41*, 781).

Amides derived from piperidine were prepared and evaluated as plasticizers (Mod et al., *JAOCS 41*, 237); their analysis by gas chromatography was studied (Parrot and Paty, *Riv. Ital. Sostanze Grasse 41*, 143); and the mechanism of decomposition of halogenated varieties was investigated (Parrot and Paty, *Ibid. 41*, 149).

A polar substrate on a neutral support was used in the GLC separation of fatty amides and other polar fatty derivatives (Morrissette and Link, *JAOCS 41*, 415). The near infrared spectra of oleoamide and saturated fatty amides were reported (Kato et al., *Yukagaku 13*, 426). A method was described for determination of small amounts of nitrile in long chain fatty amides by separation on a silica gel column and IR determination of the nitrile (Buswell and Link, *JAOCS 41*, 717).

The reaction of hexamethylenediamine with acrylonitrile, in methanol, was reported to give high yields of N,N,N′,N′-tetra (beta-cyanoethyl)-hexamethylenediamine (Nitananai, *Yukagaku 13*, 369). Nonamethylenediamine reacted similarly with acrylonitrile to form the corresponding tetracyanoethylate (Nitananai, *Ibid. 13*, 374).

The chemistry, preparation and uses of fatty amine oxides were reviewed (Jungermann and Ginn, *Soap Chem. Specialties 40*(9), 59); and the thermal decomposition of dimethyllaurylamine oxide, via a deoxygenation process, was described (Shulman and Link, *JAOCS 41*, 329).

From studies of the thermal polymerization of linseed oil in the absence of air, it was concluded that, at least at lower temperatures, only the linolenic acid is involved in the polymerization process (Fedeli et al., *Riv. Ital. Sostanze Grasse 40*, 321). The polymerizing activity of autoxidized linseed oil, on the other hand, was reported to depend largely on the presence of conjugated double bonds (Pokorny, *J. Inst. Chem. Tech. Prague 5-3*, 51).

Pyrolysis products of high polymers from unsaturated monomers were analyzed by gas chromatography as a means of developing a method for the analysis of unknown polymers and copolymers (Braun, *Farbe Lack 69*, 820). Dimers from heated 10-*trans*, 12-*trans* linoleate were analyzed by NMR, by IR and UV spectrophotometry, by mass spectrometry and chemically by ozonolysis to establish the presence of four isomers (Paschke et al., *JAOCS 41*, 723).

Investigation of the copolymerization of the methyl esters of unsaturated fatty acids showed linoleic, linolenic and conjugated linoleic esters copolymerize readily with acrylonitrile, only conjugated esters show any tendency to copolymerize with styrene, and methyl eleostearate, with three conjugated double bonds, inhibits the polymerization of both acrylonitrile and styrene (Mayo and Gould, *JAOCS 41*, 25).

Dehydro dimers of methyl oleate were prepared by the action of di-t-butyl peroxide, and from chemical oxidation and mass spectrometry data it was concluded that the dimers resulted from free radical bridging at the 8, 9, 10, and 11 positions equally (Paschke et al., *JAOCS 41*, 56). Characteristics were also reported for polymers of linoleic acid and its methyl ester, prepared using iron pentacarbonyl as catalyst (Tanabe and Hashimoto, *Yukagaku 13*, 16).

Polyglycerols were analyzed quantitatively by paper chromatography (Zajic, *Papers Inst. Chem. Tech. Prague 6*, 179). Thermal polymerization of glycerol, after prolonged heating at 200C, was reported to occur through an intermediate compound identified by IR spectrophotometry as 2,3-epoxypropanol (Zajic, *Ibid. 7-1*, 179). In the presence of alkali (Na_2O) polymerization appears to proceed by way of an alcoholate which was detected by paper chromatography (Zajic, *Ibid. 7-1*, 191).

The unsaponifiable fraction of several vegetable oils was shown by vapor phase chromatography to contain some 30 to 45 hydrocarbons plus 6 to 8 unidentified components (Capella et al., *Riv. Ital. Sostanze Grasse 40*, 603). Up to 50% of squalene and some 4.2% of C-29 and C-31 hydrocarbons were identified in deodorizer condensates comprising soybean oil unsaponifiables (Evans et al., *JAOCS 41*, 406). The fact that behenic acid generates hydrocarbons when heated at 200C in the presence of bentonite was postulated as evidence that fatty acids may have been the source of petroleum hydrocarbons (Jurg and Eisma, *Science 144*, 1451).

Temperature programs and column packings were evaluated for their effectiveness in gas chromatographic analysis of straight chain alkylbenzenes (Carnes, *Anal. Chem. 36,* 1197). Methyl oct-*cis*-2-enoate was synthesized by selective hydrogenation of methyl oct-2-ynoate at atmospheric pressure using a quinoline poisoned palladium catalyst (Mercuri et al., *JAOCS 41, 89*).

Good yields of the nitrates of fatty alcohols, hydroxy esters and vicinal glycols were obtained by reaction with acetyl nitrate formed by treating acetic anhydride with nitric acid (Malins et al., *JAOCS 41,* 44). Phosphorus containing derivatives of long chain hydroxy acids, including ricinoleic, lesquerolic and dimorphecolic acids and their hydrogenated derivatives, were prepared by reaction with dialkyl phosphorochloridates in the presence of dimethyl formamide as catalyst, or by ester interchange with a low molecular weight dialkyl phosphite (Diamond et al., *Ibid. 41,* 9).

Sulfhydryl groups were reported to add to the double bond of the cyclopropene ring in sterculene and methyl sterculate when these compounds were added to dilute solutions of methyl mercaptan or beta-mercapto propionic acid (Kircher, *JAOCS 41,* 4). The influence of n-lauryl mercaptan on the curing of unsaturated polyesters was investigated (Mewes, *Thesis, Stuttgart 1962; List of Accessions, Roy. Inst. Technology Library, Stockhlm, 1963 (147)* 13). The properties of vegetable oils sulfurished by treatment with sulfur monochloride were examined (Schiemann et al., *Deutsche Farben-Z. 17,* 408).

The autoxidation of blown linseed oil was found to be catalysed more effectively by hydroperoxides than by other peroxide types (Pokorny, *J. Inst. Chem. Technol. Prague 6-2,* 267). Vicinal methacryloxy-hydroxy-soya oils were prepared by reacting epoxidised oil with vinyl carboxylic acids. Cleavage of the oxirane ring was followed by addition of the vinyl acid through its carboxyl group (Nevin and Moser, *J. Appl. Polymer Sci.* 7(5), 1853). Unbranched omega-oxo fatty acids, or fatty acid derivatives, were prepared by enzymatic oxidation in the presence of reduced diphosphopyridine nucleotide (Mitz, *U.S. 3,116,215*).

