

Report of the Literature Review Committee *

24th Annual Review of the Literature on Fats, Oils, and Detergents

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Economic Information and Comprehensive Literature

The domestic production forecast for the fiscal year beginning Oct., 1957, preliminary figures for year beginning Oct., 1956, and data for the year previous taken from *Fats & Oils Situation 188*, (29 pp.) on fats and oils are:

| | For Year Beginning | | |
|---|--------------------|----------------------------|-------------------------|
| | Oct. 1955 | Oct. 1956 (Preliminary) | Oct. 1957 (Forecast) |
| | <i>Mil. lb.</i> | <i>Mil. lb.</i> | <i>Mil. lb.</i> |
| Butter (actual wt.)..... | 1,568 | 1,548 | 1,560 |
| Lard and rendered Pork Fat..... | 2,852 | 2,627 | 2,675 |
| Edible Beef Fat..... | 312 | 321 | 350 |
| Edible Vegetable Oils (plus oil equiv. of seed exported)..... | 6,132 | 6,378 | 6,278 |
| Soap Fats and Oils..... | 3,349 | 3,165 | 3,125 |
| Drying Oils (linseed, tung, and castor oils)..... | 824 | 856 | 567 |
| Others (neats' foot, wool grease, fish liver, and other oils)..... | 31 | 36 | 40 |
| Total..... | 15,068 | 14,931 | 14,595 |

For more details on production, prices, consumption, stocks, exports, the reader is referred to the publication from which the above data are taken. For world figures on production, trade, utilization, and price of commercially grown vegetable oils and oilseed the reader is referred to the annual review on this subject issued by the British Commonwealth Economic Commission. However, the latest issue appeared in 1957 and contains data for the year 1956.

For an excellent analysis of the soap and synthetic detergent (syndet) industries the reader is again referred to the annual review of Dee Snell (*Ind. Eng. Chem. 50*, No. 1, 41A). In 1957 syndets increased 8% tonnage-wise and soap decreased 6% as compared to the 1956 data. Dee Snell analyzes trends in raw materials used, prices, and uses of the individual products. In another review on the economics of the industry it is suggested that syndets will capture 70% of the detergent market by 1960 (Hardy—*Ibid.* 49, No. 1, 95A). In this communication, operational cost data are presented to show that a 3.6 oz. bar of soap can be produced at the cost of 1.08 cents.

In a list of per capita consumption of soaps and detergents in 36 countries, Belgium leads with 28.3 lbs., U.S.A. is second at 27.8, and Switzerland third with 26.1, followed by United Kingdom at 24.3. In a few countries at the bottom of the list less than one pound per capita per year is used (Anon.—*Soap Chem. Specialties 33*, No. 3, 46). These figures should not be interpreted with respect to cleanliness of certain peoples over others, but instead they reflect that water in Belgium is hard and among other high users much goes into the textiles, metal working, synthetic rubber, and other industries. Generally, however, high usage is a mark of higher type civilization.

Information of economic significance is an integral part of communications describing various oils, fats, and groups of these, for the authors may discuss availability, cost of production and possible uses besides source and properties. The text of some of these are: terrestrial and marine animal oils (Carola—*Olii minerali; grassi e saponi colori e vernici 34*, 233, 382), lesser-known Japanese vegetable oils (Toyama & Takai—*Mem. Fac. Eng. Nagoya Univ. 7*, 161; Kashimoto—*Nippon Kagaku Zasshi 75*, 1110; 76, 664), lesser-known Indian vegetable oils (Subrahmanyam & Achaya—*J. Sci. Food Ag. 8*, 657), a description of 26 new oil plants, with yield per hectare and oil content of the seeds (Moldenower—*Przemysl Spozycy 7*, 58), fat and oil resources and industry of Brazil (Markley—*Econ. Botany 11*, 91), products from fish oils such as poly amino fatty acids, fatty alcohols, fractionated acids and alcohols, alkyds, silicones and quaternary ammonium salts (Stansby—*Comm. Fisheries Rev. 18*, No. 8, 1; 19, No. 4a, 13, 18), oil content, properties of the oil, separation and purification of the wax, unsaponifiable, etc., of sperm whale oil (Fuji, Okura, et al.—*Nippon Suisan Kabushikikaisha Kenkyusho Hokoku No. 8*, 19, 24, 75, 77, 85, 88, 91, 95), similar

information on baleen whale oil (Okura et al.—*Ibid.* 45, 51, 65, 71), castor oil and its derivatives (Litvay—*Anais farm e quim, São Paulo 8*, No. 1-2, 20; Krajekman—*Perfumery Essential Oil Record 48*, 23), lanolin (Lowler—*Ibid.* 47, 403), perilla oil (Tischer—*Fette-Seifen-Anstrichmittel 59*, 313), radish-seed oil (Prakash et al.—*J. Proc. Oil Technol. Assoc., India 11*, 79), rapeseed oil (Beare—*Food Manuf. 32*, 378), sunflower oil (Dublyanskaya & Astakhova—*Kratkii Otchet o Nauch-Issled. Rabote 1953*, 145; Choudhuri & Banerjee—*Sci. & Culture, India, 21*, 675), tall oil (Zubranska—*Przeglad Papierniczy 13*, 24), utilization of whale oil (Bezzubor—*Masloboino-Zhirovaya Prom. 21*, No. 7, 25), and uses for sperm oil (Walton—*Ing. Chim., Milan 38*, No. 205, 107). An article on marine oils gives practical reference data and results on fractional distilling methyl esters of herring oils and indicates how the fractions may be used in various industries (Vandenheuvel & Jangaard—*Can. Chem. Processing, Mar. 1957*, 40).

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*LITERATURE REVIEW COMMITTEE

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There are many other communications that could have been included in the above list, but the writer is citing them later, under some data published in each, in a section on composition and characteristics.

A résumé of work at the Italian fat and oil experimental station is published (Jacini—*Fette-Seifen-Anstrichmittel* 59, 449).

Material not derived from natural fats is increasingly taking over some of the fatty oil markets. The competition of soap versus syndets is most obvious. Inorganic gels, silicones, resins, and other materials are being used in place of soap to thicken greases; synthetic materials are replacing drying oils in coatings and floor covering; and cutting and textile fatty lubricants are practically displaced by mineral oil products. Some such trends are evident in other sections of this review. In addition, the numerous publications on manufacture of fatty material through oxidation of mineral hydrocarbon illustrate the efforts made to synthesize fats from cheaper materials (Bashkirov—*Khim. Nauka i Prom.* 1, 273; Tyutyunnikov & Perchenko—*Mastoboino-Zhirovaya Prom.* 21, No. 7, 20; Bashkirov *et al.*—*Ibid.* 23, No. 7, 24; Mankovskaya *et al.*—*Ibid.* 22, No. 6, 20; 23, No. 1, 24; Varlamov & Sipeeva—*Ibid.* 23, No. 3, 21; Mertzweiler—*U. S.* 2,802,846; Ritter & Shuman—*U. S.* 2,808,422; Bartlett *et al.*—*U. S.* 2,808,423; Hill—*U. S.* 2,815,355; Nebe—*Ger.* 857,050 Cl. 120; Sernik—*U. S.* 2,780,664; Nelson *et al.*—*U. S.* 2,795,631; Tausent—*Ger.* 834,720 Cl. 22h; Young & Cottle—*U. S.* 2,815,296; Totonnikov & Bukhshtab—*U. S. S. R.* 106,569; Mundy & Malyan—*U. S.* 2,792,410; Fronmuller & Mirra—*U. S.* 2,798,085). Recorded data on the influence of pH of the medium on fat production and characteristics of the fat produced by *Aspergillus nidulans* reflects continued interest in mold culture as a source of fat (Singh & Walker—*J. Sci. Ind. Res., India* 15C, 222).

NEW COMPREHENSIVE LITERATURE. The 23rd review of this series is published in *J. Am. Oil Chemists' Soc.* 34, 222, 264. Several reviews which collectively are of like scope are annually published by the members of the Fat and Oil Group of the British Society of Chemical Industry in *Progress of Applied Chemistry*. A new volume (No. 4) of *Progress in Chemistry of Fats and Other Lipides* contains reviews on: synthesis of fatty acids, natural occurring acetylenic acids, synthesis of glycerides, lipide waxes, lecithinases, lipide dynamics in adipose tissue, dilatometry and ultraviolet spectrophotometry of fatty acids. Each of these is written by outstanding people in their respective fields.

The new books of interest to fat, oil, and detergent chemists that came to the writers' attention during the year are:

Am. Soybean Assoc.: Soybean Blue Book 1956. Hudson, Ia. 160 pp. \$3.00.

Annuaire de l'industrie des corps gras et de la detergence. 2nd ed. Montreuil/Seine: J. Diet. 224 pp. Fr. 400.

Bezzubov, A. P.: Khimiya Zhirov. Moscow; Gosudarst. Tekhn.-Ekonom. Izdatel. Pishchevoi Prom. 1957. r. 8, k. 25.

Davidsohn, J. & Davidsohn, A.: Polishes and Cleaning Materials. 3rd ed. London; Leonard Hill. 298 pp. 21s.

Deutsche Einheitsmethoden zur Untersuchung von Fetten, Fettprodukten und verwandten Stoffen. 3. Abt. C. 90 pp. Abt. E. Glycerin. 52 pp. Stuttgart; Wissenschaftl. Verlagsgs. DM 5.

La Margarine, sa réglementation dans le monde. Fed. Internatl. Laitière et Comité Italien Lait et Dérives. Rome. 167 pp. 1000 lira.

Frezzotti, G., Manni, M., Aten, A.: Olive Oil Processing in Rural Mills. Food & Agr. Org. of United Nations. Rome. 103 pp. \$1.00.

Liberman, S. G. and Petrowskii, V. P.: Spavocnik po proizvodstvie zhirovnykh zhirov (Animal fat processing). 2nd ed. Moscow, Gosudarst. Tekh.-Ekonom. Izdatel. Pishchevoi Prom. r. 11, k. 45.

Lundberg, W. O. (Editor): Fats-Oils-Detergents Yearbook 1956. New York; Interscience Pubs. 1146 pp. \$45.00.

Manneck, H.: Die Verfahren der kontinuierlichen Verseifung und Seifenherstellung. Garmisch-Partenkirchen, Ger., Fachlit-Ermittlungs- u. Berichtsdienst. 43 pp. DM 22.50.

Manneck, H.: Herstellung und Zusammensetzung pulverförmiger Wasch-, Reinigungs- und Spülmittel. Garmisch-Partenkirchen, Ger. Fachlit-Ermittlungs- u. Berichtsdienst. 57 pp. DM 30.

Martinenghi, G. B.: Mannale per l'industria ed il commercio degli olii, grassi e derivats. Milan: C. Manfredi. 237 pp.

Rinzler: Clinical Aspects of Arteriosclerosis. Springfield, Ill. Chas. C Thomas. 318 pp. \$8.75.

Sharapov, N. I.: Novye Zhiromaslichnye rasteniya

(New fat-producing plants). Moscow. Izdatel. Akad. Nauk. S.S.S.R. 112 pp. r. 1, k. 75.

A series of papers containing comprehensive information on production, transport, storage, and processing in the fat and oil industry has continued from the last few years, and the forty-third installment appears in the November issue of *Fette-Seifen-Anstrichmittel*, the latest available to the writer.

Several symposia containing comprehensive information on certain subjects in the fat field are published in journals, some of which are available as separate publications. The broad titles of these are: Technology of Fats (*Bull. soc. hyg. chim. 45*, 89-144), Synthetic Detergents (*J. Am. Oil Chemists' Soc.* 34, 170-215), Surfactants in the Paint Industry (*Office Dig. Fed. Paint & Varnish Prod. Clubs* 23, 418-85), Fats in Nutrition (*J. Am. Oil Chemists' Soc.* 34, 559-84), and Technical Safety in the Oil Solvent Extraction Industry (*Ibid.* 431-47). Also of general interest is the report of work by the Technical Safety Committee of the Am. Oil Chemists' Soc. (*Ibid.* 108-24).

Production Processes

ANIMAL AND FISH FAT PRODUCTION. Several suggestions are made for improving animal tissue rendering processes. Dividing the jacket on the horizontal jacketed cooker used in dry rendering so that heating is only from a lower compartment of the jacket prevents burning and charring, thus producing rendered fats of lighter color (Stover—*U. S.* 2,807,634). A new technique for the Pavia rendering process comprises heating the comminuted animal tissue at 180-212°F. until moisture drops 2-15%, filtering and then pressing the residue (Pavia—*U. S.* 2,815,356). The technique induces good filtering characteristics and high quality fats are produced. A comprehensive study of various systems used in rendering whale blubber and influences of particle size, temperature, moisture, etc. indicates that grinding to 2 cm. size, heating under vacuum at 100° until 40-50% of the moisture evaporates, skimming, and pressing the residue are compatible with good yield and quality of the oil (Golovin—*Rybnoe Khoz.* 33, No. 6, 85). Rendering pork tissue under carbon dioxide atmosphere yields lard much more stable than lard produced without elimination of air (Materanskaya—*Myasnaya Ind. S.S.S.R.* 23, No. 1, 48).

A "hammer mill" has been designed for the bone rendering equipment based on liberating fat by subjecting ground bone to repeated intense impacts delivered through water (Chayen—*U. S.* 2,803,634). Such an installation capable of extracting 81-81.5% of the fat in bones and with a productive capacity of 37 kg. of fat per hour is described (Lapsin & Skrypnik—*Myasnaya Ind. S.S.S.R.* 23, No. 3, 14). Such systems are designed so that the bone residues may be used for glue production. In another system with the same aim, the ground bone is heated to 60-70° and centrifuged while hot to release the fat (Matrozova & Safonov—*Ibid.* 27, No. 5, 34; Safonov—*U. S. S. R.* 101,365). In a review of modern rendering methods, the methods of Skipin-Ijlin and Chayen are described (Kimoto—*Nippon Nogei-Kagaku Kaishi* 30, A21).

Improved separation of oil is attained in rendering herring by buffering to pH 6.3-6.5 after boiling to coagulate the proteins (Notevarp—*Norw.* 88,486). According to another patent, improved oil separation from boiled herring pulp is obtained by evaporation to 40% solids before centrifugal segregation of the oil (Aktieselskabet Limvann—*Norw.* 89,580). A laboratory survey of various techniques for production of high quality vitamin A oil from cod liver indicates that steaming the livers in water and centrifuging at 85-90° is most practical in the compromise between yields and quality (Kempa & Tomaszewaka—*Przemysl Spozywczy* 9, 271). Productions of menhaden oil (Kern—*Com. Fisheries Rev.* 19, No. 5, 15) and salmon egg oil (Kyte—*Ibid.* No. 4a, 30) using bacterial proteolysis and commercial proteolytic enzymes, respectively, to release the oils are demonstrated.

The information pertinent to butter production concerns clumping of milk fat globules, butter churns, spreadability of butter, and sterilized butter. Splitting of fat globules is at a maximum and clumping at a minimum at pH 8 and as the pH of the cream is lowered toward pH 6 splitting decreases and clumping increases (Dolby—*J. Dairy Res.* 24, 77). Thus, in butter making, cream should not be over neutralized, but also should not be too acid because excessive clumping leads to specks of "oiled-off" fat in the butter and "mealy" body. In separation of cream from milk at 10-20° the size distribution of the fat globules is not altered, whereas at 45° and above, splitting of globules is pronounced (Lipatov—*Molochnaya Prom.* 17, No. 7, 30). The texture of butter

produced in a metal churn is improved by cooling the churn with an external spray system (Humbert—*Can. Dairy Ice Cream J.* 35, No. 8, 27). Other data from the same study show that churning under vacuum reduces the volume of the butter obtained. The vacuum churning did not enhance flavor score. A butter churn in which the lump of butter is rolled to expel air is patented (Sverker—*U. S.* 2,803,529). Instruments are developed to measure spreadability and hardness of butter and are used to show that storing the cream at above 48°F. after pasteurization and storing the butter below 25°F. are comparable with good consistency in the butter (Huebner & Thomson—*J. Dairy Sci.* 40, 834, 839). Several improvements were suggested for producing sterilized butter. One process comprises adding 2-5 volumes of water to the cream, culturing, centrifuging, and pasteurizing before converting to butter (Vermeulen—*U. S.* 2,789,910). The treatment removes thermolabile substances which may contribute off-flavors. Flocculation of proteins after pasteurization of butter is inhibited by subjection to vibration at the solidification temperatures during forced cooling (Gutestam—*Swed.* 155,385). In a process for producing canned butter, the cream is canned, pasteurized at 77-82° in the cans, cooled to 38°, churned, and vigorously shaken while being cooled to 20° (van den Berg—*Conserve, The Hague*, 5, No. 11, 334). To inhibit flavor defects in such products the air should be removed from the ingredients while warm and prior to the in-can heat treatment (Hansen *et al.*—*Can. Dairy Ice Cream J.* 35, No. 10, 31).

VEGETABLE OIL EXTRACTION. The general descriptive communications on screw-press oil extractors are written on the history of development of the equipment (Burner—*J. Am. Oil Chemists' Soc.* 34, 4), improving the refining properties of the oil produced by cooling the expeller barrel (Matsuk & Vetrov—*Masloboino-Zhironaya Prom.* 23, No. 6, 13) and by cooling the oil expelled (Begdash—*Ibid.* No. 5, 34), and on measuring pressures in the expeller barrel (Markman & Shamsutdinov—*Ibid.* No. 6, 15). Descriptive communications are also written on oil and fat processing in general (Schwitzer—*Process. Eng.* 37, 377), the filtration-extraction process (Haines *et al.*—*Ind. Eng. Chem.* 49, 920), and cost analysis for small-mill filtration extraction of cottonseed (Decossas *et al.*—*Ibid.* 930).

Solvent extraction equipment based on moving carriers supplied with drains and means of recirculating solvent has been reorganized in respect to design and communication between solids desolventizer, miscella desolventizer, and vapor condenser to decrease cost and size, and increase thermal efficiency (Karnofsky—*U. S.* 2,790,708). A new continuous extraction column contains a plurality of trays in tiered relation surrounding a rotatable shaft in which solids are discharged over the edge of the trays by rake arms connected to the shaft (Karnofsky—2,811,539). In another extraction column the seed material is forced upward along a spiral path countercurrent to solvent (Glinka—*U. S.* 2,764,474). A new unit for solvent extraction with liquefied volatile hydrocarbons contains a felt-covered piston which operates in a manner to squeeze additional fluid from drained meal (Jannetti & Fimiani—*Ital.* 515,612). Other newly patented oil milling equipment include a centrifuge to handle material of paste consistency (Giacci—*Ital.* 516,218), a solvent stripper for vegetable oil miscella (Dunning *et al.*—*U. S.* 2,779,724), and one for extracted meals (Krusse—*U. S.* 2,776,894).

Beloborodov (Zhur. Priklad. Khim. 29, 1437, *Masloboino-Zhironaya Prom.* 23, No. 5, 13) designed experimental apparatus to measure diffusion extraction of oil and used it to measure rate of oil diffusion from oil, flakes, and whole seed kernels. The data are converted to formulas and extraction curves as a function of time. Comprehensive specific gravity tables developed for oil-hexane miscella are calculated from equations to serve as reference to composition at temperatures from 40 to 100°F. when the specific gravity of the pure oil and hexane are known and the specific gravity of the miscella is determined (Skau *et al.*—*U. S. Dept. Agr. ARS-72-2*, 27 pp.).

Heating during the extraction process binds some oils into the meal in a form that is unextractable (Rzhekhin & Pogonkina—*Masloboino-Zhironaya Prom.* 23, No. 1, 11). Thus, when 20.39% oil was introduced in an oil free meal, extraction results (by alcohol followed by ether extraction) after various heating were: nonheated 20.13, heated to 100-105° 19.63, and heated to 120-124° 19.35%. In decoction processes some Maillard's reaction takes place between the sugars and protein of seeds (Rzhekhin—*Ibid.* 22, No. 6, 3). Heating castor seeds 24 hours at 85° lowers the acidity of the oil subsequently extracted by 60% (Augustowska—*Acta Polon. Pharm.* 13, 483). This observation may be applied for upgrading the

seeds. Dehulling followed by heat processing of sesame seed before oil extraction improves the flavor and stability of the oil produced (Glabe *et al.*—*Food Tech.* 11, 185). Washing kamala seeds with alkali solution permits obtaining a pale-colored oil by hexane extraction (Ojha *et al.*—*J. Sci. Ind. Res., India*, 15B, 551). Without the alkali wash the oil extracted is too dark for use in pale paints or varnish. Extraction of boleko seed first at 15-20° with hexane and next at 35-40° with alcohol, acetone or ether yields two fractions containing, respectively, the greatest portion of isanic acid and the greatest portion of isanolic acid (Lambert—*U. S.* 2,800,492). It is suggested that whole peanuts be deoiled at ambient temperatures to recover oil and yield a low calorie peanut product of mild flavor for dietary uses (Willich & Feuge—*Food Tech.* 11, 332).

The extraction technique using alcohol as the solvent was kept alive in the literature by descriptions on processing nigerseed oil cake (Murthy & Rao—*Bull. Central Food Technol. Res. Inst., India*, 5, 313), rice bran (de la Cierva—*Span.* 226, 484; Rao—*Dechema Monograph* 28, 123), and cottonseed (Rao & Murthy—*J. Proc. Oil Technol. Assoc., India*, 10, 30). As basic data for such a process using 2-propanol as the solvent, the solubilities and critical solution temperatures of 14 common vegetable oils in aqueous-solvent mixtures containing 1-8.5% water have been determined (Rao & Arnold—*J. Am. Oil Chemists' Soc.* 34, 401).

