

Annual Review of Literature on Fats, Oils, and Soaps. I.

REPORT OF THE LITERATURE REVIEW COMMITTEE *

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Introduction

Literature that became available during 1953 is noncritically reviewed as briefly as possible.

STATISTICS AND OUTLOOK. The latest available United States production data (*Fats & Oils Situation 164*, 20 pp.) for fats and oils are as follows:

	For years beginning		
	Oct. 1 1951	Oct. 1 1952 prelimi- nary	Oct. 1 1953 estimate
	<i>mil. lbs.</i>	<i>mil. lbs.</i>	<i>mil. lbs.</i>
Butter (actual weight).....	1,389	1,621	1,625
Other edible animal fats.....	3,093	2,761	2,450
Edible vegetable oils (includes oil equivalent of seed exported).....	4,686	5,002	4,978
Inedible fats (soap fats & oils).....	2,394	2,753	2,675
Drying oils.....	681	556	700
Neat's-foot oil, wool grease, fish liver oils, etc.....	28	35	25
Total.....	12,222	12,729	12,453

There will likely be a decline of three percent in the crop year beginning October 1, due for the most part to a drop in lard production. Total fat supplies, however, will be at a peak, since stocks at the beginning of the crop year were at a new high. About 70% of the stocks were owned by the government as a result of the price, support program.

The support program has kept prices fairly steady on most of the major fats. The price of unsupported lard had gained from an early low because of short supply but a downward adjustment is predicted for the present year. Large supplies of inedible tallow and greases and lack of price support will tend to keep prices of these relatively low.

In 1953 soap sales decreased about 11% compared with a six percent decrease in 1952; synthetic detergent sales increased about 24% compared with 20% in 1952. Synthetic detergent sales are now 53% of the total United States detergent market (Snell—*Chem. & Eng. News* 32, 36).

High production plus high stocks, lack of government price support, and the trend toward nonfat derived synthetic detergents have created economic problems in the animal fat business. In efforts to ease the situation Ewell (*J. Agr. & Food Chem.* 1, 552) reviews the possible technical outlets, Swern *et al.* (*U. S. Dept. Agr. Bur. Agr. & Ind. Chem. Mimeo. Circ. Series AIC346*, 58 pp.) indicate research possibilities to improve large scale use for animal fat surplus, Sills & Doty, Jr. (*U. S. Dept. Agr. Prod. & Marketing Admins., Market Res. Rept. No. 46*, 31 pp.) discuss the possibility of an increase in their use in detergents, emulsifiers, and emulsion products, and Schweigert *et al.* (*Am. Meat Inst. Foundation, Circ. 7*, 3; *Bull. 15*, 3) demonstrated that they can be added to dog and poultry feeds. The problem has also been the subject of considerable editorial comment (*J. Agr. & Food Chem.* 1, 1029, 1145; *Chem. Eng.* 60, No. 3, 198; *Chem. Eng. News* 31, 555), and of a lecture by the Director of the Production & Marketing Administration of the U. S. Department of Agriculture (Prichard—*Oil Paint & Drug Repr.* 163, No. 19, 76). European markets for surplus United States fats and oils have also been investigated (Quintus—*U. S. Dept. Agr., Foreign Agr. Rept. No. 75*, 29).

An annual review for 1952 on oilseeds, oils, oilcakes and related commodities contains information on world production, consumption, interchange, export balances, competition, etc., according to geographical and national areas (*Frank Fehr & Co., Liverpool, Eng.*, 95 pp.). Other published reports on world supply and consumption of fats have also been published for the year 1952 or earlier (Duncan—*J. Am. Oil Chemists' Soc.* 30, 358; *Food & Agr. Organization of U. N. Commodity Rept. No. 4*). Another paper reviews geographical and climatic influences on vegetable oils, acreage planted to oil crop 1878-1952, protein produced, productivity of various crops in calories, and composition of commercial oils (von Boguslawski—*Fette u. Seifen* 54, 737).