FATTY MATERIALS USED IN TEXTILE AND PAPER TREATMENT, WATER-PROOFERS, CORROSION INHIBITORS, WAXES, DEFOAMERS, WELL-DRILLING FLUIDS, INCENDIARY PREPARATIONS, AGRICULTURAL AND MISCELLANEOUS

Investigation of the possibility of prospecting for uranium by analysis of olive oil mill wastes showed up to 12% of uranium may be found concentrated in the lipotropic membrane surrounding the dispersed oil droplets in the "alpechin" or waste water (Martinez et al., *Grasas y Aceites 15,* 1). Problems in the manufacture of synthetic sausage casing from hide parings, caused by residual containing lipids, were discussed (Pokorny and Ulrichova, *J. Inst. Chem. Technol. Prague 6-1,* 207). A corrosion inhibiting composition based on tall oil amide was patented (Kirkpatrick and Seale, *U.S. 3,134,759*). The literature on sesamin and sesamolin and their pyrethrum synergist properties was reviewed (Budowski, *JAOCS 41,* 280). A procedure was described for making a carotenoid preparation (Muller and Tamm, *U.S. 3,110,598*).

Reviews covered the following topics: General tendencies of research in the fatty oil industry (Komori, *Yukagaku 13,* 335); a report of the AOCS Industrial Oils and Derivatives committee (Anon., *JAOCS 41*(1), 12A); methods for utilizing soybean oil phosphatides in soapstocks (Ramos, *Grasas y Aceites 14,* 171).

The role of the Vanaspati industry in the marketing of oils and oilseeds was described (Dharan, *Indian Oilseeds J.* 7, 196); and some aspects of the marketing of oilseeds and their products in India were reviewed (Chatterji, *Ibid.,* 179). A series of publications on marketing of oilseeds and oilseed products in India dealt with the following market areas: Andhra Pradesh (Subba Rao, *Ibid.,* 199); Mysore State (Jamakhandimath, *Ibid.,* 203); Madras State (Srinivasan, *Ibid.,* 213); Uttar Pradesh (Rana, *Ibid.,* 220); Delhi (Sharma, *Ibid.,* 223); and Himachal Pradesh (Krishna, *Ibid.,* 226).

FATTY MATERIALS IN LUBRICATION, METAL WORKING AND TEXTILE OILING

During 1964 there were no publications which could be classified in these categories.

DRYING OILS, PAINTS, RESINS AND PLASTICIZERS

Specific Oils: Source, Composition, Properties

The problems of characterizing an oil were discussed with particular reference to oils containing long-chain unsaturated acids, conjugated acids, hydroxy, epoxy and cyclopropenoid acids (Mills, *J. Oil Colour Chem. Assoc. 47*(3), 187). The oil contents of 36 species of oilseeds and fruits and the fatty acid compositions of the oils were reported (Grieco and Piepoli, *Riv. Ital. Sostanze Grasse 41,* 283). Evidence for the presence of small amounts of 11-octadecenoic acid in various oils was obtained (Tulloch and Craig, *JAOCS 41,* 322). The fatty acid distributions in the oils of 28 plant species were determined. Palmitic and stearic acids and acids of greater than 18 carbon chain length were esterified predominantly at the primary hydroxyl groups. Oleic, linoleic and linolenic acids were approximately randomly esterified at the hydroxyl groups not esterified by the former group of acids (Mattson and Volpenhein, *J. Lipid Res. 4,* 392). The glycerides of oilseed plants exposed at the seed-setting stage to radioactive carbon dioxide contained randomly C^{14}-labeled fatty acids (Mounts and Dutton, *JAOCS 41,* 537).

The fatty acid composition of castor oil methyl esters was determined by gas chromatography. Oxidative cleavage of methyl ricinoleate showed the double bond to be exclusively in the 9–10 position. The fatty acid distribution of castor glycerides was studied by countercurrent distribution. The glycerides were 69 mole % triricinolein (Achaya et al., *Ibid.,* 783). A paper chromatographic study showed that the amounts of ricinoleic, linoleic and stearic acids in castor oil increased as the castor seeds ripened (Chandra, *Ibid.,* 251).

The fatty acids of isano oil consisted of saturated acids, 6%; oleic acid, 14%; linoleic acid, 5%; five C_{18} acetylenic acids, 51%; four 8-hydroxy C_{18} acetylenic acids, 22%; and 9,10-dihydroxystearic acid, 2% (Badami and Gunstone, *J. Sci. Food Agr. 14,* 863). A review on this oil covered its history, isolation, fatty acid composition and uses, particularly in paints and related products (von Mikusch, *Farbe Lack 70,* 17, 101).

The growing of linseed and safflower in Queensland, Australia, was discussed. The linseed yield was low and studies were under way to improve it (Wagner, *Austral. Paint J. 9*(4), 17, 20). The importance of climate and plant breeding in the growing of oilseeds in Canada was discussed. The oil content and fatty acid composition of linseed was affected by temperature and the amount of rainfall (Sallans, *JAOCS 41,* 215). Under-developed linseeds contained less oil than normal seeds but this was not true in the case of sunflower and castor seeds. The iodine values of oil from under-developed seeds were the same as those of oils from mature seeds (Kartha, *Indian J. Chem. 1*(6), 280). The unsaturated structures in linseed oil were determined by NMR spectroscopy (Harlan, *JAOCS 41* (9), supp 4). When linseed was heated at 95–100C for 40–45 minutes during processing, nonhydratable phosphatides appeared in the oil (Siminov and Gribova, *Trudy Vniiz 23,* 58). Methods of removing phosphatides from linseed oil were studied. The best method was to treat the oil with 0.05–0.3% of hydrochloric acid for 15 minutes at 20–30C (Guillaumin and Drouhin, *Rev. Franc. Corps Gras 10,* 671). The refractive index of linseed oil could be used to determine its density, iodine value, fatty acid composition and viscosity (Fauve, *Double Liaison 1963*(94), 43). The strong correlation between refractive index and iodine value of linseed oil and also of safflower oil was confirmed by a study extending over three seasons (Price, *Austral. Paint J. 9*(4), 17, 20). The hydrocarbon compositions of the unsaponifiable material from several vegetable oils, including linseed oil, were determined by gas chromatography. Each oil contained from 36 to 45 hydrocarbons (Capella et al., *Riv. Ital. Sostanze Grasse 40,* 603).