The literature on trichloroethylene of interest in the oil milling field concerns the toxicity of the meal byproduct. When fed to calves or chickens such meals cause development of aplastic anemia and other pathological changes manifesting toxicity (Pritchard *et al.*—*Am. J. Vet. Res.* 17, 425, 771). The toxicity of such meals is attributed to a reaction product of trichloroethylene with the protein (McKinney *et al.*—*J. Am. Oil Chemists' Soc.* 34, 461).

Oil-bearing material (seeds) are ground to a butter-like consistency, slurried with an aqueous alkali solution at a pH optimum for the solubility of the protein, and the emulsion is mechanically broken to release the oil (Sugarman—*U. S.* 2,762,820). In this process a desirable protein byproduct is obtained from the aqueous solution by lowering the pH to the proper range. In a similar process 1.5% aqueous soap solution is used as the emulsifier, and fibrous and insoluble matter is removed before continuing to oil and protein production (Kao—*U. S.* 2,773,889).

Some communications are only of interest to olive millers. An investigation on 90 types of microorganisms isolated from stored olives indicates that *Serratia*, *Pseudomonas*, *Trichosporus*, *Pichia*, and certain molds are chiefly responsible for spoilage through hydrolysis of the oil (Cancho—*Grasas y aceites, Spain*, 8, 55). Physical and chemical problems involved in the extraction of olive oil are discussed by Herrera & Del Valle (*Rev. cienc apl., Madrid*, 11, 97). In production of the oil by centrifuging the olive paste, higher yields are obtainable by applying ultrasonic waves during the process (Cioni—*Ital.* 502,814). A new technique in the field comprises pulping and emulsifying olive drupes and subjecting to electrophoresis to break emulsion and separate oil (Castorina—*Olivicoltura*, 11, No. 7, 4; *Oleagineux* 12, 227). Other olive oil extraction literature includes patents on centrifugal (Bartolini-Salimbeni—*Ital.* 517,435) and solvent (Passarelli—*Ital.* 509,400; Ricciardi & Gualdani—*Ital.* 517,471) equipment for the purpose.

Various aspects of cottonseed milling were investigated. Oil losses and protein deterioration have been measured during storage of cottonseed at various moisture levels between 5.5 and 22% (Saleem & Hussain—*Agr. Pakistan* 7, No. 2, 105). The varieties tested in this work could be safely stored for a year when the moisture content is below 10%. Benzene extracted oils from hulled and unhulled cottonseed are equal in quality and refining methods involved (Klepikov *et al.*—*Masloboino-Zhironaya Prom.* 21, No. 7, 15). A comparison of extractions of raw, tempered, and cooked cottonseed meats shows that crude oil yields are in increasing order as listed; but, due to differences in impurities, the final yield of refined oil is the same regardless of the method of preparation of the meats (Eaves *et al.*—*J. Am. Oil Chemists' Soc.* 34, 156). A patented method for reducing the amounts of free gossypol, carotenoids, and related substance in extracted cottonseed oils and for producing oil with less tendency to undergo color reversion involves cooking the meats with sufficient aqueous alkali solution to react with substantially all the gossypol they contain (King & Thurber—*U. S.* 2,784,205). Pilot-plant data on the process are also published (King *et al.*—*J. Am. Oil Chemists' Soc.* 34, 27; Knoepfler *et al.*—*Ibid.* 61). Similar treatment for detoxification of cottonseed meal is also patented (Eagle & Bremer—*U. S.* 2,797,997).

Processing variables were studied with respect to quality of cottonseed products. Addition of moisture prior to rolling the meats reduces the amount of gossypol contained in the expressed oil, but refining loss and color of oil are increased (Wamble & Harris—*Oil Mill Gaz.* 61, No. 10, 11). Data showing values of crude cottonseed oil as affected by degrees of extraction, seed quality, preparation of meats for extraction, and original oil content of seed has been recorded as basic guide for mill operation (Eaves *et al.*—*Ibid.* 62, No. 1, 9). The course of development of colored gossypol and gossycerulin during cooking is discussed with respect to the color of the final cottonseed oil (Semendyaeva—*Masloboino-Zhirovaya Prom.* 23, No. 4, 7).

Hexane containing 0.1% triethanolamine, isobutylamine, or urea is said to be an excellent extraction solvent for cottonseed oil (Maki—*Japan* 4725-'55).

Some communications pertinent to cottonseed milling deal with the quality of the meal produced. Data by Conkerton *et al.* (*J. Agr. Food Chem.* 5, 460), show the loss of sugar, lysine, and arginine and decrease in nitrogen solubility produced during autoclaving of cottonseed meal. It is pointed out that measurement of solubility of nitrogen in the meal does not reflect the complete changes induced in cottonseed protein by heat. Feeding tests on chicks demonstrates that solubility of N of cottonseed meal in dilute lye is not a good indicator of quality of the protein and that toxicity cannot be accounted for solely on the basis of analyzed gossypol content (Eagle & Davis—*J. Am. Oil Chemists' Soc.* 34, 454). Other nutritional work with cottonseed meals suggests that low values may be caused not only by heat damage to the protein but by heat promoting a gossypol protein reaction (Baliga & Lyman—*Ibid.* 21). In this work lysine availability is improved by removing bound gossypol. When long chain (C₈-C₁₈) aliphatic amines are included in hen diets containing gossypol, the hens produce normal eggs instead of eggs characteristic with presence of gossypol in the diet (Dechary—*Ibid.* 548).

BYPRODUCT FATS. Various methods were used to separate fatty acids from tall oil. A still is designed to fractionate the oil with steam into fatty acids, rosin acids and tar (Firma W. Spangenberg & Co.—*Fr.* 1,135,270). Another still is designed for the purpose of distilling off a nondrying oil fraction (Bogoyavlensky *et al.*—*Fr.* 1,002,167). Refining the oil with phosphoric acid before distillation so as to produce light color, color stable, deodorized fractions is patented (Outterson & Eldridge—*U. S.* 2,791,577). Other means of separating the fatty constituents from tall oil are based on extracting the fatty acids with nitroalkane (Ridgway—*U. S.* 2,807,607) and precipitating the acids with urea (Gorin & Rosenstein—*U. S.* 2,785,171).

Scouring procedures for raw wool are designed so as to permit recovery of wool fat. Nonionic detergents are used by some because they are least harsh on the wool fiber. The effluent of the process can be centrifuged to recover the fat (Lievens & Bovy—*Parfums, cosmet., savons No. 138*, 27) or precipitated with alum as a sludge which is dewatered and recycled to recover the fat (Crowley—*U. S.* 2,762,631). Scouring wool with an acid nonionic detergent solution containing hexamethylenetetraamine permits efficient degreasing and improves the quality of the wool. Two papers on wool scouring are reviews (Howitt—*Proc. Intern. Wool Textile Conf.* 1955E, 315, 587; Carter *et al.*—*Congr. mondial detergence, Paris* 2, Sect. 5, 442). New processes of recovery of wool fat from wool plant effluent by aeration and skimming are patented (d'Altena—*Belg.* 518,243; Salin *et al.*—*U. S. S. R.* 104,212). Raw wool is also degreased by spraying with jets of petroleum solvents (Sinclair—*Proc. Intern. Wool Textile Res. Conf.* 1955E, 347, 588).

Wash waters from alkali oil refining are heated with lignin at 140-160°F. and acidified in a process for recovery of the fatty material contained in this waste (Foley—*U. S.* 2,784,161). The acidulation reaction in recovery of fatty material from waste soap, refining foots, etc. is accelerated by adding various synthetic dispersing agents to the reaction mixture (Sharples Corp.—*Brit.* 777,413; *U. S.* 2, 802,845).

REFINING, BLEACHING, AND DEODORIZATION. General information communications and reviews on the subject are written on: influence of commercial refining processes on heavy metal, tocopherol, and conjugated acid contents of oils (Baltes—*Rev. franc. corps gras* 4, 245), plant data on purifying miscella with a hydrocyclohexane (von Westernhagen—*Fette-Seifen-Anstrichmittel* 59, 872), refining cottonseed oil (Pilette & Bagot—*Oleagineux* 11, 373, 458, 551, 627, 715; Ramos—*Grasas y aceites, Spain* 7, 309, 8, 24), steam hydration (degumming) of oils (Litvinov—*Masloboino-Zhirovaya Prom.* 23, No. 1, 37), history of degumming and refining of cottonseed oil in miscella

(Sullivan—*Oil Mill Gaz.* 62, No. 2, 9), neutralization of babassu oil (Pinto—*Bol. tec. inst. agron. norte, Brazil*, No. 31, 207), design and operation of a commercial soybean oil refining plant, using acetic anhydride as a degumming reagent (Myers—*J. Am. Oil Chemists' Soc.* 34, 93), uses for centrifuges in the oil, fat, and soap industry (Möller—*Fette-Seifen-Anstrichmittel* 59, 345, 465), construction and use of acid-resistant concrete containers for storing oils and fats (Kozin & Zhuravlev—*Sbornik Nauch. Rabot Moskov. Inst. Narod. Khoz.* 1954, No. 5, 24), and intermittent and continuous deodorization methods (Stage—*Fette-Seifen-Anstrichmittel* 58, 358).

Degumming a lecithin-containing oil with water containing small amounts of lower aliphatic mono- and dibasic acids provides an economical means of obtaining break-free oils which may not require additional refining and the resulting oil is in proper condition for deodorization (Hayes & Wolff—*U. S.* 2,782,216). Use of tannic acid similarly permits good separation of the aqueous phase and the color of the oil is improved (Hayes & Wolff—*U. S.* 2,792,411). Coloring matter, principally carotenoids, is removed from safflower oil by hydrating the oil-miscella at 75-200°F. for 5-30 minutes (Christensen—*U. S.* 2,786,859). A method for colored bodies in cottonseed oil is based on dispersing 40° Bé caustic therein and separating the mixture (Williams—*U. S.* 2,803,636).

With a new alkali refining process, oil-miscella is treated with caustic soda solution, homogenized, and centrifugally separated (Cavanagh—*U. S.* 2,789,120). In a continuous system, system, the oil-miscella is treated with caustic solution in the presence of a third solvent, isopropyl alcohol, and separation is by decantation (Vaudervoort—*Belg.* 516,203; *U. S.* 2,786,858). A new alkaline composition for refining edible oils comprises five parts of kaolin and two parts of magnesium oxide (Arranz—*Span.* 226,657). Mono- and diglycerides are removed from alkali refined oils with a treatment with lower aliphatic dicarboxylic acids or their anhydrides (Toyama *et al.*—*Japan* 2035-'55).

A study of variables in continuous caustic refining shows, generally, that losses increase with increased concentration of caustic, excess caustic, and prolonging contact time (Naudet *et al.*—*Rev. franc. corps gras* 4, 142). Washability of neutralized oils; i.e., removal of soap, is improved by treatment with phosphoric acid (Braae *et al.*—*J. Am. Oil Chemists' Soc.* 34, 293). The technique is particularly applicable to linseed and rape oils because some soap developed in these during alkali refining is not easily washed out with water.

Means other than neutralization with alkali are used to deacidify and refine crude oils. A new method for removal of color bodies, free fatty acids, and other impurities from soybean oil by dissolving in low boiling solvent under para-critical conditions to separate refined oil, and subsequently removing remaining impurities by steam is patented (Passino—*U. S.* 2,802,849). Ternary, fatty acid-oil-alcohol, solubility data are recorded as a basis for extracting free fatty acids from peanut oil with methanol (Venkataramen & Laddha—*Trans. Indian Inst. Chem. Engrs.* 8, 42), and from peanut and mowrah oils with ethanol (Cama *et al.*—*J. Proc. Oil Technol. Assoc., India*, 9, 50). Rolling oil, palm oil used in rolling steel, is recovered by emulsification with water and either extraction with solvent (Kovacs—*U. S.* 2,807,531) or breaking emulsion with mineral acid and heat (Kovacs—*U. S.* 2,807,868). Free fatty acid precipitation technique with urea as the precipitant is worked-out for peanut oil of 1-10% and 13-25% acidities, respectively (Loury & Lechartier—*Rev. franc. corps gras* 4, 82, 372; Loury—*Oli minerali, grassi e saponi, colori e vernici* 34, 46). Anion exchange resins containing quaternary ammonium groups remove free fatty acids, gums, and flavoring agents from crude fatty oils (Boldingh—*Brit.* 762,362; Chasanov *et al.*—*U. S.* 2,771,480).

Oil bleaching capacity and methods of activation are described for the following: Saurashtra earths of India (Gohil—*Oils & Oilseeds J., India*, 9, No. 6, 9), Hyderabad earths (Joshi *et al.*—*J. Sci. Ind. Res., India*, 16A, 179), decalcinated chalk (Waksumundzki & Barcicki—*Ann. Univ. Marie Curie-Sklodowska, Sect. AA*, 8, 1), and carbon made from hulls of neem, castor, and mahua seed (Prakash *et al.*—*J. Proc. Oil Technol. Assoc., India*, 11, 58). With oils containing iron soaps, the bleaching with active earths is improved by adding 0.5% concentrated sulfuric acid solution to the oil before treatment with the earth (Yamada & Komatsu—*Abura Kagaku* 5, 296). In such a process or when sulfuric acid activated earths are used on castor oil, the hydroxyl value of the oil is reduced (Naudet & Perrot—*Rev. franc. corps gras* 4, 549). The montmorillonite type active bleaching clays, which are efficient bleaches for petroleum, have a high adsorptive capacity for both free fatty acids and glycerides which renders them in-

active for bleaching glyceride oils (Jancik—*Chem. Zvesti* 11, 267). Chemical oil bleaching processes are described in which the chemicals used are potassium permanganate (Palit—*Chemistry & Industry* 1957, 1011), combinations of alkali chlorites and alkali phosphates (Soc. d'electrochem. electromet. et acieries elect. d'Ugine—*Fr.* 1,008,761), and mixtures of chromium trioxide, hydrogen peroxide, and alkali (Elston—*U. S.* 2,795,595). Fine zinc granules which have been soaked in copper sulfate solution, decolorize warmed palm oils (Cres—*Fr.* 1,000,296).

In a patented deodorization tower the oil is lifted from tray to tray by the steam that is used for the deodorization (Suriano—*U. S.* 2,804,427). In another equipment the oil is submitted to continuous steam-vacuum distillation (Ballestra—*Ital.* 512,797). Hydrogenation odors are removed from hardened oils by treatment with a small amount of phosphoric acid at 60–95° (Merker—*U. S.* 2,783,260).

The influence of the common series of refining steps on oils has been studied spectrophotometrically. The data developed by Kurita (*Abura Kagaku* 5, 347) are used to show the effect of extraction, deacidification, bleaching, and deodorization on the structure of unsaturated acids (presence of conjugation) and on alterations in or disappearance of carotenoids and chlorophyll. In similar work on peanut oil by Danguouan & Debruyne (*Rev. franc. corps gras* 4, 478) the greatest change in spectrum is the development of a high maximum at 2680 Å during the bleaching. This maximum does not develop in unoxidized oils or if the oxidized compounds are removed on alumina before the bleaching. The maximum is attributed to geometric alterations rather than secondary products of oxidation as reported elsewhere.

A fixation of color in cottonseed oil in a manner that it is not removed by standard refining and bleaching method is attributed to interaction of gossypol with the glycerides by an ester exchange reaction (Berardi & Frampton—*J. Am. Oil Chemists' Soc.* 34, 399). In this work it is observed that gossypol added to refined and bleached oils, tripelargonin, or ethyl acetate disappears after a lapse of a short time. This reaction results in production of a red color in the product that resists removal. In dark crude cottonseed oil, part of the color arises from interaction of gossypol and amino acids impurities from seed proteins (Dechary—*Ibid.* 597).

WINTERIZING, SPLITTING, AND FRACTIONATION. Descriptive and review communications on this subject are written on: non-catalyzed continuous splitting of fat (Sheherbakov & Sokolov—*Masloboino-Zhirovaya Prom.* 23, No. 3, 29), splitting of glycerides and distillation of fatty acids (Naudet—*Rev. franc. corps gras* 4, 433; Garrigue—*Ibid.* 365), automatic control for splitting and distillation (Marecaux—*Ibid.* 359), and distillation (Wittka—*Seifen-Öle-Fette-Wachse* 82, 227, 265, 290, 330, 382; Dmitrievskaya—*Masloboino-Zhirovaya* 23, No. 2, 27).

Fatty acid and component glyceride analyses data are reported for American and South African dent corn oils and their winterized fractions (Takeda *et al.*—*Abura Kagaku* 6, 13). Partition of castor oil between petroleum ether and 96% alcohol is a means of obtaining fractions high in triricinolein (Naudet & Perrot—*Rev. franc. corps gras* 4, 547). Furfural extraction technique is demonstrated on linseed, turtle, cod liver, avocado, and sweet almond oils, giving the characteristics and vitamin A content of the fractions and suggesting application for the fractions (Rovesti—*Riv. ital. essenze profumi, piante offic., oli vegetali, saponi* 38, 547). A new technique for furfural extraction as means of segregating unsaponifiable, edible oil fractions and fractions for technical purposes is patented (Inst. rech. pour huiles de palme et oleagineux—*Fr.* 1,009,597). Ternary systems of cod-liver oil-fatty acids-furfural and cod-liver oil-furfural are discussed in regard to segregation of a high iodine value fraction and fractions high in vitamin A (Lopez—*Anales real espan. fis. y quim.* 52B, 207).

Fukuzumi & Koyama (*J. Am. Oil Chemists' Soc.* 34, 500) studied the Twitchell splitting of coconut and soybean oils at 35°. Degree of splitting of the latter is greater when the reagent is dissolved in the oil, whereas the former splits easier with the reagent originally dissolved in the aqueous phase. Autoclave splitting of fat by emulsifying with a colloidal silica gel solution and heating to 180° is patented (Quelet & Duffau—*Fr.* 1,010,729). The fatty acids of cottonseed oil refining foots are economically recovered by separating fatty fraction by treatment with mineral acid, steam distilling off the free fatty acid and recovering the fatty acids in still residue by the Twitchell process (Paquet—*Oli minerali, grassi e saponi, colori e vernici* 33, 270).

An apparatus for autoclave splitting and separation of

products according to specific gravity is patented (Gel'perin & Solopenkov—*U. S. S. R.* 105,283). Operating conditions for hydrolysis of castor oil with water in excess, in which a 2-phase system is maintained, is also patented (Lawrence & Becher—*U. S.* 2,801,257). A study of losses in autoclave splitting indicates that less than 0.1% fatty acids are lost, whereas appreciable glycerol is lost through conversion to formic acid (Irodov—*Masloboino-Zhirovaya Prom.* 22, No. 6, 13). Deaeration of both the oil and steam prior to hydrolysis reduces the losses.

A 2-compartment still for fatty acid having a prestripping zone designed to be very compact and economical to make is patented (White & Parker—*U. S.* 2,776,938). A molecular still operating on the principle of the dropping film is demonstrated with data on distillation of sunflower, linseed, soybean, and cottonseed oils (Kozin & Zlatopol'skaya—*Sbornik Nauch. Rabot Moskov. Inst. Narod. Khoz.* 1954, No. 5, 35). Some fatty acids are recovered from fatty acid still residues by saponification, extraction with water, acidification with sulfuric acid, and distillation of the raw material (Cavasino—*Ital.* 501,610).

In a thermal diffusion column where one plate is heated with low pressure steam and another cooled with tap water, triglycerides move toward the top while glycerol and monoglycerides move toward the bottom (Seelbach & Quackenbush—*J. Am. Oil Chemists' Soc.* 34, 603). With brominated mixed fatty acids there is separation of brominated and nonbrominated acids; and with polymerized oils polymeric substances concentrate toward the bottom.

Qualitative mutual solubilities of 35 solvents and their solubilities in oleic acid, plus mutual solubilities and tie-line data at 25° for oleic acid with 5 pairs of immiscible solvents are recorded as fundamental information for liquid-liquid fractionation of fatty acid mixtures (Sample *et al.*—*Chem. & Eng. Data Ser.* 1, No. 1, 17). Phase behavior of palmitic, stearic, and oleic acids in several chlorinated hydrocarbon solvents is determined at temperatures of -20, -10, and 10° and is used in designing a process for separation of oleic acid from the saturated acids (Preckshot & Nouri—*J. Am. Oil Chemists' Soc.* 34, 151). Fatty acid mixtures are also fractionated by dissolving in methanol and cooling in stages while separating the fatty acid phase after each cooling increment (Inouye & Sato—*Japan* 4726-'55). Mixed fatty acids can be congealed, finely divided, and the most unsaturated components extracted with organic solvent (N. V. Koninklijke Stearine Kaarsenfabrieken Goudo-Apollo—*Dutch* 79,195). The technique is used for isolation of technical stearic acid from linseed oil. Converting mixed fatty acids to their acetamide permits easier segregation by crystallation (Skau—*U. S.* 2,816,903). This technique is demonstrated for the purification of stearic, palmitic, myristic or lauric acids (Magne *et al.*—*J. Am. Oil Chemists' Soc.* 34, 127). A similar technique, in which the fatty acids are converted to the cyclohexylamine or benzylamine salt, is used to separate palmitic acid from the fatty acids of animal, vegetable or marine oils (Magne *et al.*—*U. S.* 2,791,596). The diactylenic acid, isamic acid, is isolated from isanic oil by saponification and extracting the soap with acetone or isopropyl alcohol (Vries—*U. S.* 2,789,993).