General articles which contain some economic information are written on: vegetable oils and waxes of El Salvador, Honduras, Venezuela, and Brazil (Markley—*Inst. Interam. Affairs, Activity Series No. 4*, 18 pp.); fat industry in Hesse and Southwestern Germany (Anon.—*Fette u. Seifen* 55, 725); some less known oil seeds of the Belgian Congo (Tihon—*Bull. agr. Congo Belge* 43, 978); the fat industry in India (Aggarwal—*Bull. mens. inform. ITERG* 7, 321); fat in the meat industry (Grau—*Fette u. Seifen* 55, 25; Liberman—*Myasnaya Ind. S.S.S.R.* 23, No. 6, 36); sea animal oil of Hokkaido (Ueno—*Fette u. Seifen* 55, 685); sources and properties of fish fats (Lovern—*Ibid.* 425; source and vitamin A content of various fish oils of Russia (Symposium, most work directed by Bukin—*Akad. Nauk S.S.S.R. Inst. Biokhm. im. A.N. Bakha Vitaminye Res. i Ispol'zovanie Sbornik* 1, 7, 71, 113, 139, 158, 182, 216, 222, 250, 278; coconut fat (Markley—*Ministerw. Agr. y g Anaderia Serv. Tec. Interam. Cooperacion Agr. Bol.* 114, 36 pp.); fatty acids and liquid fuel from cottonseed oil foots (Bhushan—*J. Sci. Ind. Res., India*, 12B, 38); dende palm of Brazil (Cardoso—*Rev. brasil. quim.* 33, 232); Gokhru seed oil (Shrivastava—*Proc. Sym. Indian Oils, Fats Nat'l Chem. Lab.* 1951, 29); grape seed oil (Huesa—*Grasas y aceites* 3, 103); herring oil (Ludorff—*Fette u. Seifen* 55, 454); mowrah fat (Kane—*Proc. Sym. Indian Oils Fats Nat'l Chem. Lab.* 1951, 55); neem oil (Mitra—*Ibid.* 40); oiticica oil (Francois—*Oleagineux* 7, 557); body fat of *Prochilotus lineatus* (Brenner—*Anales asoc. quim. argentina* 41, 61); *Raphanus sativus* seed oil (Duperon & Sosa—*Bull. Soc. chim. biol.* 35, 257); literature on utilization of rice bran oil 1876-51 (Loeb

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& Morris—*U. S. Dept. Agr. Bur. Agr. & Ind. Chem. Southern Regional Lab. 1951*, 95 pp.); wild sesame seed oil of North Rhodesia (Pearman *et al.*—*Colonial Plant & Animal Products, London 2*, 297); tung oil industry in Paraguay (*Ministerio Agr. y Ganaderia Bull. 120*); tung oil review 1951-52 (Planck—*J. Am. Oil Chemists' Soc. 30*, 587); and ucuhiba butter (Pinto—*Bol. tec. inst. agron. norte, Brazil, No. 23*, 7). The articles of general information on fat technology concerned: chemical engineering in the fat and oil industry (Lawrence—*Chem. Eng. Progress 49*, 627); solvent extraction of oils in India (Parekh—*Chem. Age, India, Ser. 6*, 139); use of cooling equipment in the fat industry (Heinze—*Fette u. Seifen 55*, 44); refining, heating, and cooling oils (Wilke—*Fette u. Seifen 55*, 801); the technical publication and patents of the Southern Regional Research Laboratory (Singleton—*U.S.D.A., AIC361*, 33 pp.); and recent advances in processing of lard (Ault & Riemenschneider—*Food Eng. 25*, No. 6, 99), of new plastic fats (Feuge—*Ibid.* 100), and of soybean oil (Cowan—*Ibid.* 103).