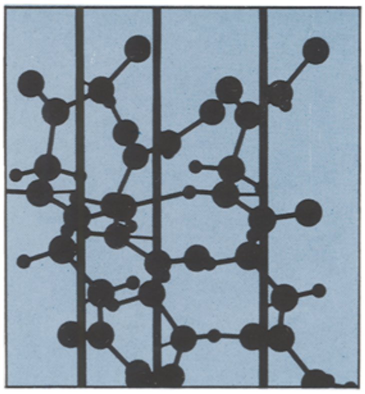
The by-product essential oil obtained from mustard seed during processing was shown to consist mainly of an equilibrium mixture of allyl thiocyanate and allyl isothiocyanate (Kirk et al., *JAOCS 41,* 599). The chemical composition of oiticica oil and its use as a drying oil was reviewed (*Rev. Argent. Grasa Aceites 5,* 62).

Extraction of soybeans with hot ethyl alcohol yielded an oil that did not need refining (Visnepol'skaja, *Trudy Vniiz 23,* 131). The degumming, refining, decoloration and deodorization of soybean oil was reviewed (Romero and Ayerbe, *Rev. Franc. Corps Gras 11,* 3). The color stability of refined soybean oil was improved by deodorizing in the presence of an alkali metal salt of a polybasic acid such as sodium or potassium citrate, tartrate or oxalate (Nisshin Oil Mills, *Jap. 5,005/63*). The presence in soybean and safflower oils of small amounts of saturated acids above arachidic, *cis*-11-octadecenoic acid and *cis*-11-eicosenoic acid was demonstrated. Safflower oil also contained 0.1% of *cis*-15-tetracosenoic acid (Kuemmel, *JAOCS 41,* 667). Refining losses of sunflower oil were approximately equal to 1.91 (% free fatty acids) + 1.16 and were unrelated to moisture content (Nosti, *Grasas Aceites 14,* 210). Saturated and monounsaturated acids with odd numbers of carbon atoms were found in sunflower oil (Bhatty and Craig, *JAOCS 41,* 508).

A number of phenols were identified by gas chromatography in the phenol fraction from the fore-run in the distillation of tall oil (Sandermann and Weissmann, *Z. Anal. Chem. 189*(1), 137). Properties, uses and paper chromatography of tall oil were discussed. The component acids were separated by reversed-phase chromatography. Best separation of the resin and fatty acids was obtained after hydrogenation of the mixed acids *in situ* on the paper (Berger and Müller, *Plaste Kautschuk 10*(9), 566; (10), 631). By countercurrent distribution of tall oil fatty acids followed by gas chromatography of the methyl esters, seven known acids (including 41% of oleic acid and 38% of linoleic acid) and small amounts of fourteen unknown acids with 17–20 C atoms were separated. The latter included a trienoic acid which was probably *cis*-5,9,12-octadecatrienoic acid (Aho et al., *Tehn. Kem. Aikl. 19,* 390). On molecular distillation of glycerides of tall fatty acids, unsaponifiable compounds were concentrated in the first fractions (Antila et al., *Suomen Kemistilehti 35B,* 84).

The galactolipids of alfalfa leaves were highly unsaturated and contained 87–94% of linolenic acid; the sulfolipid contained approximately equal amounts of palmitic and linolenic acids (O'Brien and Benson, *J. Lipid Res. 5,* 432). Cowpea seeds and *Polygonum japonicum* seeds contained 0.56% and 2.70%, respectively, of oils with iodine values of 129.7 and 126.8, respectively. Peach seeds contained 16.5% of an oil which contained solid and liquid acids, the iodine values of the acids being 74.2 and 123.1, respectively (Hamada and Ueno, *Yukagaku 13,* 195). The acids from mandarin orange seed oil contained 3.5% linolenic, 36.0% linoleic, 24.5% oleic and 36.1% saturated acids (Shalika et al., *Indian Soap J. 29,* 71). The seed oil of pimiento pepper and of two varieties of bell-type peppers contained 66–71% of linoleic acid. The oil content and composition of the former was not influenced by geographical location. Oil from the fruit wall and placenta of pimientos contained large amounts of linolenic and small amounts of long-chain polyunsaturated fatty acids (Marion and Dempsey, *JAOCS 41,* 548). Siberian pine seed oil contained a large amount of linoleic and a moderate amount of linolenic acid. This oil dried more slowly and gave softer films than did common drying oils but 50% of it could be added to linseed oil or tung oil without substantially affecting the drying rate of the latter oils. Drying was accelerated by promoters such as manganous and cobaltous salts (Pokorny and Pokorna, *J. Inst. Chem. Technol. Prague 6-1,* 285). The refining and bleaching of tobacco seed oil was best carried out by the method recommended by the AOCS for peanut oil. Addition of 0.5% of tetrasodium pyrophosphate reduced refining losses by about 30% (Readdy et al., *Research Industry (India) 8,* 227; *Oils and Oilseeds J. (Bombay) 16,* 6). The seed oils of *Echium plantagineum* (Smith et al., *JAOCS 41,* 290) and *Onosmodium occidentale,* both members of the Boraginaceae family, contained 6,9,12-octadecatrienoic and 6,9,12,15-octadecatetraenoic acids. Linolenic, 9,12-octadecadienoic, 9-octadecenoic and 11-eicosenoic acids were also reported in the latter oil (Craig and Bhatty, *Ibid.,* 209). The ranges of some of these acids in the seed oils of 29 species of this family (iodine values 88–225) were 0.3–50% linolenic, 0–27% 6,9,12-octadecatrienoic and 0–17% nonconjugated octadecatetraenoic (Kleiman et al., *Ibid.,* 459).

The seed fat of *Citrullus colocynthis* contained 18.6% saturated, 20.9% oleic, 58.8% linoleic and 1.65% linolenic acids (Gupta and Chakrabarty, *J. Sci. Food Agr. 15,* 74). The seed oil of *Chilopsis linearis* contained 5% saturated acids, 12% *trans*-10,*trans*-12-octadecadienoic acid, 15% *trans*-9,*trans*-12-octadecadienoic acid, 25% *trans*-9,*trans*-11,*cis*-13-octadecatrienoic acid and 25% linoleic acid (Chisholm and Hopkins, *Can. J. Chem. 41,* 1888; *JAOCS 41,* 42). A review covered research on *Crambe abyssinica* oil and its chemical composition and possible uses (Niewiandomski, *Tluszczc i Srodki Piorace 7,* 247). Solvent extraction of this oil was carried out on a plant scale (*Chem. Eng. News 42*(9), 50). An enzyme in *Crambe abyssinica* seed hydrolyzed the oil at moisture contents above 15% but the oil was stable under ordinary conditions of seed storage and processing (Tookey and Wolff, *JAOCS 41,* 602). *Dimorphotheca sinuata* seed oil contained 66.5% dimorphecolic, 14% linoleic, 10% oleic and 4.5% palmitic and stearic acids.