Other commercial methods of segregating fatty acids are based on precipitation with urea. A single precipitation of linseed oil fatty acids in methanol permits obtaining a linoleic concentrate containing 84% of the acid and when precipitating from a methyl ester mixture the concentrate contains about 82% linolenate (Parker & Swern—*J. Am. Oil Chemists' Soc.* 34, 43). Two precipitations of tobacco seed fatty acids, first to remove saturated acid and second to fractionate the liquid acids, yield oleic acid of 94–5% purity and linoleic acid of 92–3% purity from the second fractionation (Gupta & Aggarwal—*J. Proc. Oil Technol. Assoc., India*, 10, 34). A technique is designed for concentrating linoleic acid to 93% purity with a yield of about 30% from safflower oil fatty acid (Narayan & Kulkarni—*Ibid.* 9, 26). Data are recorded on the composition of fatty acids precipitated from safflower oil and beef tallow in ethanol with different amounts of solid urea (Subrahmanyam & Achaya—*J. Sci. Ind. Res., India*, 16B, 269). Similar data are recorded for peanut oil and beef fat using methanol as the solvent (de Luca—*Rev. fac. cienc. quim. Univ. nacl. La Plata* 28, 51). The urea precipitation technique is applied to the wax alcohols and fatty acids of sperm whale oil and data is recorded as a basis for applying the method to this oil (Abe & Watanabe—*Yushi Kagaku Kyokaiishi* 3, 164; 5, 152, 349). One technique for applying the method is patented (Schlenk—*Ger.* 855,559 Cl. 120). Many saturated and unsaturated acids and their methyl esters were precipitated with solid urea from methanol solution at several temperatures and the composition of each precipitate is recorded as basic information for the technique (Gupta & Aggarwal—*J. Sci.*

Ind. Res., India, 15B, 473). The crystal structure of the urea complexes as determined by x-ray studies are discussed with respect to possible complex formation, their stability, and composition (Smith—*Am. Chem. Soc. Symposium 33, 5*; Schlenk—*Ibid.* 35).

The urea precipitation process can be applied in a liquid-solid countercurrent distribution technique. Such a system using a series of 25–500 cc. Erlenmeyer flasks containing 10 g. of urea each and passing the methanol-fatty acid solution successively through the flask is described (Sumerwell—*J. Am. Chem. Soc.* 79, 341). Here data are presented as to how the various fatty acids become distributed among the various flasks and data are also presented on the fractionation of salmon egg fatty acids.

The α -monoglycerides of saturated even-carbon fatty acids from caprylic to stearic form inclusion compounds with urea. The preparation of pure adducts of these, crystal size, form, and other characteristics are described (Aylward & Wood—*J. Applied Chem.* 7, 583). Diglycerides also form urea complexes, but their composition and characteristics, particularly shape, is quite different than that of urea complexes of fatty acids, alcohols, or monoglycerides (Moreno *et al.*—*Grasas y aceites, Spain, 7, 235*). This is attributed to folding of the chain.

BY-PRODUCTS OF OIL PROCESSING. Rice bran wax is isolated from rice oil tank settlings (Feuge & Cousins—*U. S. 2,802,844*), filter mud from rice oil (Deicke—*Sugar Cane Technol. 24th Conf. Innesfail, Queensland 1957, 115*), and from alkali-foots of the oil (Tsuchiya & Mamura—*Tokyo Kogyo Shikensho Hokoku 52, 4*), by various solvent techniques. The wax is also obtained from rice oil by precipitation through cooling (Yamamoto & Hidaka—*Japan 2382-'55*). This wax is similar to carnauba wax.

When sesame seed oil is extracted with acetonitrile, the miscella on cooling separates to form two phases, the upper containing the unsaponifiable (Purdy & Kneeland—*U. S. 2,786,063*). This unsaponifiable matter is used as an insecticide synergist. The unsaponifiable matter of shark liver oil contains oleyl alcohol as the chief component and has been steam distilled to yield a relatively pure oleyl alcohol product (Mitsunaga *et al.*—*Aubura Kagaku 5, 355*).

Sterols are recovered from fats and oils by converting them and oils to alcohol esters, fractionally distilling, and further purifying the sterol fraction by liquid-liquid extraction and fractional crystallization (Roemheld—*Ger. 833,642, Cl. 30h*). Several new esters of stigmaterol and a new process for the isolation of stigmaterol of about 88% purity from soybean sterols via the α -naphthylcarbamate are described (Campbell *et al.*—*J. Am. Chem. Soc.* 79, 1127). High purity stigmaterol is prepared by countercurrent selective extraction of stigmaterol-sitosterol mixtures (soybean sterols) using solvents in which stigmaterol is less soluble than sitosterol (Upjohn Co.—*Brit. 774,466*).

Tocopherols are recovered from deodorization sludges by saponifying, removing glycerol, esterifying the remainder with ethanol, distilling, dissolving in acetone, cooling to -20° , and filtering, and the filtrate is evaporated to recover tocopherols (Brokow—*Brit. 774,855*). A Russian commercial method of production of phosphatides is described (Baglai *et al.*—*Masloboino-Zhirovaya Prom. 23, No. 7, 7*). Phosphatidyl-ethanolamine is extracted from the alcohol insoluble fraction of phosphatides by mixing with water saturated chloroform; adding methanol, filtering and then precipitating the compound with lead acetate (Scholfield & Dutton—*U. S. 2,801,255*).

HARDENING OILS. Priority for the discovery and the initial industrial utilization of catalytic hydrogenation of fats is being claimed for the Russian scientist, M. M. Zaitsev (Nesmelov—*Trudy Kazan Khim. Tekhnol. Inst. im. S.M. Kirova 1951, No. 16, 61*). Two methods for continuous hydrogenation of oils are based on mixing proportions of oil and catalyst, and passing them through a reaction zone (Bollens—*U. S. 2,762,819*; Deryabin *et al.*—*Masloboino-Zhirovaya Prom. 23, No. 6, 22*).

Preliminary work in applying a mathematical equation developed earlier to hydrogenation with Raney nickel is recorded (Vandenheuvel—*J. Am. Oil Chemists' Soc.* 34, 12). Here the selectivity in hydrogenating oleate and linoleate is constant even though catalyst activity declines. Hydrogenation of oil using copper-chromium oxide catalyst is zero order in respect to the molecule hydrogenated, but the rate decreases owing to reversible poisoning of the catalyst (Miyake & Shimamura—*Bull. Chem. Soc. Japan 29, 611*).

In a study of operation variables, it is shown that selectivity is only slightly affected by temperature; both selectivity and isomerization decrease with increasing pressure; catalyst con-

centration has little effect on either selectivity or isomerization; low agitation results in more selectivity and isomerization than high agitation rates (Eldib & Albright—*Ind. Eng. Chem. 49, 825*). A study of the ozonized oxidation fission products of partially hydrogenated methyl oleate indicates that during isomerization there is an equal migration of the double bond in both directions (Knegtel *et al.*—*J. Am. Oil Chemists' Soc.* 34, 336). Presence of moisture in the oil during hardening is said to favor production of "hydrogenated flavors" in the fat (Yakubov—*Masloboino-Zhirovaya Prom. 23, No. 7, 12*). A patented method of inhibiting hydrogenation odor comprises steam deodorization and promptly hydrogenating or holding under inert gas until hydrogenation (Brown & Merker—*U. S. 2,773,081*).

The accumulation and disappearance of tri-, di-, and monoenoic, and disappearance of tetra-, penta-, and hexaenoic acids are recorded during the course of hydrogenation of sardine oil with 2% nickel catalyst at 180° (Takeda & Toyama—*Aubura Kagaku 6, 10*). In similar work on rapeseed oil, characteristics such as melting point, consistency, iodine number, etc. are recorded (Nakazawa *et al.*—*Ibid.* 5, 292). Hardened peanut oil is a more suitable substitute for cocoa butter in the manufacture of chocolate candy than hardened cottonseed, sunflower or sesame oil (Vengerova & Askinazi—*Masloboino-Zhirovaya Prom. 22, No. 8, 19*). Criteria in this work are hardness and consistency of the chocolates.

Products of low iodine value and hydroxyl value can be readily obtained by hydrogenating castor oil at atmospheric pressure, 220° , and using 1.0% Raney nickel catalyst (Sreenivasan *et al.*—*J. Am. Oil Chemists' Soc.* 34, 302). Hydrogenation at room temperature, at pressures of 40 lbs./sq. in. with alcohol as solvent produces a product rich in monohydroxy stearic acid.

Lanolin is converted to a white, less acidic and odorless product more suitable for pharmaceutical and cosmetic preparations by hydrogenation at high pressures (Esperis Soc. anon.—*Ital. 520,525*; Fayaud—*Am. Perfumer Aromat. 68, No. 4, 48*; Miyakawa *et al.*—*Osaka Kogyo Gijutsu Shikenjo Iho 7, 207*).

Descriptive information and efficiency aspects are recorded on hydrogenation catalysts: made by a special controlled reduction of nickel carbonate on kieselguhr (Takumi & Kuwada—*Japan 4368-'56*), a copper oxide-chromium trioxide mixture (Hess & Schulz—*U. S. 2,782,243*), Raney nickel prepared by leaching aluminum-nickel alloy with caustic at 120 – 130° (Sokol'skii & Voloshin—*Izvest. Akad. Nauk Kazakh, Ser. Khim 1956, No. 10, 63*), and nickel prepared by the reduction of nickel formate at 450 – 485°F . (Nanavati & Aggarwal—*J. Proc. Oil Technol. Assoc., India, 12, 13*). Electrolytic methods for the manufacture of catalysts are reviewed (Tyutyunnikov & Novitskaya—*Masloboino-Zhirovaya 23, No. 3, 15*). Copper and nickel are removed from hydrogenated fatty acids by treatment of the melted acids with strong mineral acids followed by treatment with montmorillonite clays (Opie—*U. S. 2,795,543*). A subsequent distillation or a water wash is not required.

Tests on hydrogenation with hydrazine as the hydrogen donor show: that elaidic and reicinic acids can be hydrogenated in ethanol solution at 50° ; linoleic acid is hydrogenated more readily than oleic acid; and in hydrogenation of eleostearic acid no appreciable amounts of stearic acid are formed until about 75% of the eleostearic is partially hydrogenated (Aylward & Rao—*J. Applied Chem.* 6, 559; 7, 134, 137). In similar work with tetralin solution as the hydrogen donor, temperatures of 180 – 250° , and various metal oxide mixtures as catalyst, linoleates are converted to oleates and the latter remain unaffected (Sokol'skii & Gumarova—*Doklady Akad. Nauk. S.S.S.R. 111, 609*).

The extent of elaidinization of oleic acid with 0.3–0.5% selenium catalyst at 200° , and for different reaction periods is recorded in a study of the rate of reaction and as basic information for hardening oils by the process (Mehta—*Indian Soap J. 21, 37*). This not only converts the *cis* to the *trans* form, but also involves shifting of the double bond. Fitzpatrick & Orchin (*J. Am. Chem. Soc.* 79, 4765; *Univ. Microfilms, Publ. No. 19287, 102 pp.*), suggested that the reaction proceeds via π -complex formation between oleic acid and selenium, resulting in solution of the metal, the complex undergoes isomerization, selenium reacts to a different species in which it is catalytically inactivated, forming a different complex. The reaction is considered reversible.

INTERESTERIFICATION, PARTIAL GLYCERIDES AND ALCOHOLYSIS. Review and descriptive communications on these subjects are written on: chemistry of esterification and the transesterification with fatty substances (Jacquain—*Rev. fermentations inds. et aliment 12, 76*), direct interesterification of lard (Placek &

Holman—*Ind. Eng. Chem.* 49, 162), and mechanism of alcoholysis of vegetable oils (Zcidler *et al.*—*Zhur. Priklad. Khim.* 29, 1275; Shkol'man & Voroshilova—*Ibid.* 1531), continuous production of methyl ricinoleate (Semenov—*Masloboino-Zhirovaya Prom.* 23, No. 6, 44).

A newly patented interesterification process for lard is based on using a dry suspension of alkali in oil as the catalyst (Thomas Hedley & Co.—*Brit.* 779,464).

Most current activity in the manufacture of partial glycerides is on preparation of concentrated monoglycerides. Monoglycerides of high purity are prepared by reacting glycerol with triglyceride, dissolving in gaseous hydrocarbons, and adjusting to temperature that induces separation of the monoglycerides (Miller—*U. S.* 2,759,954). Other residual esters in this process are reacted with more glycerol and the fractionation is repeated. When the reaction is carried out in tertiary butyl alcohol and the moisture is added, the monoglycerides separate into the aqueous layer (Sully—*U. S.* 2,789,119; *Brit.* 763,474). Neutralizing the catalyst in a reaction mixture prepared from glycerol and oil before cooling yields products containing over 45% monoglycerides (Mares—*Czeck.* 85,770). Data are also recorded on preparing enriched monoglyceride fractions by molecular distillation of reaction mixtures (Szeplaky—*Fette-Seifen-Anstrichmittel* 59, 505).

Products rich in monoglycerides (78%) and triene conjugation (63%) are obtained by reacting dry tung oil and anhydrous glycerol in pyridine at 80° using 2% sodium methoxide catalyst (McKinney & Goldblatt—*J. Am. Oil Chemists' Soc.* 34, 585). This product is useful as an emulsifier for insecticidal sprays because after drying it retards removal of spray residue. A study of glycerolysis of linseed, coconut and sesame oils in the presence of calcium oxide shows that saturated acids are transesterified more easily than the unsaturated acids; on alcohol extraction separation is based on degree of esterification rather than on unsaturation; and diglycerides form urea adducts more easily than do monoglycerides (Mehta & Shah—*J. Am. Oil Chemists' Soc.* 34, 587). Partial fatty acid esters of nonreducing sugars are prepared by an interesterification process in which compounds containing a sulfoxide group are used as catalyst (Huber & Tucker—*U. S.* 2,812,324). In a method for making fatty acid mono esters of glycol, the formation of diester is avoided by reacting the glycol used with boron trichloride to form boron triglycol and reacting this with the fatty acid (Hartman—*J. Chem. Soc.* 1957, 1918). Fatty oil is also alcoholized with polyhydric alcohol in the presence of a moisture entrainment agent, under condition whereby distillation of the entrainment agent and moisture takes place (Tess—*U. S.* 2,806,046).

New methods for preparation of lower alcohol esters of fatty acids are based on: continuously passing alcohol and fatty acid through a reaction zone at 100 kg./sq. cm. and 250° (Bracouier & Arnould—*U. S.* 2,759,955), use of small amounts of caustic and urea as the catalyst for the reaction (Rigamonti *et al.*—*Ann. chim., Rome*, 47, 549; *Olii minerali grassi e saponi, colori e vernici* 34, 95), and preparation by reaction of soap, sulfuric acid, and ethanol under reflux conditions (Hartman—*J. Am. Oil Chemists' Soc.* 34, 165).

Products (Excepting Detergents)

HOUSEHOLD, PHARMACEUTICAL AND COSMETIC FAT PRODUCTS. Hard butter, suitable for distribution in tropical climate, is made from butter fat and saturated fatty acids by interesterification followed by distillation of the released lower fatty acids (Cochran & Ott—*U. S.* 2,783,151). A powdered butter or margarine comprises emulsified fat 40 parts, casein 3, sucrose 2, and various salts 1 part, and is made by drying the emulsion mixture to a powder form (Kozin & Khomutov—*Masloboino-Zhirovaya Prom.* 21, No. 5, 21). A stable mixed butter and fruit bread spread is made by kneading butter with fruit purée at 40° and neutralizing with sodium bicarbonate (Casas—*Spain* 226,371). Synthetic β -carotene and coal tar are equally suitable for coloring butter with respect to flavor and color stability, the former however, also supplies 3800 I.U. of vitamin A activity per lb. of butter (Riel & Johns—*J. Dairy Sci.* 40, 193).

A Japanese method of making margarine comprises emulsifying milk with vitamin emulsion additive, spraying the margarine fat into this, fermenting and churning (Oshiba & Oshiba—*Japan* 1385-56). In a newly patented continuous margarine making system, the mix is slurried, blended with more mix, reslurried, and a portion of the slurry is recycled into the system with new mix (McGowan *et al.*—*U. S.* 2,797,164). In studies on making margarine from rapeseed oil, processes and characteristics such as processing of the seeds,

refining and hardening of the oil, oil acidity, heavy metal content, chemical and physical characteristics, are appraised with respect to the quality of the final margarine (Czaplicki *et al.*—*Prace Inst. Badawczych Przemyslu Rolnego i Spozywezego* 5, No. 3, 1). The data produced are fundamental to the use of this oil in margarine making. Studies and methods are also published for making margarine from palm oil (Kellens—*Bull. Agr. Congo Belge* 47, 1611) and beef fat (Guex—*Die Fleischwirtschaft* 8, 494). The lactones of δ - and ϵ -hydroxycarboxylic acids containing 9-14 carbon atoms are prepared for use in imparting a butter flavor to margarine and other food products (Boldingh *et al.*—*Brit.* 748,661; Taylor & Weller—*Brit.* 748,801). A study of suitable coloring ingredients for fatty foods shows that ethyl bixin and β -carotene are suitable for margarines, including those which contain vitamin A, but in frying fats and popcorn oils only the certified coal tar dyes resist processing conditions (Geminder & MacDonough—*J. Am. Oil Chemists' Soc.* 34, 314).

Many fatty products are prepared for baking. A shortening comprising partially hydrogenated oil plus small amounts of very hard hydrogenated fat crystals in the β -phase retains plasticity over a large temperature range, even after prolonged storage (Lutton—*U. S.* 2,801,177). Blends of natural lard and "rearranged lard" used as shortening produce higher pound-cake volumes than either component used alone (Vander Wal *et al.*—*U. S.* 2,763,556). A newly patented shortening comprises partly acetylated triglycerides which are heat treated with titanium alcoholate catalyst to modify crystal structure (Brokaw—*U. S.* 2,808,419). A new baking emulsion contains fat, moisture or milk, milk solids, and emulsifier; the oil phase being continuous to yield a plastic product (Struble & Stephens—*U. S.* 2,787,550). An opaque to translucent liquid shortening is composed of liquid glyceride and suspended therein, is 2-10% of fully saturated glyceride, at least 80% of which is in the β -phase (Andre & Going—*U. S.* 2,815,286). Fatty acid lactylates or salts thereof are added to shortening to reduce staling of baked products (Thompson & Budde-meyer—*U. S.* 2,789,992). Sorbic acid is used similarly as a fungistat (Mehlich *et al.*—*Bakers' Dig.* 30, No. 5, 46). Metal soaps added to cake mixes stabilize the whipped cake batter foams (Pinucane—*U. S.* 2,776,893).

A patented pie crust mix contains 25-42% shortening (Callaghan—*U. S.* 2,791,506).

The American Society for Testing Materials "Grease Worker," which is a shearing apparatus, is modified to serve as a perforated disk viscometer for determining the flow properties of shortening (Loska & Jaska—*J. Am. Oil Chemists' Soc.* 34, 495). A household electric mixer is applied for plasticizing fat samples in the pound cake baking test (Steffen & Vander Wal—*Ibid.* 159). This laboratory plasticizer induces characteristics similar to those of plant equipment.

A study of influence of edible phospholipides and other emulsifiers indicates that they improve the elastic-plastic properties of the batter and porosity of the cake (Antokol'skaya—*Khlebopekar i Konditer. Prom.* 1, No. 4, 20). In baking bread containing phospholipides, adjustment should be made in procedure to compensate for decreased gas production and accelerated acid production or fermentation (Puchkova & Chistova—*Trudy, Moskov. Tekhnol. Inst. Pishchevoi Prom.* 1956, No. 4, 96).

Practical information for the baking and milling industry is evolved from studies on the role of lipides in baking. Natural lipides of defatted flour influence the baking properties and bread quality in a manner that is not reproduced by returning extracted lipides to defatted flour (Cookson *et al.*—*J. Sci. Food Agr.* 8, 105). In continuing this study it is shown that much of the lipides in the undefatted flour exist as lipide protein complexes (Fisher *et al.*—*Chemistry & Industry* 1957, 765). The lipoxidase oxidation of polyunsaturated fatty acids in flour doughs counteracts the effect of sulfur dioxide and sodium bisulfite upon the mixing properties, alters the extensogram pattern of doughs in relation to mixing, and increases the oxidation of sulfhydryl during mixing (Smith *et al.*—*Cereal Chem.* 34, 337).

A patented baking pan grease, which is applied to the dough, contains fat, starchy material, and an edible acid (Haas—*U. S.* 2,793,123). A lecithin product for the same purpose is applied to the baking pans (Lalone—*U. S.* 2,796,363).

In the continuous production of mayonnaise, the ingredients are proportioned into the system, slurried under pressure, and filled into containers (Crawford & Hirsch—*U. S.* 2,781,270).

Rational use of oils and fats in canning of fish (Vershinin—*Rybnoe Khoz.* 33, No. 4, 84) and in sausage making (Gruttner & Pfaff—*Fleischwirtschaft* 9, 107), respectively are the texts of two communications. The one on fish canning contains practical data on changes in characteristics of oils during the

processing and storage of canned fish, and suggestions for practical utilization of various oils and fats in different products. Similar studies on sausage making indicate that the pH of the fat emulsion should be no higher than 6.5 and must be incorporated into the liquid phase. The fat content of German liver sausage has increased in recent years to a current average of 40%, but the spread in fat content is very wide (Kratz—*Ibid.* 9, 269). In this survey the fat content of the product is discussed with respect to spreadability, need for declaratory label, and consumers' preference. Cycloimidine derivatives of fatty acids are used to prevent adhesion of meat to can in canned products (Edgar & Spannuth—*U. S.* 2,800,410). A white sauce base for creamed meats and vegetables contains oleo oil, wheat flour, skimmed milk, and sodium glutamate (Lesparre—*U. S.* 2,811,452).