The presence of 2.5% of 9-keto,*trans*-10,*trans*-12-octadecadienoic acid in this oil was also established (Binder et al., *Ibid.*, 108). To determine the distribution of dimorphecolic acid (9-hydroxyl,*trans*-10,*trans*-12-octadecadienoic acid) among the close relatives of *Dimorphotheca*, seed oils from 29 species in 5 genera of the tribe Calenduleae, family Compositae, were examined. Dimorphecolic acid occurred to the extent of 34-75% in all 5 of the *Dimorphotheca* species examined, in Castalis nudicaulis, and in 5 species of *Osteospermum*. In all other species of the tribe analyzed, including 14 species of *Osteospermum*, 2 of *Calendula* and 2 of *Chrysanthemoides*, the oil contained 14-60% of conjugated trienoic acids (Earle et al., *Ibid.*, 345). Methyl dimorphecolate and methyl lesquerolate could be prepared by the methanolysis of *Dimorphotheca* and *Lesquerella* oils, respectively, using an anion exchange resin as a catalyst (Binder and Goldblatt, *Ibid.*, 430). The oil of *Euglena gracilis* contained 51 fatty acids including diunsaturated acids from C_{15} to C_{20}, triunsaturated acids from C_{15} to C_{20}, tetraunsaturated acids from C_{15} to C_{22} and a hexaenoic acid (Korn, *J. Lipid Res. 5*, 352). The oil of *Euphausia superba* (iodine value 136.1) contained 68.2% of unsaturated fatty acids from C_{14} to C_{22} (Tsuyuki et al., *Yukagaku 13*, 203). The seed oil of *Gevuina avellana* contained 22% of 11-hexadecenoic acid (Tomas et al., *Rev. Argent. Grasas Aceites 5*, 53). The seed fat of *Gynandropsis pentaphylla* contained 28.7% saturated, 15.4% oleic, 53.9% linoleic, and 2.0% linolenic acids (Gupta and Chakrabarty, *J. Sci. Food Agr. 15*, 69). About 80% of the glycerides of the seed fat of *Momordica charantia* were stearodiunsaturated glycerides. The fatty acids contained 67.4% of unsaturated acids including 56.3% eleostearic (Subbaram et al., *JAOCS 41*, 691). *Podocarpus nagi* seed oil contained *cis*-5,*cis*-11,*cis*-14-eicosatrienoic acid and *cis*-11,*cis*-14-eicosadienoic acid (Takagi, *Ibid.*, 516). *Vernonia anthelmintica* (L). Willd. (ironweed) seeds from Pakistan or India contained 22-28% of oil with a high content of trivernolin, the glyceride of 12,13-epoxyoleic acid. Lipolysis during extraction of the oil was controlled by flaking the seed in the presence of the extracting solvent or by autoclaving the seed prior to flaking (Krewson and Scott, *Ibid.*, 422). Trivernolin, 1,3-divernolin and vernolic acid (epoxyoleic acid) of commercial quality and also of very high purity were prepared (Krewson and Luddy, *Ibid.*, 134). The hydrolysis of trivernolin at the secondary ester position by the lipase of *Vernonia anthelmintica* was apparently due to the specificity of the enzyme rather than to the structure of the triglyceride because the triglyceride was hydrolyzed at the primary ester positions by pancreatic lipase (Sampugna et al., *Ibid.*, 132).

Gas chromatography was used to determine the fatty acid compositions of the body fats of 14 species of saltwater fish, 3 species of freshwater fish and 4 species of shellfish, of the liver fats of 2 species and the egg fat of 1 species. The percentages of a particular acid varied widely among the species and even among samples of the same species caught during different seasons. The oils of oysters and scallops contained large amounts of pentaenoic and hexaenoic acids (Gruger et al., *Ibid.*, 662). A review of analytical data on marine oils led to the conclusion that the longer-chain polyunsaturated acids were structurally homogeneous to the extent that the double bonds were *cis* and had the divinylmethane arrangement and that, except in the C_{16} acids, the ultimate double bond was 3, 6 or 9 carbon atoms removed from the terminal methyl group (Ackman, *J. Fish Res. Bd. Canada 21*, 247). NMR spectroscopy showed the arrangement of the double bonds in highly unsaturated fish oils to be of the divinylmethane type, with no divinylethane structures and no terminal unsaturation (Harlan, *JAOCS 41*(9), supp. 4). The fatty acids of cod liver oil were determined by gas chromatography (Ackman and Burgher, *J. Fish Res. Bd. Canada 21*, 319). Codfish flesh fatty acids derived from the phospholipids had a high content of polyunsaturated and a low content of monounsaturated fatty acids. There was a correlation between the polyunsaturated acid contents of the liver and flesh (*Ibid.*, 367). The fatty acid composition of cod eggs resembled that of the flesh (*Ibid.*, 469). Mullet oil contained fatty acids with both odd and even numbers of carbon atoms from C_{15} to C_{20}. The odd-numbered chains amounted to more than 25% of the total. In all of the polyunsaturated acids the double bonds had the divinylmethane arrangement and were usually in identical positions (9,12 and 6,9,12) relative to the carboxyl group (Sen and Schlenk, *JAOCS 41*, 241). The fatty acids of the flesh phospholipids of South African pilchard contained large amounts of C_{22} hexaenoic, C_{20} pentaenoic and C_{16} saturated acids, smaller amounts of C_{18} dienoic, C_{18} monoenoic, and C_{18} saturated acids (Silk and DeKoning, *Ibid.*, 619). A simple, inexpensive refining procedure for sardine oil was described (Sen et al., *Food Sci. (Mysore) 12*, 189).

The Drying Process; Autoxidation

The mechanisms (Garoglio, *Riv. Ital. Sostanze Grasse 41*, 181) involved in the formation and decomposition of peroxides and of the formation of polymers during the autoxidation of oils, as deduced from studies of fatty acids and esters were discussed (Karnojitzky, *Peint. Pig. Vernis 39*(3), 144; Skellon, *Oil Colour Chem. Assoc. 46*, 1001). The formation of only 9- and 10-hydroperoxides during the autoxidation of methyl oleate supported the pi-electron-complex mechanism as opposed to the free-radical mechanism (Kahn, *Oleagineux 19*, 397). The autoxidation of methyl linoleate at 50-60C was studied by following changes in the concentration of hydroperoxides, dialkyl peroxides, carbonyl compounds, carboxylic acids, and conjugated double bonds and in the molecular weight (Dulog and Burg, *Deutsche Farben-Z. 17*(1), 21). Similar work on linseed methyl esters included studies of the effect of heating the autoxidized esters to temperatures up to 500C (Izumi and Yamada, *Yukagaku 13*, 418). Conjugated double bonds increased during the early stages of the autoxidation of linseed oil, then remained constant and finally decreased (Pokorny, *J. Inst. Chem. Tech. Prague 5-3*, 51). Hydroperoxides were more effective catalysts for the autoxidation of blown linseed oil than were other types of peroxides (*Ibid. 6-2*, 267). The products obtained from methyl docosahexaenoate (from cuttlefish oil) by autoxidation at 0C (Fukuzumi et al., *Kogyo Kagoka Zasshi 66*, 1675, 1846) and at 35C (*Ibid.*, 1320) were characterized by various analytical methods. Similar studies were made on conjugated methyl docosahexaenoate (*Ibid. 66*, 1846; *67*, 324).

Two mechanisms were proposed to explain the presence of aldehydes and formic acid in the autoxidation products of oleic acid, products which are unaffected by the autoxidation temperature up to 100C (Francois and Loury, *Chim. Ind. (Paris) 91*, 650). The formation of volatile aldehydes in soybean oil stored in air in the absence of light was discussed (Holm, *Acta Polytechn. Scand. (Chem. Series No. 21)* (1963) *Norw. Contr.* (16), 48). Studies on the volatile carbonyl compounds formed during the autoxidation of methyl erucate at 96C (Suzuki and Takeuchi, *Yukagaku 13*, 431) and of stearic, oleic, erucic and linoleic acids (Hrdlicka and Pokorny, *Papers Inst. Chem. Tech. Prague 6*, 161) and linseed and soybean oils (Hrdlicka and Pokorny, *Sbornik Vgs. Skoly Chem. Tech. Praze. Potrav. Tech. 7*, 113) at 180C produced data on the effect of time and temperature of autoxidation on the degree of saturation and chain length of the carbonyl compounds.

Although all metals in the form of their soaps had approximately the same catalytic effect on the autoxidation of polyunsaturated fats, significant differences were exhibited by metal chelates. Chelates of Fe^{++} had the greatest catalytic effect, those of Ni^{++}, Cu^{++} and Zn^{++} had an induction period, and those of Co^{++} were antioxidants (Fedeli et al., *Riv. Ital. Sostanze Grassee 40*, 300). In the case of chelates of salicyl aldehyde-ethylene diamine, the positions of Co^{++} and Zn^{++} in the above series were interchanged (*Ibid.*, 619). The catalysis of the autoxidation of linseed oil films on aluminum foil at 50C by vanadium resinates and various metallic salts and esters of orthovanadic acid was studied (Yukhas, *Periodica Polytechnica 7*(1), 1). The ascorbic acid-catalyzed oxidation of methyl linoleate proceeded at the same rate in buffers of pH 6.8 and 8.5. Addition of various sex hormones did not change the rate. Several nicotinic acid analogs and homologs increased the oxidation rate in the 6.8 buffer but not in the 8.5 buffer (Kritchevsky and Tepper, *Proc. Soc. Exp. Biol. Med. 115*, 841). In the histidine-catalyzed autoxidation of emulsified methyl linoleate, the rate of reaction was affected by the type of emulsifier (anionic or nonionic) and by the presence of certain inorganic salts. In the absence of histidine some of these additives produced different effects than in its presence (Coleman et al., *JAOCS 41*, 347). The products of autoxidation of unsaturated fatty acids finely distributed in a proteinoid substrate prepared from hide parings consisted in part of a chemical compound formed from the oxidized fatty acids and the substrate (Pokorny et al., *J. Inst. Chem. Technol. Prague 6-1*, 185). The rate of autoxidation of the fatty acids was proportional to their degree of oxidation (*Ibid.*, 195). Polyunsaturated fatty acids and their glycerides and ethyl esters were rapidly autoxidized in the presence of a proteinoid paste obtained by treating hide parings with lime and hydrochloric acid (*Ibid. 6*, 153). Peroxides accumulated in methyl linoleate during irradiation under vacuum and during postirradiation storage under oxygen. The effect of antioxidants in the irradiated material was less than in the nonirradiated ester. Propyl gallate was less effective than butylated hydroxyanisole (Chipault and Mizuno, *JAOCS 41,* 468).

The effectiveness of a mixture of 2- and 3-t-butyl-4-hydroxyanisole (BHA), 2,6-di-t-butylmethylphenol (BHT) and nordihydroguaiaretic acid (NDGA) in soybean oil and partly oxidized linseed oil was studied. At higher concentrations the first and third of these compounds acted as pro-oxidants (Pietrzyk, *Roczn. Technol. Chem. Zywnosci 9,* 81). The effect of the type and location of substituent groups on the antioxidant activity of phenols was studied (Hedenburg, *Ind. Eng. Chem. Fundamentals 2,* 265). The triethanolamine salt of linoleic acid exhibited increased stability to oxidation and the arginine and lysine salts were unusually stable (Chang and Linn, *JAOCS 41,* 780). Urea addition compounds of oleic and linoleic acids were stable to oxidation when stored at low temperatures. The monohydric alcohol esters of these acids were less effectively stabilized by addition compound formation. Linolenic acid was not stabilized by this method (Zalud et al., *Prum. Potravin 13,* 660). Herring oil of iodine value 192 produced from small spring herrings was more stable to oxidation than was oil of iodine value 130 produced from winter herrings, apparently due to the presence of efficient antioxidants in the former oil (Astrup, *Chem. Ind. (London) 1964,* 107).

Products Formed Via Double Bond Changes

ISOMERIZATION. The isomerization of methyl oleate to methyl elaidate in the presence of mercaptans was studied (Kircher, *JAOCS 41,* 351). Erucic acid was isomerized to brassidic acid by heating colza oil in the presence of selenium (Jakobowski, *Tluszczc i Srodki Piorace 7,* 260). Isomerization by heating in the presence of selenium was studied with olive, safflower and linseed oils and with the ethyl esters of oleic, linoleic and linolenic acids. Conjugation and polymerization also occurred. Glycerides reacted more rapidly and with less polymerization than did the ethyl esters. The isomerization reaction was accelerated by the presence of oxygen, benzoyl peroxide or hydrogen peroxide and was retarded by an antioxidant (BHT) (Subrahmanyam and Quackenbush, *JAOCS 41,* 275). A study of the alkaline isomerization of linoleic acid partially confirmed the theory that the *cis*-9,*cis*-12 acid was 50% converted to the *cis*-9,*trans*-11 isomer and 50% to the *trans*-10,*cis*-12 isomer. Some *trans,trans* isomers were formed (Cartoni et al., *Riv. Ital. Sostanze Grasse 60,* 482).