Fat "bloom" on chocolates is considered with respect to an unstable form of cocoa butter changing to a stable form during storage (Becker—*Fette-Seifen-Anstrichmittel* 59, 636). Cooling from high temperatures and adding other fats such as butter inhibits blooming; the mixing regime also has an influence. Analysis of the bloom fat as compared to the original chocolate coating fat shows an increase in saturated glycerides and decrease in olein (Cerbulis *et al.*—*J. Am. Oil Chemists' Soc.* 34, 533). In this work the type of center, i.e., high oil, high moisture or low moisture has no influence on the composition fat, but high oil (nuts) centers delay blooming. A fat which is used as a substitute or partial replacement of expensive cocoa butter, is made by treating vegetable oil with sulfur powder and hydrogenating (Commercial Henkel, S. A.—*Span.* 226,999). An artificial chocolate coating contains high-lauric acid type fat, cocoa powder, antioxidant, milk solids, sugar and alkali-N-lauroyl-sarcosinate as the emulsifier (Colten & DiPietro—*U. S.* 2,796,348-9). Partial polyglycerol esters of ricinoleic acid estolides are used as the emulsifier in chocolate coatings for ice cream briquets (van der Graht—*U. S.* 2,785,978). Acetylated fats containing two acetyl and one normal fatty acid radical are patented for making food coating emulsions (Brokaw—*U. S.* 2,808,421).

Fats are incorporated into livestock feeds by impregnation in toasted bread crumbs and mixing with the other ingredients (Feld—*U. S.* 2,784,093), and by emulsifying into molasses and then dispersing into the feed (Rawlings—*U. S.* 2,793,952).

The literature on chemistry of lanolin with respect to cosmetic uses is reviewed (Dietrich—*Inds. parfum.* 12, 177). Information on surfactant influence on the release of radio-labeled ions from an emulsified ointment base appears in a dissertation (Stark—*Univ. Microfilms Publ. No.* 21315, 109 pp.). The ability of 47 nonionics to replace sodium lauryl sulfate in hydrophilic ointment of U.S.P. XIV formula is recorded giving their capacity for moisture retention as affected by concentration (Lesshaft & DeKay—*Drug Standards* 25, 45). Neat's-foot oil is fractionated to produce a fraction of marked suitability for hardening pharmaceutical preparations (Bogs & Knepper—*Pharmazie* 12, 186).

A cream-like oral fat emulsion for dietetic purposes contains coconut oil, sugar, monoglyceride, and polyoxyethylene sorbitan monostearate (Kalish—*U. S.* 2,803,336).

Several vitamin oil products are described. One contains fish liver, butter and rice bran oils (Campbell—*U. S.* 2,797,185). In a powdered product the vitamin oils and antioxidant are adsorbed on flour (Hochberg & MacMillan—*U. S.* 2,777,797-8). Another powdered product contains the palmitate of vitamin A and vitamin D, antioxidant, and polyoxyethylene sorbitan monooleate (Maietta—*U. S.* 2,796,380). The latter induces a spheroid shape to the particles. A vitamin oil product is put up in medical capsules (Green & Schleicher—*U. S.* 2,800,457-8). An aqueous solution of fat soluble vitamin contains polyoxyethylene sorbitan fatty ester as the emulsifier (Sobel—*U. S.* 2,816,355). Thiamine is converted to thiamine monolauryl sulfate to modify its solubility properties (Utsumi & Harada—*Japan* 2496-'56).

Fatty materials are also involved in antibiotic preparations. In studies on variables and culture media components in the biosynthesis of penicillins, fats and oils serve exceptionally well as food for the molds (Noguchi *et al.*—*Nippon Nogei-kagaku Kaishi* 30, 45, 163; Di Accadia—*Congr. intern. biochem.* 3^e, 1955, 150). Highly unsaturated fats stimulate production of unsaturated penicillin F, and a more saturated fat favors saturated straight-chain penicillin, dihydro F and K (Macmorine—*Applied Microbiol.* 5, 386). A preparation for treatment of mastitis of udder is antibiotics in a gel carrier of aluminum stearate and peanut oil (Woodard—*U. S.* 2,792,329). Another base for antibiotics is obtained by heating 5% ethylcellulose in oil until the mixture clears (Borst—*U. S.* 2,796,381).

The hydrazides of the fatty acids of oils of the *Hydnocarpus* species (Tyoki—*Japan* 3369-'55) and glycerides of mixed C₈-C₁₄ fatty acids (Imhausen *et al.*—*Ger.* 842,940, Cl. 120) are said to have tuberculostatic properties. A significant reduction in severity of carious lesions in rats results from adding a small amount of sodium laurylsarcosinate to the ration (Zipkin & McClure—*J. Am. Dental Assoc.* 55, 15).

EMULSIFIERS. Reviews, usage, and descriptive information are published on the following emulsifiers: phospholipides (Hutt—*Chemistry & Industries* 1956, 1409, 1412), monostearin (Cressey—*Food Manuf.* 32, 165), diethylene glycol fatty acid esters (Schneider—*Fette-Seifen-Anstrichmittel* 59, 875), lauric acid-diethanolamine condensation products (Kroll & Lennon—*Drug & Cosmetic Ind.* 79, 186, 272), and surface-active agents used as food additives (Vivas—*Farm. nueva, Madrid*, 21, 435).

An equation relates the log of emulsion stability to the square root of the ratio of concentration to the critical micelle concentrations for a homologous series of emulsifiers (Osipow *et al.*—*J. Am. Oil Chemists' Soc.* 34, 34). This is in accordance with the experimental observations on emulsions stabilized with sodium lauryl sulfate and sodium cetyl sulfate. In studies on size frequency analyses and the distribution of globules in emulsions, the stability is related to the reciprocal of the decrease of specific area of interface per gram of oil (Mukerjee & Barthwal—*Z. physik. chem., Leipzig*, 207, 17). In this work certain combinations of emulsifiers have a greater "stability factor" than the individuals alone. The rate of flow of 1% soap solution in a capillary is reduced 10 times by 0.7 moles per liter of cetyl alcohol and such a combination emulsifies much more benzene than the plain soap solution (Kremnev—*Kolloid Zhur.* 19, 68). Such emulsions are very stable. They are reviewed with regard to smetic phase, surface tension, viscosity, refractive index, and factors involved in their stability (Lawrence—1st Congr. mondial detergence, Paris, I, 31). Stability of lecithin sols in the presence of electrolytes depends on mildness of treatments used to prepare the lecithin (Elsworth & Saunders—*J. Chem. Soc.* 1957, 330). Addition of a small amount of soap to such sols increases their stability. The good solubility of polyoxyethylene emulsifiers as compared with its lower and homologous compounds is explained on the basis that it is symmetrical and linear rather than zigzag (Rosch—*Fette-Seifen-Anstrichmittel* 59, 745).

Data are recorded on the influence of amount of emulsifier upon the composition of emulsion copolymers of acrylonitrile and water-insoluble monomers (Uchida & Nagao—*Bull. Chem. Soc. Japan* 30, 314). It is observed that the emulsion copolymer contains less acrylonitrile as the amount of emulsifier used increases. Data on Henry's law constants of butadiene in aqueous solutions of cationic emulsifiers show that that solubilization results from the presence of association micelles in solution (Ross & Hudson—*J. Colloid Sci.* 12, 523).

Nonionic emulsifiers, such as the "Tweens" inactivate preservatives, such as *p*-hydroxybenzoate, possibly through combining to form an oil soluble complex, (DeNavarre & Bailey—*J. Soc. Cosmetic Chemists* 7, 427). In another study of the same phenomenon it is suggested that the preservative is ineffective when inside the micelle, and to be effective one must select preservatives that dissolve in the aqueous phase outside the micelles (Aoki *et al.*—*J. Pharm. Soc. Japan* 76, 939).

The toxic effects of polyoxyethylene type emulsifiers may result, at least in part, from the non-absorption of the oxyethylene moiety from the gut inducing stress through long-continued diarrhea (Bush *et al.*—*J. Nutr.* 62, 601). Hamsters are more susceptible than mice to the effects of diarrhea or other stresses associated with these agents. Analytical determinations of polyoxyethylene ester by extraction followed either by hydroiodic acid cleavage or by precipitation with phosphomolybdic acid and barium chloride give equally satisfactory results, but the latter technique is more easily applied (Garrison *et al.*—*J. Assoc. Off. Agr. Chemists* 40, 1085). These methods are applied for the analysis of bread and rolls.

Newly patented emulsifiers for general usage are: *N*-aldonyl fatty acid esters of amino acids (Jonas—*U. S.* 2,785,152), reaction products of chloroformates of polyhydric alcohols and fatty alcohols (DEHYDAG G. m. b. H.—*Ger.* 848,187, Cl. 120), reaction products of fatty acids, ethylenediamine tetraacetic acid and a stoichiometric excess of dialkanol amine (Ruedrick—*U. S.* 2,794,000), condensed products of aliphatic quaternary ammonium or sulfonium salts and disulfonamides (DEHYDAG G. m. b. H.—*Brit.* 749,138), and reaction products of sulfonyl chloride, diethylene glycol, and a fatty acid (Suzuki & Hashimoto—*Japan* 6275-'55). A zein emulsion is made with various fatty emulsifiers (McDowell—*U. S.* 2,810,656). Castor oil fatty acid methyl esters are treated with phosphorus pentoxide and blown with ethylene oxide to yield emulsifiers

for parathion sprays (Koyama—*Japan 9093-55*). Use of polyoxyorbital tall oil esters as emulsifiers for insecticides is also patented (Behrens—*U. S. 2,786,013*). Polyalkylene glycol fatty acid esters are used to emulsify the insecticides or fungicides used to impregnate wood (Moren & Centerwall—*Swed. 157,802*). Another insecticidal and disinfectant emulsion contains alkyl sulfate as the emulsifier (Castel—*Fr. 995,735*). Lecithin emulsifiers are liquefied and standardized with respect to viscosity by additions of vegetable oils and mono-glycerides (Werly—*U. S. 2,777,817*).

ESTERS, ACIDS, ALCOHOLS AND OTHER FAT DERIVATIVES. Specific glycerides are synthesized by reaction of a fatty acid chloride and glycerol in chloroform and *N,N*-dimethylformamide to prepare an α -monoglyceride; making a diglyceride with a second fatty acid chloride from equimolar reaction mixture; and a third fatty acid may then be added (Hartman—*J. Chem. Soc. 1957, 3572*). The procedure is of interest because naturally occurring glycerides can be duplicated synthetically. Aceto- and butyro-monoleins and -dioleins are prepared and characterized with regard to polymorphic forms, expansibility, density, melting dilation, etc. (Gros & Feuge—*J. Am. Oil Chemists' Soc. 34, 239*). Symmetrical and unsymmetrical stearoyl and palmitoyl diacetins are characterized with respect to their polymeric forms (Lutton—*J. Am. Chem. Soc. 79, 5137*). Mixtures of symmetrical and unsymmetrical isomers show greatly increased alpha stability over that of the pure compounds. The commercial acylated glyceride products are reviewed with respect to uses in cosmetics (Sisley—*Ind. par-fum. 12, 80*).

The following lipide materials are synthesized for use in biological and biochemical studies or to gain fundamental information on synthesis, structure, or properties of natural counterparts: cholesterol-21-C¹⁴ (Kurath & Capezzuto—*J. Am. Chem. Soc. 78, 3527*, cholesterol-24-C¹⁴ (Kurath et al.—*Helv. Chem. Acta 40, 933*), 22-oxocholesterol-23-C¹⁴ and cholesterol-24-C¹⁴ (Kautsky—*Univ. Microfilms, Ann Arbor, Mich., Publ. No. 20583, 95 pp.*), cholesterol methyl ether and 3 β -methoxy-bisnor-5-choleonic acid (Romeo & Villotti—*Ann. chim., Rome, 47, 618*), 6 β -chloro-3 β , 5-cholestanediol 3-acetate (Shioto—*Nippon Kagaku Zasshi 76, 402*), certain sterols like those found in wool fat (Woodward et al.—*J. Chem. Soc. 1957, 1131*), *L*- α -lecithin and other lecithin products (Tattrie & McArthur—*Can. J. Biochem. & Physiol. 35, 1165*), oleic-1-C¹⁴ acid (Nevenzel & Howton—*J. Org. Chem. 22, 319*), ricinoleic acid (Gensler & Abrahams—*Chemistry & Industry 1957, 47*), DL-ricinoleic acid (Bailey et al.—*J. Chem. Soc. 1957, 3027*), nonacosanedioic acid (Fuqua—*Univ. Microfilms, Ann Arbor, Mich., Publ. No. 21853, 195 pp.*), ximenynic acid, homoriein-stearolic acid and two fatty hydroxy acids with allenic side-branches (Crombie & Jacklin—*J. Chem. Soc. 1957, 1622*), α - and β -eleostearic and trichosanic acids (*Ibid.* 1632), di-, tri-, and tetra-homologous series of the methylolmethane fatty acid series (Breusch & Oguzer—*Chem. Ber. 88, 1511*), various ω -derivatives of chaulmoogric and hydno-capric acids (Kariyone & Ageta—*Kakugaku Zasshi 77, 764*), ω -trifluorostearic acid and α -trifluoro-*n*-octadecyl amine (Gavlin & Maguire—*J. Org. Chem. 21, 1342*), and various branched chain fatty acids (Crombie et al.—*J. Chem. Soc. 1957, 479*). Various randomly C¹⁴-labeled fatty acids are obtained through biological synthesis by *Chlorella pyrenoidosa* grown on culture medium containing labeled material (Schlenk & Mangold—*Fed. Proc. 16, 243*). Reviews are written on synthesis of cephalin, phosphatidyl serine, and acetal phospholipides (Malkin—*Fette-Seifen-Anstrichmittel 59, 77*), of linear, branched, ethylenic, acetylenic, hydroxy and cyclic fatty acids (Gensler—*Chem. Revs. 57, 191*) and of branched fatty acids (Koch—*Fette-Seifen-Anstrichmittel 59, 493*).

Many α , ω -diaryl fatty acids are synthesized and their properties are recorded (Oura et al.—*J. Pharm. Soc., Japan, 76, 1071*). Oleic acid esters of aryloxy alcohols are made for use as intermediates, as plasticizers and in lubricants (Neely & Rocchini—*U. S. 2,809,936*). Fatty acid-phosphonic acid compounds are rust inhibitors, lubricant oil modifiers, and insecticide ingredients (Reis—*U. S. 2,784,208*). Various means for protection of the double bond in cholesterol are studied to gain information basic to the design of methods for converting wool fat sterols to hormonal products (Ruiz & Panizo—*Anales real soc. espan. fis. y quim., Madrid, 52B, 571, 579*).

Halogen derivatives of fats are prepared as intermediates for other preparations. A continuous process for making fatty acid chlorides is based on reaction with thionyl chloride and vaporizing the excess chlorinating agent (Humphlett—*U. S. 2,808,420*). A method involving treating the fatty acids with phosphorus chlorides in petroleum solvent eliminates purification of the product by distillation (Youngs et al.—*J. Am. Oil*

Chemists' Soc. 34, 107). Fatty chlorides are also prepared from fatty alcohols by reaction with thionyl chloride (Rohm & Haas Co.—*Brit. 764,149*). Halohydrins of fatty acids are prepared by treating the acid with *tert*-butyl hypochlorite or hypobromite (Rohm & Haas—*Brit. 774,946*).

A thesis contains information on the chemistry and formation of aluminum soaps (Rakowitz—*Univ. Microfilms, Ann Arbor, Mich. Publ. No. 21814, 151*). Aluminum trilaurate is prepared as micelles of high molecular weight in solution, by reaction of trimethylaluminum or aluminum isopropoxide with lauric acid (Leger et al.—*Can. J. Chem. 35, 799*). The effects of dilution, hydrolysis, and hydrochlorination on this product are assessed by viscosity, light-scattering, and infrared measurements. Mono- and di-isopropoxy laurates and stearates are also prepared and described with regard to behavior in organic solution (Pande & Mehrotra—*J. Inorg. & Nuclear Chem. 4, 128*).

On heating lead stearate the normal form is stable up to 110°, an amorphous form then appears and is converted to a second crystalline form at 130° which changes to another amorphous form that persists up to 200°; and on cooling a new form appears along with the other forms, but on storage all revert to the original form (Trillat et al.—*Compt. rend. 243, 71*). Similar transformations occur with the laurate but the temperatures of transformations are lower. In a continuation of this work copper soaps are also studied and x-ray crystal data are recorded and discussed with respect to orientation of the molecules in the various forms (Trillat et al.—*Bull. soc. franc. mineral. et crist. 80, 67*). Data are recorded on the effect of mechanical work, tempering, and storage temperature and time on the rigidity and thixotropic behavior of peanut oil-aluminum stearate systems as basic information for making suspensions in pharmacy (Matsumura et al.—*Pharm. Bull., Tokyo, 5, 21*). Data on the viscosities of solutions of magnesium caprate and magnesium laurate in eight organic solvents at different concentrations and temperatures are interpreted to support the conclusion that molecular aggregation of soap molecules in solvents is not affected by the temperature (Bose & Dixit—*J. Am. Oil Chemists' Soc. 34, 386*).

Monolayers of fatty acids on aqueous metal salt solutions combine with the metal in a manner similar to reactions in ionized solutions (Sasaki & Muramatsu—*Bull. Chem. Soc. Japan 29, 35*). In such systems lithium, calcium and barium form condensed solid films; thorium, iron, aluminum, cobalt, copper and zinc form expanded solid films; and sodium, potassium, rubidium, and ammonium salts yield no solid film (Kimizuka—*Ibid.* 123).

Aluminum monostearate is prepared by dissolving stearic acid in a warm 2:1 ethanol:water mixture, alternately adding small amounts of potassium hydroxide and aluminum salt, acidifying to pH 3, filtering and washing the product (Uchiumi & Samejima—*Japan 3232-55*). In a process for making the distearate, aluminum salt solution is added to a warm sodium stearate solution with vigorous stirring (Shioneri et al.—*Japan 2983-55*). A patented basic zirconyl soap is useful in lubricants, water proofers, and cosmetics (Blumenthal—*U. S. 2,802,847*). Solutions of metallic soaps in liquid hydrocarbon solvents are rendered stable with polyoxyalkylene glycol (Fischer—*U. S. 2,807,533*).

The latest developments in manufacture of fatty alcohols by catalytic hydrogenation (Engel—*1^{er} Congr. mondial detergences, Paris, 1, 369*) and by sodium reduction (Hug—*Ibid.* 367) are reviewed. Data on yields and composition of products from applying both methods to various commercial oils is recorded as practical information (Kane & Kulkarni—*J. Proc. Oil Technol. Assoc., India, 10, 43*). In these studies, crude or refined neem oil could not be converted to alcohols by hydrogenation. In a study of 35 catalytic methods for hydrogenating methyl laurate to dodecyl alcohol, 4% copper chromite catalyst, 300° temperature, 2600–3000 lbs./sq. in. hydrogen pressures and high rates of agitation permit the best (90%) yields (Church & Abdel-Gelil—*Ind. Eng. Chem. 49, 813*). A method for preparation of pure linoleyl alcohol comprises sodium reduction of walnut or corn oil, urea fractionation of the alcohols, and isolation of the product by the bromination-debromination method (Masuyama—*Nippon Nogei-kagaku Kaishi 28, 432*).

New techniques for fatty alcohol manufacture are patented. Converting the fatty material to alcohol esters prior to hydrogenation to fatty alcohols permits recovery of glycerol, and improves catalyst life and ease of conversion (Wilson & Bayerian—*U. S. 2,809,206*; Toland & Levine—*U. S. 2,776,323*; Reed—*Brit. 749,069*). Novelty in production of alcohols from the sodium reduction of safflower (Wilson—*U. S. 2,803,671*) and oiticica (Wilson—*U. S. 2,800,516*) oils is based on suitability of use of the products in paints, lubricants and de-

tergents. Phosphate esters of several fatty alcohols and natural sterols are prepared and characterized in regard to some of their physical properties (Calderon—*Anales real soc. espan. fis. y quim., Spain* 53B, 69).

The Woodward *cis*-hydroxylation synthesis is successfully applied to natural fatty acids and fats to convert the unsaturated radicals to *cis*-glycerols (Gunstone & Morris—*J. Chem. Soc.* 1957, 487). The method is based on use of iodine and silver acetate to induce hydroxylation and it has certain advantages over other methods. The method of preparing 9-oxo-10-hydroxystearic acid by oxidation with potassium permanganate is investigated for the influence of variables and the data are used to improve the technique (Coleman *et al.*—*J. Am. Chem. Soc.* 78, 5342). In the preparation of trihydroxystearin, the oil of *Euphorbia lathyrus*, which contains about 90% olein, is subjected to hydroxylation technique (Pigulevskii & Saprokhina—*Zhur. Priklad. Khim.* 30, 1104). Isomeric succinic acid monoesters of oleyl and elaidyl alcohols oxidized with peracetic acid, hydrogen peroxide-osmium oxide, or alkaline permanganate and resolved with cinchonidine give optically active succinates, which upon hydrolysis furnish optical active octadecanetriols (Sasaki—*Nippon Kagaku Zasshi* 76, 454). Unsaturated fatty compounds can be formylated by treatment with formic acid and the products may be hydrolyzed to the corresponding hydroxyl compounds (Swern *et al.*—*Brit.* 756, 981). Autoxidation of erucic acid to 13, 14-dihydroxybehenic acid is explained by oxygen attached at the double bond and homolytic fission of the peroxide formed to two hydroxy radicals (Skellon & Taylor—*J. Appl. Chem.* 7, 67). Kamlolenic acid, derived from kamala oil is converted to a hydroxy and dicarboxylic acid by hydrogenation followed by oxidation with potassium dichromate (Gupta & Aggarwal—*J. Proc. Oil Technol. Assoc., India*, 11, 81).