The effects of several variables in the production of cyclic fatty acids by heating linolenic acid in ethylene glycol in the presence of sodium hydroxide were studied. Yields were improved by increasing the solvent ration, reaction temperature and catalyst concentration and by using ethylene instead of nitrogen as the overhead gas. Ethylene apparently added to conjugated double bonds to produce C_{20} cyclic acids (Eisenhauer et al., *JAOCS 41,* 60). A continuous process for making cyclic acids from linseed oil appeared to be feasible (Beal et al., *Ibid.,* 683). One of the cyclic products from linseed oil was shown to be ethyl 11-(2-methylcyclohex-2-en-1-yl)undec-*trans*-9-enoate (Hutchison and Alexander, *J. Org. Chem. 28,* 2522).

POLYMERIZATION. Heating of *trans*-10,*trans*-12-linoleate produced a dimer by Diels-Alder reaction between 2 molecules, one acting as diene and one as dienophile, the product consisting of four isomers with a tetra-substituted cyclohexene structure (Paschke et al., *JAOCS 41,* 723). A polymerized fatty acid was produced by heating castor oil in a closed vessel in the presence of an activated clay or acid clay (Nitto Chem. Ind. Co., *Jap. 5,130/63*). Heating linseed oil at 240C for eight hours produced 10% of dimeric glycerides. Tetramers appeared after sixteen hours at 260C while longer times or higher temperatures produced even higher polymers (Fedeli et al., *Riv. Ital. Sostanze Grasse 40,* 321). Heat-bodied dimorphotheca oil dried more rapidly than the raw oil. Dehydroxylation as well as polymerization occurred when the oil was heated at 282C (Rheineck and Sobol, *Off. Dig. 34*(451), 862). Treatment of methyl oleate with stoichiometric amounts of di-t-butyl peroxide gave a noncyclic dehydro-dimer formed by carbon to carbon linkage at the 8,9,10 or 11 positions of the oleate chain (Paschke et al., *JAOCS 41,* 56). Heating of methyl linoleate or linoleic acid in the presence of iron pentacarbonyl produced a dimer (Tanabe and Hashimoto, *Yukagaku 13,* 16).

ADDITION REACTIONS. The copolymerization of styrene with methyl oleate and of styrene and acrylonitrile with methyl linoleate, methyl linolenate and conjugated methyl linolenate was studied. Styrene reacted only with conjugated unsaturation. Methyl eleostearate, with 3 conjugated double bonds, inhibited the polymerization of both styrene and acrylonitrile (Mayo and Gould, *JAOCS 41*, 25). A number of phenols and thiols were added to methyl linoleate using methane sulfonic acid as a solvent-catalyst. Some di-addition and polymerization occurred (Eisner et al., *Ibid.*, 557). In an effort to improve polycyclopentadiene films, solutions of cyclopentadiene polymers were heated with tung, dehydrated castor and linseed oils. The strongest and most water-resistant films were obtained from products containing 30% of the polymer (Chernobai, *Zh. Prikl. Khim. 35*(10), 2346). Trichlorosilane and methyl dichlorosilane were added to the double bonds of oleic, linoleic and 10-undecenoic acids, their methyl esters and methyl acetyl ricinoleate using diacetyl and benzoyl peroxides as catalysts. The products reacted vigorously with water and alcohols and polymerized on standing (Takoaka and Mihara, *J. Jap. Soc. Col. Mat. 35*(10), 439). The properties of the products obtained by treating a number of vegetable oils and derivatives with S_2Cl_2 were studied (Schiemann et al., *Deutsche Farben-Z. 17*, 408).

Oxidation. Epoxidation of butyl oleate with hydrogen peroxide, acetic acid and sulfuric acid gave a product of higher purity than did epoxidation with peracetic acid. Oleic acid and the butyl ester of tall oil were also epoxidized with the former reagents (Suhara, *Yukagaku 13*, 279). The heat stability and odor of epoxidized fatty oils were improved by oxidizing and then neutralizing the epoxidized oils (Harrison and Simmerman, *U.S. 3,150,153*).

Products Formed Via Carboxyl Reactions

The zinc-catalyzed esterification of fish fatty acids with glycerol produced 75–78% yields of triglycerides (Lehman and Gauglitz, *JAOCS 41*, 533). The kinetics of the alcoholysis of linseed oil with glycerol at 180 and 200C in the presence of Pb, Ca, Ba and Na oxides were studied (Hippe and Mauberg, *Przem. Chem. 42*(6), 309). A monoester was prepared by esterification of a polyethylene glycol with a maleic anhydride adduct of a fatty acid, esterification taking place at the anhydride group (Crecelius et al., *U.S. 3,141,897*). Moisture and weather resistant varnishes were prepared from a pentaerythritol alkyd modified with di-t-butylbenzoic acid and linseed oil or synthetic fatty acids (Izyumov and Koposova, *Lakokras. Mat. 1963*(4), 2). Lactic acid modification of linseed glycerol alkyds was more effective at shorter oil lengths, where good film hardening resulted. The products gave good industrial enamels. As primers the resins had good water and humidity resistance, and improved corrosion resistance but poorer resistance to salt water immersion (Touchin, *Paint Technol. 28*, 14). Polyesters prepared from dimeric fatty acids, maleic anhydride and propylene glycol and dissolved in styrene dried to elastic films without surface air inhibition (Penczek et al., *Plaste Kautschuk 10*(5), 262). The effect of n-lauryl mercaptan on the curing of unsaturated polyesters was studied (Mewes, *Thesis, Stuttgart 1962*, 117 pp.; *List of Accessions, Roy. Inst. Technology Library, Stockholm 1963*(147), 13).

Products Formed Via Oxirane and Hydroxyl Reactions

Vinyl monomers were prepared by the reaction between the oxirane group of epoxidized oils and the carboxyl group of vinyl carboxylic acids, for example, epoxidized soybean oil and methacrylic acid. The monomers homopolymerized to solid thermosetting resins or copolymerized with other vinyl monomers to give products ranging from viscous liquids through soft gels to hard resins (Nevin and Moser, *J. Appl. Polymer Sci. 7*(5), 1853). Vinyl ethers of conjugated soybean and conjugated linseed alcohols, homopolymerized or copolymerized with isobutyl vinyl ether and cyclopentadiene (and in some cases styrene) gave good results as metal coatings (Brand et al., *JAOCS 41*, 597). Solvent-blown rigid urethane foams prepared from a mixture of blown castor oil and triisopropanolamine had higher compressive strengths than the corresponding foams in which raw rather than blown castor oil was used. The properties of the foams could be correlated with the degree of oxidation of the blown oil (Lyon et al., *Ibid.*, 23).

Drying Oil-Resin Combinations

Varnishes prepared from nonreactive phenolic resins and heat-bodied dimorphotheca oil were comparable to those prepared with tung oil (Rheineck and Sobol, *Off. Dig. 34*(451), 862). Thermally stable oils were prepared from fatty derivatives and functional silanes (Ismail, *Fette Seifen Anstrichmittel 66*, 356). Dialkoxysilyl-fatty acid methyl esters were prepared by the reaction of methyl 11-methyldichloroundecanoate and methyl dichloromethylsilylstearate with various alcohols. Glossy, heat-resistant stable polysiloxanes were prepared by the polymerization of methyl 11-methyldialkoxysilylundecanoate at 300C (Takaoka and Mihara, *J. Jap Soc. Col. Mat. 35*(10), 439).

Analytical Methods

A review on the applications of infrared spectroscopy to the study of oils referred to autoxidation products, dimers, oxidation polymers of ethyl linoleate, epoxy compounds and drying mechanisms (Kauffman, *JAOCS 41*(8), supp. 4). The Industrial Oils and Derivatives Committee of the American Oil Chemists' Society reported on the status of development of methods for analyzing epoxidized oils, polymerized fatty acids and drying oils (*JAOCS 41*, 614). The subject of rheometry was reviewed (Date and Fukada, *Yukagaku 13*, 52).

The conjugated double bond content of autoxidized linseed oil was approximated by determining iodine values by the Hanus method and the bromine vapor method. Because conjugated double bonds reacted quantitatively in the latter method but not in the former, the difference could be used as a measure of the number of bonds of this type (Pokorny, *J. Inst. Chem. Tech. Prague 5-3*, 51). The positions of double bonds in unsaturated fatty esters and glycerides were determined by oxidation with permanganate-periodate reagent, determination of the relative amounts of dibasic and monobasic acids in the oxidation product by gas chromatography, and study of these results in combination with data on the compositions of the oils, also determined by gas chromatography (Tulloch and Craig, *JAOCS 41*, 322). Reduction of conjugated fatty acids followed by gas chromatographic analysis of the products, together with data from permanganate-periodate oxidation, was suggested as a method for the structural study of the conjugated acids (Takagi and Craig, *Ibid.*, 660). Double-bond migration and *cis,trans*-isomerization occurred during gas chromatographic analysis of methyl eleostearate. This method therefore had to be used cautiously in the study of fatty acid unsaturation (Mikolajczak and Bagby, *Ibid.*, 391). A review of the applications of nuclear magnetic resonance in the oil industry mentioned its use in the determination of the types of unsaturation present in oils (Harlan, *Ibid.* (9), supp. 4).

The titanium chloride colorimetric method was stated to be more accurate than the iodometric method for the determination of the hydroperoxide content of oxidized oils (Pokorny, *J. Inst. Chem. Technol. Prague 6-2*, 267). Recommended solvents for the determination of peroxides with sodium iodide were refluxing isopropyl alcohol for easily reduced peroxides, refluxing acetic acid-6% water for diaralkyl peroxides, and refluxing acetic acid-hydrochloric acid for stable di-t-alkyl peroxides (Mair and Graupner, *Anal. Chem. 36*, 194). Modifications in the official AOCS method for the determination of oxirane oxygen by titration with hydrobromic acid were reported (Barlow et al., *JAOCS 41*, 86).

Because of high loss of polyunsaturated fatty esters in gas chromatography the amounts of these constituents should be confirmed by other methods. There were indications that this loss could be largely eliminated by using less polar solid supports (Gerson et al., *Biochem. J. 91*, 11C). Column chromatography of free fatty acids could be carried out by using a strongly acidic (C_{54} tribasic acid) and a naphthalene-disulfonic acid additive in the liquid phase, the wall of the column being coated with a similar liquid. Another approach was to use a completely inert support material (Teflon) and butanediol succinate or trimer acid as the liquid phase (Kabot et al., *Riv. Ital. Sostanze Grasse 41*, 131). The adulteration of pumpkinseed oil with other oils could be shown by detecting the presence of foreign fatty acids using chromatography on narrow paper strips in test tubes. The presence of a small amount of rapeseed oil was shown by the presence of erucic acid, soybean or linseed oil by linolenic acid, and sunflower oil by behenic and lignoceric acids (Gorbach and Weber, *Fette Seifen Anstrichmittel 65*, 989). A silicone rubber (SE-30) was selected as the best stationary phase for the analysis of methyl epoxyoleate by gas chromatography (Herb et al., *JAOCS 41*, 222). Silicic acid-silver nitrate chromatography could be used for the quantitative determination of saturated fatty acids, the concentration and analysis of small amounts of unusual fatty acids, in combination with oxidative degradation for detecting small quantities of positional isomers, and the preparation of purified polyunsaturated fatty esters (Bhatty and Craig, *Ibid.*, 508).

The identity and purity of a number of oils, including linseed, castor and soybean, could be determined by thin-layer chromatography of either the glycerides or the fatty acids using liquid paraffin on kieselguhr as the stationary phase and acetic acid as the liquid phase (Anker and Sonanini, *Pharm. Acta Helv. 37*(5-8), 360). Two thin-layer chromatographic methods for determining the glycerides of seed oils were described. In the first method the adsorbed spots were extracted, mixed with a known amount of methyl heptadecanoate and converted to methyl esters for gas chromatographic analysis. In the second method three fractions obtained from the oil by low temperature crystallization were further separated chromatographically (Gunstone et al., *Chem. Ind. (London) 1964,* 483). The glyceride structures of a number of oils, including linseed and soybean, were determined by oxidation with permanganate-periodate, esterification of the oxidized glycerides and gas chromatographic analysis of the oxidation products (Youngs and Subbaram, *JAOCS 41,* 218, 595).