Many compounds are produced by oxidation and pyrolysis techniques applied to fatty materials. When the 9(10)-hydroxy-10(9)-oxostearic acid, produced by permanganate oxidation of oleic, is further oxidized with oxygen 9,10 dioxostearic acid is the main product and on continuing the oxidation this is converted to highly oxygenated polymeric acids (Parkinson & Skellon—*J. Chem. Soc.* 1957, 851). Reaction of methyl oleate with selenium dioxide introduces a ketone group in the ester rather than hydroxyl groups as previously recorded (Watanabe *et al.*—*J. Sci. Hiroshima Univ., Ser. A*, 20, 203). Oxidizing ethyl 10-undecylenate in acetic anhydride with the same reagent produces 11-acetoxy-9-undecylenic acid (Watanabe—*Ibid.* 21, 151). Pyrolysis of ricinoleate or castor oil at 500–700° with water vapor leads to high yields of *n*-heptaldehyde and undecylenate (Wetroff *et al.*—*U. S.* 2,807,633). Mono- and dibasic acids are prepared by caustic fusion of cyano-hydroxy fatty acids and esters derived by reaction of epoxidized fat with alkali metal cyanide (Riener—*U. S.* 2,811,538). Equipment is designed for production of azelaic acid by treating fatty compounds with ozone (Emery Inds. Inc.—*U. S.* 2,813,113; *Brit.* 757,355). In the alkali fission of ricinoleic acid to produce sebacic acid and other products, the formation of capryl alcohol is inhibited and heptanoic acid is favored by fusing at a temperature above the melting point of caustic (Logan—*U. S.* 2,777,865). Sebacic acid is separated from mixtures of other mono- and dibasic acids by adding benzene to aqueous solution of the sodium salts, neutralizing to the extent of one mole hydrogen ion per mole dibasic acid to precipitate the sebacic acid, which is further purified by crystallization from salt solution (Greenberg—*U. S.* 2,749,364). Water insoluble dicarboxylic acids are separated from other water insoluble components by extracting with hot aqueous solutions of partial salts of polycarboxylic acids (Stein—*U. S.* 2,793,093). Iron is removed from ketones, prepared by treatment of fatty acid with iron, by treatment with oxalic acid and filtering (Christensen—*U. S.* 2,813,125).

Vapor-phase decarboxylation of commercial oleic acid at 400° by magnesium oxide results in 65% yield of liquid product having a 95% reduction of acid value (Chakrabarty & Palit—*Sci. & Culture, India*, 22, 464). In such a process the catalyst is conserved by maintaining the temperature over 340° so that magnesium soaps are not formed (Chesrown—*U. S.* 2,811,559). The literature on reaction mechanism of decarboxylation is reviewed (Franzen—*Chem.-Ztg.* 81, 424).

Some communications contain information on preparation properties, or uses for various derivatives of fats containing nitrogen. Schwitzer (*Ind. Chemist* 32, 426) discusses cationic nitrogen derivatives of fatty acids with respect to uses in flotation, road construction, as antistatic agents, corrosion inhibitors and bactericides. Fatty amines quaternized with a glycerol chlorohydrin ether of sorbitol are useful waxy water-soluble compounds (Zech—*U. S.* 2,775,604). Such compounds

dissolve in mineral oil if they are first dissolved in fatty alcohol or vegetable oil (Consortium produits chimiques—*Fr.* 1, 045,186-7). Lipoprotein preparations made by the Schotten-Baumann reaction are refined by washing with organic solvent of density greater than the lipoprotein (Cavanaugh—*U. S.* 2,794,796). A review on fatty acid amides contains information on preparation, properties, uses and analysis (Kaufmann & Skiba—*Fette-Seifen-Anstrichmittel* 59, 340, 498). *Sec*-hexyl amine, formaldehyde and fatty acid amides are condensed in the preparation of compounds useful in the lubricant, detergent, wax, and resin industries (Zech—*U. S.* 2,813,091). X-ray data of binary mixtures of fatty acid amides (Kuroda—*Nippon Kagaku Zasshi* 78, 239) and molecular compounds of acetamide with various fatty acids (Mod *et al.*—*J. Am. Chem. Soc.* 79, 5129) are interpreted, respectively, with regard to structure and polymorphic modifications. The solidification points of binary mixtures of stearic acid, stearamide, octadecanol, and stearonitrile are recorded and discussed with regard to occurrence of eutectic mixtures (Sassin *et al.*—*J. Am. Oil Chemists' Soc.* 34, 76). Similar data from binary mixtures of 2-aminopyridine indicate that molecular compounds are formed, and that the degree of dissociation of the compounds in the molten state varies with the chain length and the degree of unsaturation (Mod *et al.*—*J. Phys. Chem.* 60, 963, 1651).

Two patents are on manufacture of fatty acid nitriles. In one the fatty acids are contacted with ammonia in the liquid phase in the presence of dehydration catalyst and in a second step the reaction is completed in the vapor phase (Jansen & Roha—*U. S.* 2,794,043). In a three-stage process, fatty acids are contracted with ammonia to give amides; the second stage converts 70% of the amides to nitriles, and vents moisture and ammonia; and the reaction is completed in a final stage (Potts & Smith—*U. S.* 2,808,426). The patents on hydrogenation of fatty nitriles to make fatty amines pertain to use of nickel borate (Terry & Jakobsen—*U. S.* 2,784,232), nickel on diatomaceous earth (Komori *et al.*—*Japan* 6464,55), and copper oxide-chromium trioxide (Shapiro—*U. S.* 2,811,556) as catalyst in the processes, and to adding ammonia and hydrogen alternately, during the hydrogenation (Shapiro—*U. S.* 2,781,399). Boiling point and vapor pressure information are recorded as basic data for separation of C₁₀–C₁₄ amines from C₁₂–C₁₄ amines by distillation (Fürst & Schwetlick—*Chem. Tech., Berlin*, 8, 417). A mixture of fatty amines, formaldehyde and formic acid reacts in the presence of sulfuric acid to yield tertiary amine products (Erickson—*U. S.* 2,776,314). The reaction of fatty amines with lower alkyl esters of α , β -unsaturated acids to yield secondary and tertiary products is accelerated by addition of an alcohol or dioxane (Harrison & Aelony—*U. S.* 2,787,633). A colorimetric method for determination of primary fatty amines is based on reaction with salicylaldehyde in the presence of acetic acid (Milun—*Anal. Chem.* 29, 1502). Measurements of the dipole moments of fatty acid salts of dodecylamine in benzene solution indicate that association occurs not only from dipolar interaction, but also from chain interaction (Shirai—*Sci. Papers Coll. Gen. Ed. Univ. Tokyo* 6, 147).

FATTY MATERIALS USED IN TEXTILE TREATMENT, WATER-PROOFERS, ANTI-CORROSIIVE COMPOUNDS, DEFOAMERS, WELL DRILLING FLUIDS, INCENDIARY PREPARATIONS, AGRICULTURE, AND MISCELLANEOUS PRODUCTS. Reaction products of fatty amines with certain sugars (Erickson—*U. S.* 2,815,339–40, 2,808,401) and soaps of certain hydroxy fatty acids and polyvalent metals (Davis & Winston—*U. S.* 2,785,989; Davis *et al.*—*U. S.* 2,793,132) are made for water proofing textiles. Such products, fatty amine resins, silicones and other textile waterproofing materials are reviewed in regard to ease of application, water absorption and swelling (Ruile—*Melliand Textilber.* 38, 313). Condensation products of a polyalkylenepolyamine and a fatty acid are deposited on textiles to soften them (Hiestand & Albrecht—*U. S.* 2,785,092). The newly patented textile antistatic agents are a mixture of polyethylene oxide wax and ricinoleic acid sulfonate (Lange & Henkel—*U. S.* 2,790,764) and a water dispersible ethoxylated hydroxy tertiary amine containing a fatty acid group attached to amine-nitrogen (Welles & Edelstein—*U. S.* 2,809,159).

A paper size contains saponified tall oil fractions, alum, and clay filler (Poradowska—*Prace Inst. Celuloz.-papier* 6, No. 1, 31). A condensate of fatty acid amine with methylenebisacrylamide is also used for sizing paper (Lundberg & Reynolds—*U. S.* 2,801,169).

Wax-like polymers are prepared by condensing glycerol distearate with fatty derivatives of hydroxymethylmelamine, and/or *N*-hydroxymethyloctadecyl-carbamate (Hiestand & Albrecht—*Swiss* 317,474). Soaps and/or various fat-derived surface active agents are added to waxes for coating paper as

defoamers; to inhibit adhesion, scuff and "blocking"; and to improve slip characteristics (Higgins—*U. S. 2,796,355*; Moyer—*U. S. 2,803,612*; Ritson—*U. S. 2,764,509*; Simerl *et al.*—*U. S. 2,773,045*; Pagett—*U. S. 2,783,161*). Soap and/or fat derived surface active agents are ingredients of new polishing waxes (Micheletti—*Ital. 499,506*; von Rosenberg & Sauer—*U. S. 2,782,124*; Lerner—*U. S. 2,780,544*; Kondrath *et al.*—*Soap Chem. Specialties 33*, No. 9, 77). One communication contains information on formulating buffing compounds to permit easy degreasing of buffed metals with solvent or alkaline cleaners (van Zandt—*Products Finishing 21*, No. 3, 41).

Coating compounds for inhibiting rusting of metals comprise petroleum oil or solvent, waxy materials and fatty additives such as: fatty acid-amino acid ester (Spivack *et al.*—*U. S. 2,790,779*), sorbitan monooleate plus phenyl ethanol amine (Oathout & Scott—*U. S. 2,783, 156*), calcium and lithium soaps plus amine compounds (Westlund & Furey—*U. S. 2,816,842*), degrass plus sorbitan monooleate (Sproule & Tiedje—*U. S. 2,791,510*), sulfonated detergent (Dieman & Ravenscroft—*U. S. 2,771,371*), and tall oil (Lanteri—*U. S. 2,785,089*). The compounds for inhibiting corrosion of oil well equipment by petroleum-brine mixtures are: reaction products of polycyclic amines with blown fatty oil (DeGroot & Cheng—*U. S. 2,781,352*), products of equal moles of diethylenetriamine and fatty acids (Hughes—*U. S. 2,793,997*), metal soaps plus reaction products of alkylolamines and fatty acids (Fischer & Scheffel—*U. S. 2,782,164*), and nonionic detergent plus quaternized nitrogen-heterocyclic base (Beiswanger & Mayhew—*U. S. 2,814,593*). Gasoline, kerosine, fuel oil, Diesel oil, etc. are rendered non-corrosive by addition of soluble fatty oil distillation pitch (Brown *et al.*—*U. S. 2,807,527*). A rust-preventive oil tester is designed to duplicate use factors such as various humidities, moisture condensation, specific temperatures, etc. (Roden—*ASTM Bull. 1957*, No. 223, TP121).

The newly patented oil well drilling muds or fluids contain such materials as water, clays, petroleum oil, fatty oils, asphalt, cellulose materials, and other substances plus a dispersing agent which is usually some type of fat derivative (Engle—*U. S. 764, 546*; Swain & Schuessler—*U. S. 2,793,188*; Schuessler—*U. S. 2,793,189*; Lummus—*U. S. 2,793,996*; Dunn & Lummus—*U. S. 2,797,196*; Nelson & Messenger—*U. S. 2,798,851*; Lacey *et al.*—*U. S. 2,799,646*; Nowak & Keller—*U. S. 2,802,784-5*; Stratton—*U. S. 2,816,073*). A slurry of sand and water-hydrocarbon emulsion, which is used for fracturing earth formations around wells, can be emulsified with fatty acids, fatty alcohols, or any of numerous fat derived detergents (Cardwell *et al.*—*U. S. 2,802,531*). A solution of sodium alkaryl sulfonate is used to water-flood oil wells so that more petroleum oil is released (Kirk—*U. S. 2,808,109*). Laboratory tests on water-flooding agents indicate that non-ionic wetting agents should give better results than ionic agents (Labbe—*Rev. inst. franc. petrole et Ann. combustibles liquides 12*, 185). Some improved procedures for use of non-ionic detergent and citric acid solutions in secondary petroleum recovery are described and operating data are presented (Johansen *et al.*—*U. S. Bur. Mines, Inform. Circ. No. 7797*, 18 pp.).

Reviews and general information communications on ore flotation agents are on the influence of alkalinity on flotation with fatty acids (Desnoes—*1^{er} Congr. mondial detergence, Paris 1954*, 3, 931), influence of electrolytes (Wrobel—*Ibid.* 905), and behavior of various detergents in the process (Pormanek—*Ibid.* 895). A pine oil oleate soap emulsion is particularly suited in ore flotation at low temperatures (Klassen—*Izvest. Akad. Nauk S. S. R. Otdel. Tekh. Nauk 1957*, No. 5, 136). Data on the flotation characteristics of a Florida leached zone phosphate ore with fatty acids as influenced by fineness, impeller speed, pH, various fatty acids, and various metal ions are recorded as basic information for design of processing (Sun *et al.*—*Trans. Am. Inst. Mining Met. Petrol. Eng. 208, Tech. Publ. 4, 411B.*). In this work the collecting power with respect to the fatty acids increases with increased unsaturation up to two double bonds. In the flotation of quartz with use of barium oleate, the usefulness of tensiometric measurements as a precise means of controlling conditions for flotation is confirmed (Guastalla—*Compt. rend. 243*, 1314). A study on using sodium alkaryl sulfonates of mole weight 300-50 for flotation of ores indicates good action with galena, chalcopyrite and sphalerite; much less with pyrite, and none with quartz (Plaksin *et al.*—*Trudy Inst. Gornogo Delo, Akad. Nauk. S. S. R. 3*, 239). Standard procedures are worked out for using fatty acids from fish oils for floating Michigan specularite or Jasper Ore (Cooke & Stansby—*Comm. Fisheries Rev. 19*, No. 4a, 24). A patented flotation agent comprises fatty amine, dispersant, and alkoxyalkanol solvent (Lentz *et al.*—*U. S. 2,816,870*).

New patents and descriptions on suppressing foams deal with the use of: a mixture of turkey-red oil, cerfak and cetyl pyridine bromide (Doss & Agarwal—*J. Proc. Oil Technol. Assoc., India, 11*, 41), a mixture of solid fatty alcohols, solid fatty acids and an amine soap (Chappell—*U. S. 2,797,198*), salt of monofatty acid phosphonates (Bradley & Giammaria—*U. S. 2,792,374*), and sugar cane oil with or without a carrier solvent (Hlavacek—*U. S. 2,809,168*).

The new gelling agents for incendiary fuel are the aluminum soaps of mixtures of fatty and naphthenic acids (Shulman—*U. S. 2,763,621*) and mixtures of oleic and dibasic acids (Aylesworth & Stegemeyer—*U. S. 2,795,492*).

Fatty *N*-sorbitolcarbamates are patented for use as fungicides (Boettner—*U. S. 2,808,402*). Another fungicide composition is made by mixing a benzene solution of copper and nickel oleates with 8-hydroxyquinoline dissolved in oleic acid (Heymons & Schnabel—*U. S. 2,799,615*). Agricultural spray powders adhere better when oiled with vegetable oils than when oiled with petroleum oils, but treatment with mixtures of vegetable and mineral oils is most suitable in a compromise of all factors such as adherence, storability, and permanence after dusting (Takita & Fukunaga—*Bull. Natl. Inst. Agr. Sci. Ser. C. No. 4*, 23 pp.).

Still-pitch remaining after the commercial distillation of soybean and cottonseed fatty acids is effective for improving the tilth of certain agricultural soils (Paschke *et al.*—*J. Am. Oil Chemists' Soc. 34*, 149).

A comparison of mineral oil, sulfated mineral oil, cod oil, and oleic acid for increasing the tensile strength and elongation of vegetable-tanned split hides shows that all four oils are equal and interchangeable (Pomeroy—*J. Soc. Leather Trades Chemists 41*, 106). General descriptive communications are written on using cod oil, seal oil and fatty acids in oil-tanning of leather (Airoldi—*Chim. Ind., Milan, 39*, 283) and on preparing soluble vegetable and animal oils for use in fat-liquoring leather (Neel—*1^e Congr. mondial detergence, Paris, 1954*, 2, 579).

A communication on brake fluid manufacture contains information on using castor oil, castor oil derivatives, and other materials for this purpose (Carr—*Soap Chem. Specialties 33*, No. 11, 61).

Addition of oxyalkylated fatty amine salts, derived from soybean oil, to chromium plating baths provides greater "throwing power" and retards chromic acid fumes (Chester—*Brit. 772,104*).

A gel product made by reacting glycerol polyether esters of tall oil with polyphenols is useful as a linoleum cement (Bradley—*U. S. 2,786,773*). Oxidized esters of tall oil and a polyhydric alcohol are made for the same purpose (Forsythe—*U. S. 2,815,295*). A binder oil for sand molds and cores is a drying or semidrying oil containing a small amount of sodium chlorite (Moser—*U. S. 2,816,041*).

Solutions of long chain amines are shown to be excellent extractants for many mineral, organic and complex metal acids (Moore—*Anal. Chem. 29*, 1660). In this work, extractions of some anionic complexes of polonium, plutonium, uranium, zirconium, hafnium, and protactinium are demonstrated.

Addition of 0.01-0.05% of cottonseed oil soap to cement mortars and concrete has a plasticizing effect, permits reduction of water-cement ratio and the amount of cement; and at the same time it increases the strength (Salidzhanov—*Trudy Inst. Sooruzhenii Akad. Nauk Uzbek S. S. R. 1954*, No. 5, 53).

DRYING OIL PRODUCTS—PAINTS, RESINS AND PLASTICIZERS. Communications containing reviews and descriptive information on this subject are written on: theory of bodying of linseed oil (Sigwalt—*Peintures, pigments, vernis 33*, 544), use of linseed oil in bodied oils, alkyd resins and white paint (Fauve—*Ibid.* 32, 672), oxidation and film formation of drying oils (Kaufmann—*Fette-Seifen-Anstrichmittel 59*, 153), new tung oil derivatives (Hoffman & Bickford—*U. S. Dept. Agr. ARS-72-7*, 16 pp.), metallic soaps used in the protective coating industry (Licata—*Off. Dig. Fed. Paint & Varnish Prod. Clubs 29*, 485), use of synthetic fatty acids in drier manufacture (Tyutyunnikov & Bukhshtab—*Masloboino-Zhirovaya Prom. 23*, No. 1, 21), standards for driers used in oil plants (*Brit. Standard 332*, 12 pp.), surfactants in the paint and allied industries (Moore & Bell—*Paint Technol. 21*, No. 237, 199), nonyellowing oils, especially tobacco seed oil (Greaves—*J. Oil & Colour Chemists Assoc. 39*, 934), review of conjugation changes in oils and their correlation with paint properties (von Mikusch—*Off. Dig. Fed. Paint & Varnish Prod. Clubs 28*, 44), polymerization of rosin and uses for the products (Parker—*Paint Manuf. 26*, 454), use of pyrolyzed rosin for varnish production (Zacharewicz *et al.*—*Przemysl Chem. 9*, 610), improving the drying properties of oil by furfural treatment (Rigamonti

& Spacecamela-Marchetti—*Chim. ind., Milan, 39, 261*), improved surface coatings using epoxidized oils (Chatfield—*Paint Manuf.* 27, 51), organic reactions with hydrogen peroxide (Manly—*Chem. Products 20, 399*), epoxy resins and amine catalyst (Bourne—*Chemistry & Industry 1957, 578*), chemistry of epoxide resins in relation to their application (Bruin—*Ibid.* 616), chemistry and structure of epoxide resins in relation to properties and applications (abstract from a symposium—*Ibid.* 1255), epoxidized tung oil (Greenfield—*Paint Ind. Mag.* 71, 16), epoxy resins in surface coatings (Anthony & Howe—*Product Finishing, London, 9, No. 9, 78*), structure of epoxy resins and their reactions with fatty acids, anhydrides of dibasic acids, and phenol-formaldehyde resins (Bruin—*Peintures, pigments, vernis 33, 622*), stearylamine-epoxide resin characteristics of the polyamide-epoxy system (Floyd *et al.*—*J. Applied Chem.* 7, 250), formulating with isophthalic alkyds (Wampner—*Off. Dig. Fed. Paint & Varnish Prod. Clubs 28, 663*; Carlston & Lum—*Ind. Eng. Chem.* 49, 1051), thixotropic alkyds (North—*J. Oil Colour Chemists' Assoc.* 39, 695), review of analytical methods for alkyd resins (Gulinsky & Schmuling—*Fette-Seifen-Anstrichmittel 59, 860*), rosin in surface coatings (Kamath & Shetye—*Paintindia 6, No. 8, 23*), polyamide resins in surface coatings (Keenan—*J. Oil Colour Chemists' Assoc.* 39, 299), coatings based on blends of polyamide and epoxy resins (Glaser *et al.*—*Off. Dig. Fed. Paint & Varnish Prod. Clubs 29, 159*), theories of structure of alkyd resins (Tawn—*J. Oil Colour Chemists' Assoc.* 39, 223), modified polyamide resins of U. S. patent 2,663,649 (Erlandsen—*Fette-Seifen-Anstrichmittel 58, 992*), and styrene in the surface-coating industry (Allsebrook—*Brit. Plastic 28, 383*).

Some drying oil studies pertain to the mechanism of the polymerization reactions. Molecular distillation and hydrogenation data on polymerized oils are interpreted to indicate that: more than one double bond per molecule can be consumed; with nonconjugated oils, the kinetic order of polymerization increases from unity to approach a value of two; second order characteristics are maintained with conjugated oils; and calculated rate constants and over-all energies of activation agree with those obtained from viscosity measurements (Sims—*J. Am. Oil Chemists' Soc.* 34, 466). Polymers formed during the oxidation of linoleate are isolated and analyzed for peroxide, hydroxyl groups, epoxy groups, and ketone-carbonyl groups (Witting *et al.*—*J. Am. Oil Chemists' Soc.* 34, 470). On the basis of oxygen known to be present but not detectable in the functional groups, the possibility that polymerization takes place through an oxygen linkage is considered. A monomeric cyclic compound isolated as a non-urea-adduct-forming distillate from bodied safflower oil is identified as a cyclized product of linoleic acid with a mean unsaturation of one double bond (Mehta & Sharma—*Ibid.* 448). Thermal oxidation of corn oil proceeds through an induction phase and a second phase characterized by rapid increase in viscosity (Johnson & Kummerow—*Ibid.* 407). Increasing the rate by aeration, speeds the magnitude in changes but does not alter the over-all two-phase reaction.

Oils destined for use in protective coatings are improved by isomerization. New information on how to do this and on the characteristics of the product pertain to: isomerization of linseed oil with iodine (Prakash *et al.*—*J. Proc. Oil Technol. Assoc., India, 11, 19*), tung oil isomerization with inorganic iodides (Paek & Planck—*U. S. 2,760,968*), isomerization of tung oil by hydrogenation with nickel catalyst poisoned with sulfur or selenium (Mills—*J. Oil & Colour Chemists' Assoc.* 40, 10), and isomerization of dehydrated castor oil with sulfur dioxide, nickel and aluminum iodide catalysts (Chowdhury & Mukherji—*J. Sci. Ind. Res., India, 16B, 175*). In the later work best results are obtained with aluminum iodide catalyst containing a small amount of zinc chloride. In tests with inorganic sulfate catalyst, the nickel, manganese, and chromium sulfates are most active and the copper, aluminum and cobalt salts are less so (Tanabe—*Japan 8056-55*). A triene compound isolated from conjugated methyl linoleate hydroperoxide appears spectroscopically identical with β -eleostearic (Banks *et al.*—*Nature 179, 1078*). The isomerization of methyl linoleate with palladium on carbon catalyst, at 200° is about 24% after four hours (Takemura & Goldblatt—*J. Am. Oil Chemists' Soc.* 34, 15).

Continuous polymerization of an oil is carried out in a froth layer by blowing air through it while continuously supplying fresh oil and removing froth containing oxidized oil (Konshin & Orobchenko—*U. S. S. R. 105,844*). Castor, cuttle fish, and soybean oils polymerized under the influence of silent electrical discharge are suited for manufacture of coating materials (Gotoda—*J. Electrochem. Soc., Japan 24, 177, 313*). Kamala seed oil gels quicker under heat treatment than tung

oil (Ojha & Aggarwal—*J. Sci. Ind. Res., India, 15B, 656*). Monoethylene fatty acids heated with montmorillonite clays form dimers (Barret *et al.*—*U. S. 2,793,219*), whereas polyunsaturated acids form higher polymers under these conditions (Barret *et al.*—*U. S. 2,793,220*). Bodied drying oils are made to dry to a wrinkle finish by addition of a bentonite amine reaction product (McBride—*U. S. 2,763,568*), or ethyl silicate (Collins—*U. S. 2,798,057*).

Hydroxy oils, especially castor oil, are converted to drying oils by dehydration. Such processes for castor oil using potassium acid sulfate (Drimus—*Acad. rep. popolare Romine, Studi cercetari chim. 3, 252*), 1,3-benzenedisulfonic acid (Dole—*J. Proc. Oil Technol. Assoc., India, 9, 75*), and sodium acid sulfate or zinc dust (Sivasamban *et al.*—*Indian 55,423*) as catalysts; and a vapor phase process with alumina as the catalyst (Maruta & Iwama—*Nippon Kagaku Zasshi 76, 480*) are described with some operational data. The film properties of oil dehydrated with phosphoric acid are superior to those of oil dehydroxylated with sodium acid sulfate catalyst (Dole & Deshpande—*J. Proc. Oil Technol. Assoc., India, 11, 47*). During heat treatment of the dehydroxylated oil, isomerization reactions are dominant at 200°; and at 250° and above, polymerization predominates over isomerization (Chowdhury & Mukherji—*Proc. Natl. Inst. Sci. India 22A, 190*). Spectrophotometric data on a polymerized castor oil product are interpreted to indicate that the polymers are linear (Stefanescu & Stanescu—*Rev. univ. "C. I. Parhon" Bucuresti, Ser. Stiint, nat. 4, No. 8, 73*). Practical data on esterifying castor oil with phthalic anhydride are recorded for use in the protective coating industry (Mitra & Saha—*Sci. & Culture, India, 22, 510*). Interpolymers formed by heating castor oil with polysilicic ester are used as lacquers or insulating coatings for electrical conductors (Weigel—*Ger. 833,239, Cl. 22h*). Novel drying oil products are made by sodium reduction of drying oils to fatty alcohols and isomerization of the product (Masuyama—*Japan 875-55*); *Nippon Nogei-Kagaku Kaishi 28, 383*). The drying and protective coating properties are further improved by esterification with acrylic or methacrylic acid (*Ibid.* 30, 290) or anhydrides of organic dibasic acids (Rizzo—*U. S. 2,801,934*); or by conversion to vinyl ether alcohols followed by polymerization (Schneider *et al.*—*J. Am. Oil Chemists' Soc.* 34, 244; Gast *et al.*—*Ibid.* 307). The reference to polymers of the vinyl ethers contains information on molecular weights, viscosity of solutions, and experience in making baked films.

Drying of kamala seed oil, which dries too rapidly, is slowed by conversion to butyl and amyl alcohol esters (Ojha—*J. Sci. Ind. Res., India, 16A, 213*). The drying of linseed oil is also modified for certain uses by conversion to ethylene diglycerol esters (Mlejnek *et al.*—*Chem. prumysl 7, 41*).

Many methods are used to convert nondrying and semi-drying oils to drying oil uses. Published experience on such work contains methods and descriptions of products; and are on: reaction products of styrol with sunflower, olive and linseed oils (Yukhnovskii & Popenker—*Zhur. Priklad. Khim.* 30, 603), vinyl fatty acids (Seher—*Fette-Seifen-Anstrichmittel 58, 1077*), fatty acid polyvinyl alcohol esters (Seavell—*J. Oil Colour Chemists' Assoc.* 39, 99), copolymers of olefinic hydrocarbons and unsaturated fatty acids (Block—*U. S. 2,794,747*), conversion of the fatty acids to pentaerythritol esters (Wakabayashi—*Japan 8722-55*; Schoenemann—*Ger. 865,899, Cl. 12a*) and glycoside esters (Gibbons—*U. S. 2,759,922-3*), reaction products of the fatty acids with 2,6-bis(hydroxymethyl)-*p*-cresol (Misonou *et al.*—*Abura Kagaku 5, 275*), heat treating semidrying oils with nickel nitrate (Nakamura—*Japan 3931-56*), partial saponification of animal oils to separate soaps of drying fatty acids (Toyama & Marumo—*Japan 6124-55*), and oxidation and dehydration of oil of the antarctic whale (Plisov & Tomson—*Trudy Odess. Tekhnol. Inst. Pishchevoi i Kholodil Prom. 6, 73*). Tall oil is processed to specific drying oil products by: esterification with pentaerythritol (Hess—*U. S. 2,791,502*), heat treatment with zinc powder and distillation to separate a drying fraction (Nevin—*U. S. 2,763,638*), polymerization, segregation of polymers and esterification with polyhydric alcohol (Palmer *et al.*—*U. S. 2,794,017*), and heating with vinyl acetate (Culemeyer—*U. S. 2,781,386*).

Diels-Alder reaction products, alkyds of fatty oils are studied in regard to the nature of the reaction and are also the subjects of many patents. The reaction of chloromaleic anhydride and maleic anhydride with eleostearic acids conforms to second order kinetics, but those of methylmaleic anhydride are not strictly second order (Bickford *et al.*—*J. Org. Chem.* 22, 1080). The Diels-Alder adducts of acrolein, acrylic acid, and acetylene carboxylic acid with *trans*, *trans*-9,11, octadecadienoic acid contain six membered rings (Teeter

et al.—*Ibid.* 512). In this work adducts are also made with nitroethylene, β -nitrostyrene, acrylonitrile, methyl vinyl ketone, and methyl vinyl sulfone; and the products are analyzed for structural characteristics. Data are recorded on making soybean oil alkyds with mixtures of phthalic anhydride and isophthalic acid as reference material to maintain oil length and design schedules in varnish-cooking (Miner & Hind—*Off. Dig. Fed. Paint & Varnish Prod. Clubs*, 28, 17). Reference data for making kamala-seed oil alkyds show how the addition of various other oils, solvents, and driers affect "oil length" and gelation; and the scratch resistance, gloss, flexibility, and water resistance of the final films (Sharma & Aggarwal—*J. Sci. Ind. Res., India*, 15B, 608). With this oil alone, "oil length" is poor; it gels and becomes resinous rapidly. Data are recorded, especially for alcoholysis, on reaction rates during the manufacture of pentaerythritol alkyd as reference material (Mraz *et al.*—*Off. Dig. Fed. Paint & Varnish Prod. Clubs* 29, 256). Effects of various catalysts and other factors are considered. Use of iodine as the catalyst in alkyd manufacture is patented (N. V. Koninklijke Stearine Kaarsenfabrieken, Gouda-Apollo—*Brit.* 762,123-4). For making alkyds from tall oil, co-reaction with pentaerythritol (Parker & Pieper—*U. S.* 2,813,841) and a preliminary isomerization treatment (Kleim—*U. S.* 2,776,277) are patented.

Materials are added in alkyd manufacture to induce specific characteristics. Choline chloride inhibits gelation in oil products (Andrews & Peterson—*U. S.* 2,777,329). Substituted carbocyclic monocarboxylic acids containing at least one six-carbon-ring linked to a carboxylic group are added to improve gloss, toughness and adhesion of varnish films made from the alkyds (N. V. Bataafsche Petrol. Maatschappij—*Dutch* 79,614). Replacing some of the polyhydric alcohol with tris (hydroxymethyl)-phosphine oxide in the alkyd yields slightly amber flexible polymers (Albright & Wilson Ltd.—*Brit.* 764,314). Colored batches of alkyds are made for use in inks (Vesce—*U. S.* 2,773,044). Alkyds made with the addition of allyl alcohol are used as adhesives for plastics (D'Alelio—*Ger.* 912,400, Cl. 39c). Alkyds are also modified by mixtures of styrene and acrylonitrile (Daniel & Corkum—*U. S.* 2,748,092), styrene (Toyama *et al.*—*Japan* 5977-55; Schlenker & Hultzsche—*Ger.* 923,931, Cl. 22h) and hydroxylated polybutadiene (Hutchinson—*U. S.* 2,778,806).

Resinous polymers are prepared by: copolymerization of fatty acids with vinyl aromatic compounds and alkoxy polysiloxanes (Lewis Berger & Sons—*Brit.* 758,724), co-reaction of fatty acids, resin acids, and paraformaldehyde (Gayer—*U. S.* 2,744,889), and condensing fatty acids, oils or partial glycerides with polymers of dihydrodicyclopentadienyl vinyl ether (Abel *et al.*—*Ger.* 941,815, Cl. 39c). Copolymerization of butadiene and unsaturated acids yields elastomers that can be vulcanized with sulfur and other materials (Farben-fabrieken Bayer A. G.—*Brit.* 776,305). A porous foam rubber is made from rubber and a reaction product of sulfur monochloride and unsaturated oil (Agesolbakken—*U. S.* 2,810,654).

Isocyanate radicals are incorporated into various oil compounds to produce products ranging from paint base oil to resins and plastics. Placing isocyanate groups at the free hydroxyl groups in partial polyhydric esters of tall oil yields oil suitable for paint making (Culemeyer—*U. S.* 2,812,337). A condensed product of fatty oil acids, fumaric acid, formaldehyde and dicyandiamine or guanlyurea is resinous (Schmutzler—*U. S.* 2,816,874). Foam or cellular plastic material result from: condensation of hydroxy fatty acid glycerides and arylene diisocyanate (Detrick & Barthel—*U. S.* 2,787,601), reacting castor oil, an epoxy resin and aromatic diisocyanate (Barthel—*U. S.* 2,788,335), and by modifying alkyd resins with *m*-tolylene diisocyanate (Phillips—*Brit.* 758,433). A cement suitable for closing the side seams of metal cans is prepared by mixing stoichiometric proportions of polymers of methylenebis (4-phenyl isocyanate) and castor oil (Yoho—*U. S.* 2,769,826).

Much of the literature on epoxidation and epoxide compounds involves fat derivatives. Novel methods developed for epoxidation of fatty materials are based on reactions with hydrogen peroxide in the presence of: sulfuric and acetic acids (Greenspan & Gall—*U. S.* 2,801,253, 2,810,732; Food Machinery Corp.—*Brit.* 769,127; Krimm—*U. S.* 2,813,896; Wahroos—*U. S.* 2,813,878), acetic acid and Amberlite resin (E. L. du Pont de Nemours & Co.—*Brit.* 776,757), formic acid (Food Machinery Corp.—*Brit.* 772,151, *U. S.* 2,774,774), boron trifluoride (Gross—*U. S.* 2,806,045), and sulfuric acid (Swern & Parker—*U. S.* 2,813,885). A method for epoxidation and hydroxylation comprises adsorbing the unsaturated fatty material on alumina and treating with hydrogen peroxide (Payne & Smith—*U. S.* 2,776,301). Fatty material is also peroxidized with *tert*-alkyl peroxy

compounds (N. V. Bataafsche Petrol. Maatschappij—*Dutch* 79,636).

A two-step synthesis of a water-free solution of peracetic acids involves autoxidation of acetaldehyde at low temperature to the monoperoacetate, pyrolyzing this to peracetic acid and acetaldehyde which separate rapidly (Phillips *et al.*—*J. Am. Chem. Soc.* 79, 5982). This product is used for the epoxidation.

Epoxy-modified linseed oil alkyd resins are made to yield tough, flexible, and mar-resistant films (Glenydd & Williams—*Brit.* 762,764). Novel coating compositions are derived from copolymers of epoxy resins and styrene-acrylate (Segall & Cameron—*U. S.* 2,798,861), styrenated esters of epoxy resins (Am. Cyanamid Co.—*Brit.* 757,078), reaction products of cyanuric acid esters of epoxy alcohols and natural oils (N. V. Bataafsche Petrol. Maatschappij—*Brit.* 768,470), blends of amine catalyzed epoxy resins and fatty material (Taylor—*U. S.* 2,794,007), and *in situ* prepared alkyd-epoxide product (Henkel & Cie—*Brit.* 758,728). Glycidyl polyethers of polyhydric phenols are reacted with drying oil fatty acids and then with phosphoric acid in the preparation of a metal primer ingredient (N. V. Bataafsche Petrol. Maatschappij—*Dutch* 81,230).

The text of some communications on epoxidation involved rate, properties of products, and other basic information. The effects of drying, amount and fineness of catalyst (Dowex I), temperature, and acetic acid concentration on epoxidation of tetrahydrophthalic-1,5-pentandiol, -1,4-butanediol, -ethylene glycol, and diethylene glycol polyesters, unsaturated alkyd, and butyl oleate are determined to indicate the optimum conditions (Pearce & Kawa—*J. Am. Oil Chemists' Soc.* 34, 57). From polarographic data the specific reaction rate constants for perlauric acid epoxidation of vinyl laurate, methyl oleate, and vinyl oleate are determined and expressed with the Arrhenius equation (Ricciuti *et al.*—*Ibid.* 134). Data on preparation, polymerization and properties of polymers and copolymers of vinyl epoxystearates are recorded for practical use (Silbert *et al.*—*J. Polmer. Sci.* 21, 161). In the epoxidation of vinyl oleate with peracetic acid for the preparation of vinyl epoxystearates, internal bonding is 225 times that of vinyl double bonding. The epoxy polymerization with boron trifluoride catalyst can be controlled by employing catalyst-ether complex absorbed in a paraffin barrier layer (Wagner—*Ibid.* 26, 329). Two newly described catalytic processes for *in-situ* epoxidation involve, respectively, circulation of reaction mixture through a fixed resin catalyst bed and employment of expendable amounts of resin catalyst (Gall & Greenspan—*J. Am. Oil Chemists' Soc.* 34, 161). In preparation of resin esters by reaction of the epoxy resin of the 4,4'-isopropylendiphenol-epichlorohydrin type, tung oil fatty acids and long chain fatty acids, the relative order of activity of hydroxy and epoxy groups in the resin is the same and the esters retain about half of the conjugated triene originally present in the tung oil acids (Goldblatt *et al.*—*Ind. Eng. Chem.* 49, 1099).

Phosphatized metallic soap complexes, useful as driers, are formed by the coprecipitation of alkali metal soaps and alkali ortho- poly-, or pyrophosphates (Collins & Leopold—*U. S.* 2,793,962). The catalytic effect of cobalt on drying of oils is suppressed by increased complexing with 1-nitroso-2-naphthol (Doadio & Montequi—*Grasas y aceites, Spain*, 8, 76). The oxygen gain during air drying of pentaerythritol tall oil varnish and raw linseed oil with and without drier is recorded with correlations to setting time and other properties (Mueller & Smith—*Ind. Eng. Chem.* 49, 210). Paints are made with addition of zinc stearate as a flattening and sealing agent (Davis & Stafford—*U. S.* 2,809,121) and addition of barium borate as a moldicide (Buckman—*U. S.* 2,818,344).

The fat derivatives made to serve as plasticizers, mostly for vinyl type plastics, are: alkenyl esters of diepoxy fatty acids (Phillips & Frostick—*U. S.* 2,779,771), epoxidized fatty acid diacetylated glycerides (Ault & Feuge—*Brit.* 775,326), epoxy stearic acid esters (Sprules & Marks—*U. S.* 2,802,800), epoxidized ricinoleic acid derivatives (Dazzi—*U. S.* 2,786,039), epoxidized oils (Greenspan—*U. S.* 2,810,733), various alkyd preparations (Shigeno & Komori—*Technol. Repts. Osaka Univ.* 6, 179; Büchner—*U. S.* 2,780,643; Harrison—*U. S.* 2,808,418), carbonato nitriles of fatty acids (Riedeman—*U. S.* 2,811,533), acetoacetic acid esters of castor oil (Bader—*U. S.* 2,812,340), carbonato amides of fatty acids (Rohm & Haas Co.—*Brit.* 769,622), interesterified products of pentaerythritol and oil or fat (Shimo *et al.*—*Japan* 7730-55), aromatic-fatty acid ester compounds (Watanabe—*Japan* 4345-55), and natural oils heated with copolymers of butadiene and styrene (Nelson & Koenecke—*U. S.* 2,802,842). Plasticizers made by isomerizing soybean oil fatty acids, condensing with maleic anhydride and acrylic acid, and converting to esters of methyl, ethyl, propyl, butyl and allyl alcohols, and epoxy derivatives of these are characterized with respect to plasticizing polyvinyl chloro-

ride (Danzig *et al.*—*J. Am. Oil Chemists' Soc.* 34, 136). All the esters, except allyl, and all the epoxy esters are compatible with the plastic and appear to be primary plasticizers. The use of esters of epoxy hydroxy-acetoxy, and polyacetoxystearic acids as plasticizers for copolymers of vinyl acetate and vinyl epoxystearate are described with regard to preparation of the plastics and properties induced by the plasticizers (Silbert & Port—*Ibid.* 9). Addition of polyethylene-fatty acid amides to polyethylene plastic reduces film-to-film coefficient of friction and tendency to blocking (Barker *et al.*—*U. S.* 2,770,608, Symonds—*U. S.* 2,770,609). Hydroxy fatty acid soaps are added to vinyl chloride containing resin dispersions as dispersing agents and to improve the heat stability of the plastigels (Patton & Hall—*U. S.* 2,794,791).

Methods and studies on analytical and test methods for protective coating and fatty resin products are published. A scheme for modified unsaturated oils contains reduction titration for epoxides, determination of saponification and hydroxyl values, method of separating styrenated oils with solvents, procedure for determining molecular weights, and directions for interpreting the data (Winterscheidt—*Seifen-Öle-Fette-Wachse* 82, 154, 181). A cascade fractionating-type molecular still is applied to segregation of styrenated methyl esters of linseed oil fatty acid for further analysis and identification (Tiong *et al.*—*Anal. chim. acta.* 14, 228). Polystyrene in oils, alkyds, and epoxy resin esters can be determined quantitatively by infrared absorption spectrometry (Fraser & Pross—*Off. Dig. Fed. Paint & Varnish Prod. Clubs* 29, 75). A committee on determination of oxirane oxygen in epoxy compounds recommends direct titration with hydrogen bromide-acetic acid (Holt *et al.*—*J. Am. Oil Chemists' Soc.* 34, 476). A method for small amounts of thiocyanate in polyurethane resins and coating is based on adding standard hydrochloric acid and a few crystals of quinhydrone to a cyclohexanone solution containing the sample and some ethyl amine, and titrating potentiometrically with standard alkali (Navyazhskaya—*Khim. Prom.* 1956, 432). Infrared spectra data on linseed oils, glycerophthalate resins, and modified oils are recorded as reference material for determining oxygen compounds, olefinic bonds, conjugation, catalyst efficiency and course of bodying (Helme & Molines—*Peintures, pigments, vernis* 33, 524; *Rev. franc. corps gras* 4, 189). Similar information on the ultraviolet absorption of raw and bodied tobacco seed oil is recorded (Tugtepe—*Rev. fac. sci. univ. Istanbul* 21c, 157). Drying oils of the same kind show a constant relation between viscosity and acetone value irrespective of the manner of bodying, thus providing a means of identification (Montegui *et al.*—*Grasas y aceites, Spain* 8, 11).