A gas chromatographic method for determining the polymer content of fatty esters using the internal standard technique was described (Zielinski, *Ibid.,* 249). Polyglycerols were determined quantitatively by paper chromatography (Zajic, *Papers Inst. Chem. Tech. Prague 6,* 179). Film-forming polymers and copolymers were analyzed by subjecting them to controlled pyrolysis and separating the products gas chromatographically (Hippe, *Polimery 8*(11), 414). The applications of various instruments in paint research included studies of bronzing, paint film yellowing, resin identification, pigment identification, polymer and oil analysis, study of wash primers, measurement of corrosion resistance and determination of pigment size (Valentine, *Peint. Pig. Vernis 39*(4), 211; (5), 295). A method was described for determining the rate of chalking of a paint by measuring the weight loss at intervals during natural or accelerated weathering (Boers, *Deutsche Farben-Z. 17*(4), 155).

Plasticizers were separated from mixtures by column chromatography and then analyzed by infrared spectroscopy (Criddle, *Brit. Plast. 36*(5), 242). Plasticizers in nitrocellulose, vinyl and acrylic lacquers were identified and quantitatively determined by programmed temperature gas chromatography (Esposito, *Anal. Chem. 35,* 1439).

Paint Technology

Results of an extensive 3-year exposure test conducted by the National Flaxseed Processors' Association showed that conventional linseed oil house paints compared favorably to latex paints (Morris, *Am. Paint J. 47*(55), 93). Studies were made of the water absorption, extensibility, breaking strength and permeability of free paint films in an effort to correlate these properties with paint failures due to blistering, weathering and cracking (Harris, *Can. Paint Var. 37*(2), 22; (3), 42, 73). A coating composition was described in which the vehicle was a coconut oil-phthalic alkyd resin (Langstroth, *U.S. 3,128,260*). Improvement of oil-modified alkyds by heating with a bivalent metal hydroxide was described (Friedsam, *U.S. 3,110,690*). A summary of new developments in lacquers referred to lacquers for various purposes made from polyurethanes, vinyl resins and acrylate resins, hydroxyethylcellulose as a protective colloid in acid- and alkali-resistant latex paints, and plasticized poly(vinyl acetate) latex paints (*Chem Progress Union Carbide, Internat. Ed. 14*(4), 1).

Nyasaland (Montana) and Fordii tung oils gave comparable results in corrosion resistant zinc chromate primers under salt spray conditions (Chatfield, *Paint Oil Colour J. 146,* 228). A corrosion-resistant paint consisted of a drying oil, alkyd resin, epoxy resin, or poly(vinyl acetate) vehicle containing 0.5–1.0% of a barium salt of cyanuric, (2-ethylhexyl)phosphate, capric, caprylic or phytic acids (Bryan and Tidridge, *U.S. 3,137,583*). Exhaustively chlorinated oleic and stearic acids were used in the manufacture of fire-retardant paints (Cheng, *Thesis, Karlsruhe, 1962,* 47 pp; *List of Accessions, Roy. Inst. Technology Library, Stockholm, 1963*(147), 11). The formulation and testing of water-resistant, intumescing fire-retardant paints was described. The three major components were the vehicle, a spumific melamine phosphate and a carbonific polyurethane (Verburg et al., *JAOCS 41,* 670). An introduction to the field of electro-deposition of paint was presented (Berry, *Paint Technol. 27,* 13). An improvement in the preparation of drying oil-impregnated porous carbon electrodes consisted of subjecting the impregnated electrode to 300 psi steam pressure and then quickly releasing the pressure (Bailey and Best, *U.S. 3,120,454*).

In corrosion-resistant primers the critical pigment volume concentration varied with the specific surface of the red iron oxide pigment. While ion permeabiltiy of such a paint gave an indication of its porosity, it was not an absolute measure of the effectiveness of the paint (Kresse, *Farbe Lack 69*(4), 255). The use of heavy metal soaps as dispersing, bodying, drying, flatting and antifouling agents in paints was summarized (Pilpel, *Paint Technol. 27,* 16), as were the uses of thickening agents in solvent- and water-thinned paints (Rabate, *Peint. Pig. Vernis 39*(6), 347).

Plasticizers

Butyl stearate, butyl oleate, butyl ricinoleate and methyl linoleate were evaluated as plasticizers in poly(vinyl chloride). As sole plasticizers, these esters had limited compatibility with the resin. In combination with dioctyl phthalate they gave improved properties over resins containing only DOP (Riser et al., *JAOCS 41,* 172). Twenty diesters of brassylic (tridecandioic) acid were evaluated as poly(vinyl chloride) plasticizers. Several had excellent low-temperature properties and light resistance (Nieschlag et al., *Ind. Eng. Chem. Prod. Res. Develop. 3,* 146). N,N-bis(2-alkoxyethyl) amides of terpenic acids, naphthenic acids, a variety of fatty acids, and the acids of animal fats, rapeseed, *Limnanthes douglassii* seed, parsley seed and hydrogenated cottonseed oils were evaluated as plasticizers in poly(vinyl chloride-vinyl acetate). Some showed good low-temperature properties but most had poor thermal stability (Mod et al., *JAOCS 41,* 781). Forty-six N-acyl derivatives of cyclic imines (containing essentially the same acyl groups as the amides described immediately above) were also evaluated in poly(vinyl chloride-vinyl acetate). These amides of 5-, 6-, and 7-membered cyclic imines showed exceptionally high plasticizing efficiencies and good compatibilities. Several of them had good low-temperature properties and antifungal activity (Mod et al., *Ibid.,* 237).

Miscellaneous Products

Nitrocellulose particles were coated with 0.02–0.5% of their weight of sorbitan trioleate (Coffee, *U.S. 3,118,797*). A valve-packing composition consisted of polymerized castor oil, 30–60%; triethanolamine, 3–7%; stiffener (carnauba wax, N,N′-ethylenebisstearamide, aluminum ricinoleate or dimethyldioctadecylammonium bentonite), 5–20%; and filler, 30–60% (Puttroff, *U.S. 3,109,744*). A composition consisting of at least 99.5% of maleic anhydride and at least 0.07% of a 10–25 carbon fatty acid was described (Gans and Russell, *U.S. 3,140,300*). The effect of temperature on the formation of polyglycerol by heating glycerol was studied. Polyglycerol did not form at 180C but formed slowly at 200 C. Sodium oxide accelerated the reaction (Zajic, *J. Inst. Chem. Tech. Prague 7-1,* 179, 191).

The Annual Review of Literature will be continued in September

• *Obituary*

C. L. Manning, Fort Worth-Southwestern Laboratories, Fort Worth, Texas, died suddenly on Saturday, July 3, 1965, following a heart attack.

Mr. Manning had been with the Fort Worth-Southwestern Laboratories since 1927 and was well known in the cottonseed and related industries.