A method of approximating the degree of polymerization of oils is based on the molecular weight of the acetone-insoluble as determined by the static osmotic method (Boyer—Kawenoki & Petit—*Bull. soc. chim. France* 1957, 690). Viscosities of variously polymerized oils are recorded and interpreted with regard to elasticity, temperature dependence of viscosity, and other aspects (Nakagawa & Seno—*Bull. Chem. Soc. Japan* 29, 471).

Methods are published for testing baking properties of alkyd resin films and the properties of the films, such as resistance to water, alkali, and acetic acid; impact flexibility; initial color, color retention, initial gloss, and gloss retention; and zinc oxide stability; and mineral spirits tolerance (Bratt—*Western Paint Rev.* 41, No. 1, 15A).

During the drying of chinawood oil and linseed oil mixtures with fast driers humidity at temperatures above 20° has no effect, with intermediate driers humidity is unimportant only above 40°; whereas with weak driers drying depends a great deal on relative humidity (Kaufmann & Gulinsky—*Deut. Farben-Z.* 11, 90). Wax present as an impurity in linseed oil causes visible defects in films of high-gloss enamels (Mort—*J. Oil & Colour Chemists' Assoc.* 40, 24). In studies on epoxy coatings: absorbed oxygen, increased density, and loss in weight are recorded; film failure is attributed to development of stress from dimensional changes; and most stable films are thicker than 1 mm., are cured at below 250°, and formulated to contain the least unreacted epoxide groups after curing (Park & Blount—*Ind. Eng. Chem.* 49, 1897). A study of changes in the constants of linseed oil films as a function of aging indicates that change of saponification value with time may be utilized as an index of serviceability of the films (Pamfilov & Ivancheva—*Zhur. Priklad. Khim.* 30, 305). The effect of a range of organic solvents on aged linseed-oil films is recorded and discussed with regard to swelling, diffusion, and leaching (Stolow—*Nature* 179, 579). Exposure characteristics of many clear finishes for exterior wood surfaces (*Golden Gate Paint & Varnish Club—Off. Dig. Fed. Paint & Varnish Prod.*

Clubs 28, 1001) and of tall oil and soybean oil phthalic alkyds films (Sill & Mueller—*Ibid.* 29, 170) are evaluated to serve as reference for the coating industry.

Application of electron microscopy to films of drying oils indicates that crystalline segregations are partly formed spontaneously and partly produced by fatty acids and waxes; and blooming is connected with the chlorophyll-(phaeophytin-) content (Kaufmann *et al.*—*Fette-Seifen-Anstrichmittel* 59, 522).

Methods, basic data, and reference curves are recorded to serve for the polarographic analysis of driers (Kaufmann & Bernard—*Ibid.* 843). Measurement of the heat of reaction caused by action of metal catalyst on defined peroxidized turpentine offers the possibility of a direct evaluation of the drying effect (Morgner & Sutor—*Ibid.* 58, 1067).

FATTY MATERIALS IN LUBRICATION, METAL WORKING AND TEXTILE OILING. New techniques and formulas in grease making are devised to simplify production and to attain special characteristics in the final product. One lubricant grease gel is manufactured by *in situ* saponification of lower alcohol esters of hydroxy fatty acids in a still in a manner that the released lower alcohols evaporate (Smith—*U. S.* 2,801,220). In another *in situ* made grease process, the original oil is extracted from the soap gel with liquefied gaseous hydrocarbons and replaced by a more desirable lubricant oil (Peterson & Bondi—*U. S.* 2,782,165). Novelty in one process is based on subjecting to very high rates of shear to reduce processing time and temperature (Esso Res. & Eng. Co.—*Brit.* 776,548). In one process the soap thickener is made in aqueous solution and then incorporated in hydrocarbon oil with heating (Petrovicki—*Ger.* 833,533, *Cl.* 23c). Soap thickener for high temperature greases is made by saponification and fusion of alkali and fat (Morway *et al.*—*U. S.* 2,790,769). A grease of high water resistance and high drop point contains lubricant oil, soaps of high and low molecular weight acids and soap of tridecylmethoxyacetic acid (Mikeska *et al.*—*U. S.* 2,799,656). "Polyol" compounds are added to grease containing some lead soap and menhaden oil to raise the Timken test values (Langer—*U. S.* 2,785,130). In another extreme-pressure grease, part of the gelling soaps are lead soaps of fish oils (Fainman & Swenson—*U. S.* 2,790,770). A lubricant grease with high drop point and good water-resistance is thickened with aluminum soaps containing equal moles of aromatic and fatty acid radicals (Hotten & Echols—*U. S.* 2,768,138). Two patented greases are thickened with soaps of acids derived from petroleum (Morway *et al.*—*U. S.* 2,779,736; Giammaria—*U. S.* 2,790,767). Addition of ditridecyl azelate and other diesters improves high temperature performance of greases made with sodium or lithium soaps (Dilworth & Roach—*U. S.* 2,791,560). The novelty of two greases containing lithium fish oil soaps pertains to special mineral oil components (Bartlett *et al.*—*U. S.* 2,801,970). A high viscosity product contains mineral oil, lithium soap and a synthetic oil made by esterifying fatty alcohols with dibasic acids (Kolfenbach *et al.*—*U. S.* 2,782,166). One review on lubricant greases treats mainly lithium soap greases (Luckenbach—*Footnote Prints* 26, No. 2, 10).

Fat derived additives are incorporated into greases to induce specific characteristics. Fatty alcohols stabilize the gel of lithium stearate greases (Butcosk—*U. S.* 2,769,781). Fat derivatives of 9,10-dihydroxy-1,4-diaminoanthracene are used to inhibit oxidation and to serve as a soap substitute (Cottle *et al.*—*U. S.* 2,816,074). Difatty acid derivatives of *p*-aminophenols inhibit oxidation and discoloration (Young *et al.*—*U. S.* 2,795,546). Difatty acid derivatives of dithiocarbamic acid are metal deactivators and rust inhibitors (Liche—*U. S.* 2,812,306). Grease is stabilized against decomposition during heating and corrosion by addition of a fatty acid selenide (Woods & Bollinger—*U. S.* 2,813,828).

Various materials are used as partial or complete substitutes for soap as thickening agents. Those recently patented are amino alcohols having 10-50 carbon atoms (Morway & Bartlett—*U. S.* 2,801,969), oxo alcohols and acids of very high molecular weight (Bartlett *et al.*—*U. S.* 2,801,971), alkali fusion products of high molecular weight alcohols (Bartlett *et al.*—*U. S.* 2,801,972), alkali fusion products of aliphatic aldehydes and cyclic alcohols (Morway *et al.*—*U. S.* 2,801,973-4), alkali fusion products of polyvinyl esters (Bartlett & Morway—*U. S.* 2,801,975), alkali fusion products of polysaccharides (Morway & Bartlett—*U. S.* 2,801,976), alkyl or aryl esters of orthoboric acid (Phorilla & Smith—*U. S.* 2,815,325), *N*-octadecylterephthamate (Dreher *et al.*—*NLG1 Spokesman* 20, No. 11, 10), bentonite-quaternary ammonium compound complexes (Erickson—*U. S.* 2,767,175), and organosiliceous materials (Alexander & Iler—*U. S.* 2,818,385; Liche & Hayne—*U. S.* 2,805,994). Greases thickened with inorganic

gelling agents are rendered water resistant by addition of 5-35% quinoline or alkyl-substituted quinoline (Marshall & Steininger—*U. S. 2,766,205*).

A study of retention of cetane by calcium stearate-cetane gels shows that: only additives that reduce the viscosity at the high temperature-melt are effective stabilizers at room temperature; and the observed behavior cannot be interpreted in terms of the phases present in the gel or compound formation between additive and soap, but must be related to such variables as particle size and shape, degree and kind of particle interaction, rate of cooling, and degree of dispersion at the temperatures of the phase from which the gel is formed (Vold & Coswell—*J. Phys. Chem.* 61, 529). An apparatus is described for studying lithium soap-oil greases by using differential thermal analysis (Cox & McGlynn—*Anal. Chem.* 29, 960). The apparatus is used to reveal a previously unreported mesomorphic phase occurring at 225-9°. Known short methods for analysis of lubricant greases are reviewed with discussion on interpreting data with respect to composition (Kaiser—*Chem. Tech., Berlin* 8, 388).

Various fat derived additives are used in lubricant oils. Rust inhibiting properties are imparted by: a mixture of dimerized acid and alkyl ammonium dialkyl orthophosphate (Cantrell & Peters—*U. S. 2,773,032*), small amounts of a mixture of fatty-alkenylsuccinic acid, 2,6-di-*tert*-butyl-4-methylphenol, sulfurized oleic acid, nonanic acid and dimethyl silicone polymer (Lurton & Westcott—*U. S. 2,775,560*), mixtures of alkyl (fatty) succinamate (Messina—*U. S. 2,783,206*), product of reaction of isoamyl octyl acid phosphate with fatty amine (Gottshall *et al.*—*U. S. 2,786,028*), fatty acid derivatives of terephthalamide (Lowe & Stewart—*U. S. 2,810,696*), fatty alcohol esters of molybdic acid (Benoit & Abbott—*U. S. 2,805,997*), fatty alcohols of metals of group I or II of the periodic table (Sacony Mobil Oil Co.—*Brit. 774,119*), and certain mono fatty acid amides (Clementson—*Swed. 156,915*). Copolymers of acrolein or methyl vinyl ketone and fatty amines, mercaptans or alcohols (Hollyday—*U. S. 2,769,783*) and difatty acid esters of substituted propanediols (Bell & Lappin—*U. S. 2,798,083*) are added to depress pour point and improve viscosity characteristics. The extreme-pressure additives include a combination of lead soaps of tall oil, chlorinated wax, and dichlorostearic acid (Fainman—*U. S. 775,558*), a mixture of tartaric acid, sorbitan monoöleate and polyoxyethylene sorbitan monoöleate (Bondi *et al.*—*U. S. 2,788,326*), thio-orthoformate plus fatty mercaptoles (Matuszak & Ready—*U. S. 2,796,405*), sulfurized esters of fatty acids, alcohols or amines (Chapman *et al.*—*U. S. 2,809,935*), and sulfurized products of chlorinated fatty acids (Saives—*U. S. 2,812,307*). Lubricant oils stabilized against oxidation contain: reaction products of polyisobutylene, phosphorus pentasulfide and fatty alcohols, mercaptans, and esters (Fields—*U. S. 2,768,954*), dilauryl selenide (Lindert—*U. S. 2,792,346*) and mixtures of castor oil and fatty alcohol esters of dicarboxylic acids (Thornley—*U. S. 2,796,400*). A mineral steam turbine lubricating oil contains dimeric fatty acids as emulsion depressors (Cunningham & Dinsmore—*U. S. 2,794,782*). A combination of dimerized linoleic acid, sorbitan monoöleate, and isoamyl octyl acid phosphate is used in the same type of lubricant to increase resistance to moisture and oxidation (Gottshall *et al.*—*U. S. 2,789,950*).

Synthetic esters useful as lubricants are the diester of 2,2-dimethyl-3-isopropyl-1,3-propanediol and fatty acids (Bell & Lappin—*Brit. 767,455*) and reaction products of ethylene oxide with *tert*-dodecyl mercaptan (Esso Res. & Eng. Co.—*Brit. 763,543*).

A metal cutting oil, used as a coolant and lubricant for metal working, is made by treating castor oil or ricinoleic acid with potassium hydroxide (Epstein—*Fr. 1,001,883*). Other new metal cutting oils are aqueous emulsions of mineral lubricating oil emulsified with various fat derived emulsifiers (Cafecas—*U. S. 2,780,598*; Prust—*U. S. 2,790,771*, Oathout—*U. S. 2,802,786*; Laug—*U. S. 2,811,489*; Francis & Piatt—*U. S. 2,818,386*; N. V. Bataafsche Petrol. Maatschappij—*Dutch 80,211, 80,597*).

Bacteriological studies are made on soluble cutting oils because they harbor bacteria which cause spoilage of the product and some may be pathogenic. Fatty materials in these that may be oxidized by bacteria and types of bacteria that are involved are indicated by Ellis *et al.* (*Applied Microbiol.* 5, 345). An evaluation of 232 bacterial inhibitor compounds for control of aerobic microorganisms in such oil is published (Bennett—*Soap Chem. Specialties* 32, No. 10, 47; No. 11, 46). In a test where known positive tuberculous sputum was added to different soluble oil emulsions, viable mycobacteria and staphylococci were isolated six weeks later (Samuel-Maharajah

et al.—*Applied Microbiol.* 4, 293). Twenty-six species of organisms, including among others, *Diplococcus pneumoniae*, *Klebsiella pneumoniae*, *Shigella madampensis*, *Micrococcus pyogenes*, and *Streptococcus pyogenes* were isolated from used metal cutting oil emulsions (Tant & Bennett—*Ibid.* 332).

Metal sheets intended for shaping by drawing dies are coated with soap solution and dried as a means of lubrication (Peterson—*U. S. 2,809,129*). A mixture of aluminum stearate and aviation oil is used as a lubricant of dies for forming plastic glass substitutes (Bocharnikov *et al.*—*U. S. S. R. 104,830*). An emulsion containing fatty acid-glycol esters, graphite, and sugar is used to lubricate glass molds (Cupper—*U. S. 2,784,108*).

Emulsions for lubricating textiles for spinning contain mineral oil and: olein plus diethylene glycol alkyl ether (Stewart—*U. S. 2,787,594*), a combination of bodied sperm oil, alkyl phosphate, dibutyl ethanalamine and a nonionic surface active agent (Fortess *et al.*—*U. S. 2,810,692*) and a combination of alkyl phenol and polyoxyethylene fatty acid condensate (McLean & Way—*U. S. 2,810,694*).

Deterioration of Fatty Materials

REVIEWS. The reviews and general information communications pertaining to stability and deterioration of fatty substances were written on: influence of fat spoilage on the storage of some raw materials and products in the food industry (Rutkowski—*Wiss. Z. Karl-Marx-Univ. Leipzig Math-naturwiss. Reich* 5, 327), causes and effects of rancidity (Demott & Brandt—*Milk Plant Monthly* 46, No. 1, 30), peroxide estimation in fats (Hartman—*J. New Zealand Inst. Chem.* 20, 132), history and standardization of the active oxygen method for determining fat stability (Newby—*J. Am. Oil Chemists' Soc.* 34, 394), home-made equipment for the active oxygen method for fat stability (Gonzales-Quijano—*Grasas y aceites, Spain*, 8, 64), symposium on antioxidants (abstracts of papers) (*Chemistry & Industry* 1957, 721), stabilization of food by means of nordihydroguaiaretic acid (Milhelie—*Farm. Glasnik.* 13, 56), antioxidant action of ascorbic acid (Cerutti—*Olii minerali, grassi e saponi, colori e vernici* 34, 41), and deterioration reactions involving phospholipides and lipoproteins (Lea—*J. Sci. Food Agr.* 8, 1).

TESTS FOR DETERIORATION, STABILITY, AND ANTIOXIDANTS. Because of the difference in nature of fat-containing materials and of fats themselves, and differences in preparation, deterioration tests may be fitted to the material involved. Thus, because the peroxide value of ham fat is affected during rendering of test samples, the test for free fatty acids was considered most significant in checking age of ham or deterioration of the ham fat (Kemp *et al.*—*Food Tech.* 11, 321). Acidity indicator tests were favored over determination of peroxide value for appraising deterioration and stability of unsaturated fats (Täufel & Vogel—*Ernährungsforschung* 1, 142). This test was unsuitable for fats containing low molecular weight glycerides. Baker *et al.* (*Cereal Chem.* 34, 226) correlated the relationship between fat acidity and moisture in stored grains and showed the effect of damaged grain, as basic information for evaluation of grain deterioration. In studies on stored lard and beef tallow, organoleptic observations were the best criteria for both; and among laboratory tests the peroxide value, α -carbonyl value and acidity test were recommended for lard; and spectral curves, color changes and acidity were most significant on beef fat (Lyaskorskaya *et al.*—*Trudy, Vsesoyuz. Nauch.-Issledovatel Inst. Myasnaya Prom.* 1955, No. 7, 78). A "heat test" recommended for distinguishing fresh and old samples of butter and margarine is based on observing the odors at 20° intervals during heating and also determining the smoke points (Täufel and Serzisko—*Ernährungsforschung* 2, No. 1, 121). Characteristic odors of old and fresh samples at the various temperatures were described. A combination of peroxide value and aldehyde value was proposed as an indication of the extent of oxidation that had taken place in a fat (Holm *et al.*—*J. Am. Oil Chemists' Soc.* 34, 606). These data may give an insight to the amount of deterioration that has occurred in the fats before refining and may also bear a relationship to the flavor stability of the refined material and the margarine made from it.

A modification of the iodometric method for determination of peroxide value in fats and one for its application to meat products dealt principally with handling and reaction under carbon dioxide atmosphere to improve accuracy (Sedlacek *et al.*—*Roczniki Panstwowego Zakladu Hig.* 7, 293; Solov'ev & Rubashkina—*Myasnaya Ind. S. S. R.* 28, No. 2, 41). The method was also designed so the color developed is read as absorbancy at 450 m μ for the measure of the rancidity (No-

gami *et al.*—*Yakuzai-gaku* 16, 7). Another colorimetric peroxide test is done with a spot of fat on filter paper and is based on the color developed with a ferrous ammonium thiocyanate reagent (Täufel—*Fette-Seifen-Anstrichmittel* 59, 87; Täufel & Serzisko—*Ibid.* 827; Täufel & Vogel—*3rd Intern. Bread Congr.* 1955, 105). Exposing the spots to ultraviolet light to accelerate oxidation before spraying on the reagent will give data on the relative stability of fats such as is obtained by the Swift stability test. Coleby (*Chemistry & Industry*, 1957, 1123) found that the ferric thiocyanate procedure results for peroxide value are affected by the presence of phosphate groups (phospholipides) and thus are of doubtful quantitative validity. Studies have shown that data from the Wheeler-Hadorn idiometric peroxide method are mostly higher than those of the modified Sully method (Schmidt—*Fette-Seifen-Anstrichmittel* 59, 837).

A study of the specific refraction of aging fatty oils has indicated that this characteristic may be useful in some cases to evaluate extent of deterioration (Lüdde—*Pharmazie* 11, 254).

The A. S. T. M. bomb method for testing stability of fats and fatty foods (potato chips, crackers) is 14 times faster than the "Accelerated Oxidizability Method" and 40–50 times faster than the Schaal oven method and results correlate well with them (Gearhard *et al.*—*J. Am. Oil Chemists' Soc.* 34, 427).

The thiobarbituric acid method for determination of oxidative changes in butter was applied to sweet-, normally soured-, and cultured-cream butters after six months storage (Cerna *et al.*—*Z. Lebensm.-Untersuch. u. -Forsch.* 105, 86). The data were recorded as fundamental information for applying the test on different types of butter. On cathode ray irradiated milk the results of this test did not correlate with the off-flavors produced (Streuli—*Mitt. Lebensm. u. Hyg.* 47, 221). Other reports on the thiobarbituric acid test for rancidity have shown that on lard it agrees closely with the peroxide value (Romeo & Gonzales-Quijano—*Grasas y aceites, Spain*, 7, 229, 8, 73), it is a sensitive indicator for the keeping quality of bacon (van Koetsveld—*Landbouwk. Tijdschr.* 68, 824), and it may be used as an index of quality of stored fishery products (Yu & Sinnhuber—*Food Tech.* 11, 104).

The method of evaluation of fat spoilage based on carbonyl value was altered by replacing the titration until a color change with evaluation from a curve developed from potentiometric titration (Barro-Raffel & Jacini—*Olii minerali, grassi e saponi, colori e vernici* 33, 381).

The method for determining decomposition of cream and butter based on separation of volatile acids from water insoluble acids was modified by using column chromatography for the separation of these acids (VanDame—*J. Assoc. Off. Agr. Chemists'* 40, 407). This modification is suitable for butter but not on cream samples.

Spectrophotometry was applied to appraising deterioration in certain fats. The transmittance at 280 μ of the steam distillate of fresh milk fat is over 80%, for unstable milk fat 79–40% and at below 40% the sample is considered unsuitable for human consumption (Machulis—*Molochnaya Prom.* 18, No. 6, 31). In virgin oil and cocoa oil increases in absorption at 270 μ indicate progressing oxidation (Wolff—*Ann. fals. et fraudes* 50, 149).

Various schemes were developed for determination of the antioxidant present in oils. Wolff (*Rev. franc. corps gras* 4, 254) recorded extinction coefficients of ethanol solutions of gallic acid, butylated hydroxyanisole and dibutyl hydroxytoluene at 72° at wave lengths of 242, 253, 274, 285, 290, and 300 μ ; and with solvent segregation techniques used the data in a scheme to analyze fats for these antioxidants. A scheme of analysis based on paper chromatography by Mitchell (*J. Assoc. Offic. Agr. Chemists* 40, 909) is for the same antioxidants plus nordihydroguaiaretic acid. In a procedure for determination of 2,6-di-*tert*-butyl-*p*-cresol in fats, the compound is separated from other antioxidants and fat by column chromatography, eluted, and measured through absorbance at 284 μ (Phillips & Hinkel—*J. Agr. Food Chem.* 5, 379). A method for analysis of commercial butylated hydroxyanisole is based on the ratio of absorbances at 10.74 and 10.92 μ (Whetsel *et al.*—*Ibid.* 602). The alkyl gallate antioxidants in fats may be determined quantitatively by taking up oil in petroleum ether; extracting propyl gallate with water, adding ferrous tartrate and acetate buffer, and measuring at 530 μ ; the octyl- and dodecyl-gallate are then extracted from the petroleum ether solution with methanol and measured at 550 μ (Vos *et al.*—*Analyst* 82, 362).

MECHANISMS. In a study of the kinetics of autoxidation of pure methyl oleate and pure methyl elaidate, the rate of re-

action increased proportionately with the degree of oxidation, and the energy of the reaction was 17 calories per mole (Kern *et al.*—*Makromol. Chem.* 22, 47). In the autoxidation of cuttlefish oil exposed to air, the peroxide value reached a maximum in 25 days, conjugated dienoic acids increased up to 25 days while higher unsaturates decreased, and the amount of sesamol and sesamolin remained unchanged for 50 days (Oikawa—*Kagaku to Sosa* 9, 15). In mixtures of peanut oil and ethyl stearate, the relative proportion of unsaturated acid glycerides present mainly determine the end of the induction period and peroxide values during the initial stages of autoxidation, but induction periods of the mixtures terminate at the same peroxide value as did that of the peanut oil (Karthar—*J. Sci. Ind. Research, India*, 16B, 270). In similar investigations on several oils and fatty acids, peroxides were formed not only more readily from linoleic than from oleic acids, but also were more stable (Kirjakka & Nieminen—*Suomen Kemistilehti* 27A, 207). When using phthalocyanines of iron, magnesium and nickel as autoxidation catalysts for methyl oleates, peroxides are decomposed rapidly even at low temperatures and at 60° only the hydroperoxides are present (Paquot & Gallelaud—*Olii minerali, grassi e saponi, colori e vernici* 34, 330). Data on the rancidification of sesame oil in the presence of aluminum, tin, copper, brass, iron, stainless steel, glass and oxides of various metal was determined to serve as fundamental information for storing the oil (Prakash *et al.*—*J. Proc. Oil Technologists' Assoc., India*, 12, 1).

Analysis of the autoxidation products of erucic acid and its methyl and propyl esters showed the presence of keto-hydroxyl, unsaturated ketonic, and epoxy derivatives, in addition to 13,14-dihydroxybehenic acid (Skellon & Taylor—*J. Applied Chem.* 7, 67). In an 80-year-old sample of almond oil, 75% of the double bonds had been autoxidized to produce alcohols, ketones, epoxy compounds, and glycol (Paquot & Querolle—*Oleagineux* 12, 225). The physical and chemical characteristics of 25 samples of butter after 25 and after 52 years of storage were recorded (Inikhov—*Sbornik Dokladov Vsesoyuz. Soveshchan po Molochnomu Delu* 1955, 317).

In a comprehensive study of oxidative deterioration of fish products the following aspects were investigated: (a) mechanism of oxidation of oil in fish tissue, (b) reaction between fish oil and protein in fish tissue, (c) oxidation of extracted fish oil and (d) oxidation of tuna pigments (Stansby *et al.*—*Com. Fisheries Rev.* 19, No. 5a, 24, 27, 32, 35). The hematin compound content of various species of fish was directly correlated with catalytic effect on linoleate oxidation and rate of oxidation of the fish tissue; this effect was inhibited by antioxidants. Reactions involved in development of brown color include: (a) reactions between oil and protein at carbonyl and amino groups, (b) oxypolymerization and (c) oxidation of unsaturated oils. Antioxidants block b and c; whereas pro-oxidants divert reaction from a and c.

STABILITY OF FAT PRODUCTS. Beef fat rendered from unsalted tissues at 70–75° in a continuous apparatus could be stored at just above 0° for a year (Komarova—*Trudy, Vsesoyuz. Nauch. Issledovatel. Inst. Myasnoi Prom.* 1955, No. 7, 96). This report contains data on changes occurring in the fat when stored at various temperatures down to –18°. Similar work on lards was on storage temperatures of 40°, 18° and 4° and for periods of 10 days to 8 months (Materanskaya—*Myasnaya Ind. S. S. S. R.* 28, No. 1, 48). A sample that was rendered under carbon dioxide was of good quality. Walnut oil is of general low stability; one must therefore select good raw material, store and handle with care, and treat extracted oil with an antioxidant (Durant-Veron and Prevot—*Rev. franc. corps gras* 4, 502). A survey of samples of vanaspati (Indian hydrogenated fats) has indicated that the stability correlates with the amount of polyunsaturated compounds present (Patil & Magar—*J. Sci. Ind. Res., India*, 16B, 43).

In a comprehensive study on storability of beef fat, the criterion used was loss of carotene; and the influence of moisture, salt, sugar, lactic acid, acetic acid, ascorbic acid, air and various storage temperatures were evaluated (Laphev—*Myasnaya Ind. S. S. S. R.* 28, No. 3, 49). Best preservation of carotene content, color, and organoleptic quality of beef fatty tissue was attained by dipping in 1% salt solution and chilling at –8° refrigeration temperature before freezing.

Nonwashing of butter, as compared to washing, is not a significant factor in deterioration of flavor (White *et al.*—*Can. Dairy Ice Cream J.* 36, No. 1, 25). During autoxidation at 98°, Indian buffalo milk fat absorbs about twice as much oxygen as does cow milk fat. (Vachha *et al.*—*Indian J. Dairy Sci.* 10, 6). During the oxidation of butterfat, the decomposition of carotene is high during the induction period, but the loss rate decreases with increasing oxidation (Schuller—*Milch-*

wissensch. Ber. 7, 1). The carotene and vitamin A added to margarine in commercial fortification practices are retained to the extent of 94% or better for a two month storage period at 75°F. (Marusich *et al.*—*J. Am. Oil Chemists' Soc.* 34, 217). The influence of various commercial packaging materials on the deterioration of margarine during storage at various temperatures from -25° to 20° has been recorded (Schmidt—*Neue Verpackung* 10, No. 1, 32). Parchment and plastic laminated aluminum foil are superior to others.

Experimental animal diets made with glucose are much more susceptible to rancidification than are comparable diets made with sucrose (Thomson & Hegsted—*J. Nutr.* 60, 361). Data on rancidification of fat in canned soups and its effect on quality of the soups have been recorded as fundamental information for soup canning (Rutkowski—*Olii minerali, grassi e saponi, colori e vernici* 34, 333). In this study the influence of antioxidants on the keeping quality of the soups was smaller than in the case of pure fats. The half-lives in days of tocopherol alone, in many solvents, and in methyl esters of fatty acids at 60°, 80°, and 120° have been recorded as fundamental information on the stability of this material (Lips—*J. Am. Oil Chemists' Soc.* 34, 513). In the fatty ester tests, deterioration increased rapidly as the unsaturation of the solvent increased.

Feeding dairy cows rations containing commercial antioxidants increased tocopherol levels of the blood plasma and of the milk, and improved stability of the milk (DeLuca *et al.*—*J. Dairy Sci.* 40, 877). The most effective antioxidant and inhibitor of copper induced off-flavor in the milk was *N,N'*-diphenyl-*p*-phenylenediamine. In tests on broilers, there was no difference in the stability of depot fats from birds fed unstabilized fat, or fat stabilized with commercial antioxidants (Seidler *et al.*—*Poultry Sci.* 36, 449).

ANTIOXIDANTS. Kartha (*J. Sci. Ind. Res., India*, 16B, 220, 271) studied the influence of various antioxidants on the characteristics (iodine and peroxide values) of peanut oil and also the disappearance of the antioxidants. The antioxidants, in addition to inhibiting oxygen adsorption, destroy some peroxides present and peroxide developed in the presence of antioxidants did not reduce iodine value. Heimann and Pezold (*Fette-Seifen-Anstrichmittel* 59, 330) suggested that the prooxidant action of antioxidants is caused by an activation energy being higher than that of stabilization. This develops at higher temperature, through action of light, and with large excesses of antioxidants.

Several studies are on comparing the efficiency of antioxidants. Everson *et al.* (*J. Am. Oil Chemists' Soc.* 34, 81) rated the efficiency in lard of the present commercial antioxidant compounds on the bases of a "catechol index." That is, their efficiencies for prolonging the induction period were compared to that of catechol. The α -glycerophosphoric acid had antioxidant activity comparable to orthophosphoric acid while the β -isomer was inactive (Urakami—*Bull. Chem. Soc., Japan* 29, 921). Among alkylated phenols, those with alkyl groups in the *o*-position are antioxidants, two alkyl groups here increase the activity, and additional alkyl groups in the *p*-position further increase activity (Miller & Quackenbush—*J. Am. Oil Chemists' Soc.* 34, 249). In comparisons of phenols containing various inorganic and organic substituent groups, antioxidant activity is exhibited by all phenols which had electron-repelling groups in the 2- and 6-positions and no activity is apparent with electron-attracting groups in these positions (*Ibid.* 404). In similar comparisons by Penketh (*J. Applied Chem.* 7, 512), it was observed that as the oxidation potential of the substituent of the phenol derivative decreases the antioxidant activity increases. He recommended the measurement of oxidation potential as a preliminary assessment of possible antioxidants and recorded data for over 100 compounds. Antioxidant properties of various alkylresols (Kawai *et al.*—*J. Pharm. Soc.* 76, 660) and catechol derivatives (Tamura *et al.*—*Nippon Nogeikagaku Kaishi* 28, 4, 24, 28, 490, 679) were also compared. With cresol, introduction of alkyl group having 4-5 carbon atoms gives the strongest action. Most of the alkyl-catechols are apt to produce eczema on human skin and hence are unsuitable.

Trihydroxybutyrophenone, a food grade antioxidant, is equal in effectiveness to propyl gallate, but it has little or no carry-through properties into baked goods (Stuckey & Gearhart—*Food Tech.* 11, 676). In a study of control of rancidity of stored fish, ascorbic acid appeared to be the best antioxidant, propylene glycol (an antioxidant solvent) accelerated deterioration, and combinations of other antioxidants with ascorbic acid were not synergistic (Boyd *et al.*—*Prog. Rept. Pac. Coast (Fishery) Station, Canada*, 108, 21). With salted herrings, a

relatively high concentration of ascorbic acid is necessary for antioxidant action, while small amounts can act as prooxidants (Marcuse—*Fette-Seifen-Anstrichmittel* 58, 1063).

A method of synthesizing the antioxidant nordihydroguaiaretic acid has been described (Sugimoto & Okumura—*Ann. Rept. G. Tanabe Ltd.* 1, 14). In a pharmacological study of butylated hydroxyanisole, the metabolic products of this antioxidant formed in the rabbit were determined (Dacre *et al.*—*Biochem. J.* 64, 777).

Some communications recorded studies on natural antioxidants. The antioxidant present in garden cress, wild mustard, and flax seed oil was identified as tocopherol (Lofty *et al.*—*J. Am. Oil Chemists' Soc.* 34, 96). This activity in sunflower seed flour is determined by the presence of chlorogenic acid, and in the flour of hemp and flax seed by the proteins (Kretovick & Rall—*Biokhim. Zernia Sbornik*, 1956, No. 3, 188). Acacatechin, a constituent of catechu, is an effective stabilizer for peanut and mustard oils (Husaini *et al.*—*J. Sci. Ind. Res., India* 16A, 128). The petroleum ether extract of dried tomato fruit is highly effective for stabilizing lard (Henze & Quackenbush—*J. Am. Oil Chemists' Soc.* 35, 1). Hydroxychavicol (4-allylpyrocatechol) isolated from betel leaf has been shown to exert an antioxidant effect on refined vegetable oils and lard (Sethi & Aggarwal—*J. Sci. Ind. Res., India*, 15B, 34). Among essential oils, phenols, etc. extracted from turmeric, pepper, garlic, onion, ginger, and cinnamon, a yellow solid obtained from onion exhibited the most antioxidant activity, but it was not of high order (*Ibid.* 16A, 181). Tea polyphenols show antioxidant activity on a weight basis of the same order as that of propyl gallate; on a molar basis they should be considerably better (Lea & Swoboda—*Chemistry & Industry*, 1957, 1073). With gossypol, its inhibition of autoxidation is attributed to the four -OH groups in 6,6'- and 7,7'-positions, and the aldehyde groups in 8,8'-position increased the antioxidant activity (Wachs—*Fette-Seifen-Anstrichmittel* 59, 318). The antioxygen effect of skimmed milk and beef serum is explained on the basis of an inactivation of prooxygen metal traces, and it is strongly pH dependent (Becker *et al.*—*Ibid.* 599). Enzyme treatment of dog-rose fruit, extraction with ethanol, and purification of the extracts yield crystalline aglucone fractions of flavone glucosides which are effective for preservation of butter during storage (Novotel'nov *et al.*—*Trudy Leningrad. Tekhnol. Kholodil. Prom.* 7, 3). Rancidification is inhibited in sealed mayonnaise by addition of glucose oxidase and catalase enzyme to remove oxygen (Bloom *et al.*—*Food Packer* 37, No. 13, 16).

Patents have been issued on use of the following stabilizers for fats and oils: alkylated hydroquinone monoglyceride ethers (Bell—*U. S.* 2,758,119), hydroxyindoles (Bell & Lappin—*U. S.* 2,787,551), benzhydrazide and its substituted derivatives (Bell & Lappin—*U. S.* 2,808,416), 2,4,5-trihydroxybutyrophenone (Bell *et al.*—*Brit.* 777,811), β -(alkylthio)-propionamides (Chenicek—*U. S.* 2,792,307), combinations of alkyl phenylenediamine and methoxy-, ethoxy- or propoxypropylamine (Chenicek—*U. S.* 2,793,944), β -(carboxymethylthio)-tricarballic acid (Evans & Schwab—*U. S.* 2,797,231), 5,6-diamino acenaphthene (Gleim—*U. S.* 2,780,550), mixtures of gallic esters, ascorbic acid, and phospholipides (for treating meats) (Hall—*U. S.* 2,772,169), amino-glycose-reductones (Hodge & Evans—*U. S.* 2,806,794), 1-phenyl-3,5-pyrazolidinediones (Lappin & Tholstrup—*U. S.* 2,801,254), substituted 1,3-dithianyl pentanoic acids (Latham *et al.*—*U. S.* 2,752,249), water extracts of liver or pancreas of mollusca (Sekine *et al.*—*Japan* 5786-'55), ω,ω' -bis(3,4-dihydroxyphenyl) alkanes (Sumiki & Tamura—*Japan* 8124-'55), pyrazolidone derivatives (Tholstrup—*U. S.* 2,808,417), 3,4,5-trialkoxyphe-nols (Thompson—*U. S.* 2,801,926), 2,4,6-trialkoxyphe-nols (Thompson—*U. S.* 2,801,927), 1,4-dihydroxy-5,8-methano-5,6,7,8-tetrahydronaphthalene (Thompson & Symon—*U. S.* 2,801,928), an indane having an -OH group in the 4-position and either an -OH or ether group in the 7-position (Thompson & Symon—*U. S.* 2,801,651), amino-1,8-methylenedioxy-naphthalene (Thompson—*U. S.* 2,771,349), and rhodamine and/or rhodamine derivatives (Willis—*U. S.* 2,800,491). Stabilized fish oils are produced if the fish are cooked in antioxidant solution in the rendering process (Matsubashi—*Japan* 3929-'56). The polyoxyethylene derivatives of fatty acid partial esters of hexitol anhydride with citric acid are effective synergists for phenolic antioxidants (Hall—*U. S.* 2,761,784). Phenolic ketimines, containing ortho -OH groups, are effective for deactivating prooxidant metals (Gleim—*U. S.* 2,789,912).

OTHER TYPES OF DETERIORATION. Acidification of stored palm oil of Belgian Congo is due to hydrolysis by dissolved

water and is catalyzed by the fatty acids; enzymatic hydrolysis is rarely observed (Loncin—*Ind. chim. belge* 20, *Spec. no.* 883). An activation factor which initiates the acidification is destroyed by heating the oil to 80° (Thuriaux—*Ibid.* 915). When palm oils were acidified through action of natural enzymes, monoglycerides were highest (12.3%) in samples containing 49% free fatty acids, and diglycerides were highest (32%) in samples containing 32.8% free acids (Desnuelle *et al.*—*Rev. franc. corps gras* 4, 203).

Irradiating fats with β - or γ -rays produces peroxides and carbonyl compounds, and results in odor and flavor changes which do not correlate well with chemical changes (Chipault *et al.*—*Ind. Eng. Chem.* 49, 1713). With oleic acid, γ -irradiation forms mixtures of 8-, 9-, 10-, and 11-hydroperoxido-oleates and C₈ and C₉ mono- and dicarboxylic acids (Slover & Dugan—*J. Am. Oil Chemists' Soc.* 34, 333). When meat is subjected to γ -radiation for sterilization, carbonyl compounds are formed which are different from those developed during irradiation of fat (Batzer—*J. Agr. & Food Chem.* 5, 700).

p-Aminobenzoic acid was mixed with crude cottonseed oil before refining to form insoluble derivatives of gossypol and related pigments (Dechary *et al.*—*U. S.* 2,787,625). Thus treated oils refine and bleach easier and are color stable. Color reversion in tallow is inhibited by addition of a small amount of hexamethylenetetramine to the tallow after it has been decolorized with propane (Sims & Nelson—*U. S.* 2,783,256).

Chlorine dioxide treatment of flour does not have a significant effect on the supply of essential fatty acids in the flour and it is thus unlikely that such treatment will result in essential fatty acid deficiency (Fisher *et al.*—*Chemistry & Industry* 1957, 1179).

Sorbic acid is efficient for inhibiting microbiological spoilage of margarine; it preserves taste; and is physiologically unobjectionable (Becker & Roeder—*Fette-Seifen-Anstrichmittel* 59, 321).

The control of garlic flavor and other flavors in butter has been discussed with regard to removal of the contributing plants with the use of herbicides (Dibbern—*Ibid.* 58, 1043).

The Reaction of Mercaptoacetic Acid with Methyl Linoleate and Linoleic Acid

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DURING THE COURSE of an investigation of the addition of various compounds to the ethylenic bonds of linoleic acid the reaction between mercaptoacetic acid and methyl linoleate was explored, and it appeared to merit more detailed study. Most of the previous work on the addition of mercaptoacetic acid to unsaturated compounds has been confined to addition of the reagent to terminally unsaturated olefins. However Koenig and Swern (6) have studied the addition of mercaptoacetic acid to undecylenic, ricinoleic, and oleic acids, and its addition to peanut oil has been reported (14). Earlier workers (2) reported that mercaptoacetic acid adds quantitatively to both double bonds of methyl linoleate although the reaction product was not isolated. In addition to the isolation and characterization of the di-adduct it appeared to be of especial interest to ascertain the possibility of selective addition of mercaptoacetic acid to methyl linoleate since the preferential addition of bromine to form 12,13-dibromo-9-octadecenoic acid (7) and of thiocyanogen to form 9,10-dithiocyano-12-octadecenoic acid (13) has been reported. Consequently the present investigation was directed toward the preparation, purification, and the characterization of the mono-adduct (carboxymethylthio-octadecenoic acid) and the location of its residual ethylenic bond as well as the characterization of the di-adduct, di(carboxymethylthio)-octadecanoic acid, and its trimethyl ester.

Experimental and Discussion

Analytical Methods

1. The progress of reactions was followed by periodic determination of unreacted mercaptoacetic acid in the reaction mixtures. Mercaptoacetic acid, alone or in reaction mixtures, was determined as follows. An accurately weighed sample of sufficient size to

contain 0.7 to 1.0 milliequivalent of mercaptoacetic acid was dissolved in 5 ml. of carbon tetrachloride, 10 ml. of distilled water were added, and the sample was titrated to a permanent yellow color with a standard 0.1 N solution of iodine in glacial acetic acid and back-titrated with standard 0.1 N sodium thiosulfate until the yellow color just disappeared. In the analysis of reaction mixtures containing a large excess of mercaptoacetic acid, determinations were carried out on a scale five times as large as that described above.

2. Chromatographic separation of adducts was utilized for determination of the approximate amounts of the various components in crude and purified reaction products, and on a larger scale for preparative purposes. A modification of the Ramsey-Patterson procedure (10) for the separation of short chain monocarboxylic acids was employed for analyzing addition products of methyl linoleate and mercaptoacetic acid. The column was prepared as described by Ramsey and Patterson, but the neutralization step was omitted. Since the products were not completely soluble in iso-octane (the mobile phase), samples were added to the column in iso-octane containing about 15% of ether. Methyl linoleate moved rapidly through the column and was eluted principally at an effluent volume of 10 to 20 ml. The threshold volume (the volume at which the band reached the bottom of the column) of the half-ester of the mono-adduct generally amounted to about 40 ml. The monomethyl ester of the di-adduct was not eluted by iso-octane. The amount of mono-adduct was determined directly by titration of appropriate fractions of the eluate. The acid which was not eluted from the column by iso-octane was assumed to be the di-adduct. The amount of unreacted methyl linoleate was determined by subtracting the total calculated weights of mono- and di-adducts from the sample weight. Where the threshold volume of the mono-adduct was large enough to permit good separation from methyl linoleate, the weights

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