MICROBIOLOGICAL, PETROLEUM, AND WASTE AS SOURCES FOR FATS. Experiments are still being conducted to develop microbiological sources for fats. Ikeda (*J. Fermentation Technol., Japan, 28*, 20, 69) selected *Aspergillus candidus* as the best fat producer from potato starch and sugar media, and has ascertained suitable conditions for the culture process. An *Endomyces* organism isolated from hemp retting solution readily converts glucose, fructose, galactose, mannose, lactic acid, malic acid, alcohol, succinic acid, and glycerol to fat (Nakahama—*Ibid.* 29, 331; 30, 11). Work with *Rhodotorula gracilis* shows that it can produce 27.4 grams of fat per 100 grams of sugar consumed (Nokay—*Türk İjten Tecrübi Biol. Dergisi 11*, 344); it assimilates glucose more rapidly than xylose but may be adapted to assimilate both at the same rate (Kleinzeller & Malek—*Chem. Listy 46*, 674); and the fats are more saturated and of lower molecular weight when formed at high temperatures (Bass & Hospodka—*Ibid.* 243). Various strains of *Rhodotorula* consume only small amounts of sugar from koji extracts and produce small amounts of fat (Shibasaki—*J. Fermentation Technol., Japan, 30*, 65). Fat formation by *Fusarium lini* is better from glucose than from acetate but certain combinations of these in the media induce production of fat of high unsaturation (Nord *et al.*—*Arch. Biochem. & Biophys. 38*, 377, 385; 39, 219, 406; 40, 102; 45, 183). Presence of naphthoquinone also increases formation of unsaturated fats, i.e., olein and linolein. The effects on synthesis of fats by yeasts of aeration, pH, strains, and many alcohols, phenols, esters, aldehydes, aromatic hydrocarbons, ethers, volatile oils, chlorinated hydrocarbons, and some compounds occurring in plants have been determined (Kunze—*Mitt. Versuchsanstalt Gärungsgewerbe u. Insts. Angew. Mikrobiol. Hochschule Bodenkult. 4*, 44). The mold, *Penicillium chrysogenum* contains 2.67% fat (Abe—*Proc. Fac. Eng. Keiojyuku Univ. 2*, No. 7, 15).

The new information on production of fatty acids or fats from petroleum are: on products formed on oxidation in the presence of sulfuric acid (Pardum—*Erdöl u. Kohle 6*, 10); a review (Anders—*Industria vernici, Milan, 7*, 12); mechanism of the reactions that occur (Langenbeck & Pritzkow—*Fette u. Seifen 55*, 435, 506; Anders—*Seifen-Öle-Fette-Wachse 78*, 48); use of potassium permanganate in the process (Rabinovich—*Maslobotno Zhirovaya Prom. 18*, No. 1, 16); effect of temperature (Tsykovskii—*Ibid.*, No. 6, 20); catalyst for the process (Riethof & Brown, Jr.—*U. S. 2,636,047*); and separation of fatty acids from the oxidized mixture (Dickinson—*U. S. 2,660,601*; van der Waals—*Dutch 69,921*). Well made synthetic fats are as acceptable to poultry as natural fats (Enders—*Arch. exptl. Path. u. Pharmacol. 214*, 404). Synthetic fats contain some branched chain acids which are more toxic, as measured intraperitoneally, than acids of natural fats (Hauschild—*Arzneimittel-Forsch 3*, 86). Products that can act as substitutes for drying oils are also prepared from petroleum by condensation and polymerization reactions (Geiger—*U. S. 2,596,235*; Lee & May—*U. S. 2,614,132*; Pritzker—*U. S. 2,627,479*; Hoffman—*U. S. 2,645,649*; Block & Hoffmann—*U. S. 2,644,847*; Linn—*U. S. 2,631,160*).

Fatty and other organic material suitable for preparation of waxes, varnishes, and soap can be extracted from cork (Guillemonat—*U. S. 2,617,814*; Delaville & Delaville—*Fr. 876,891*). A catch basin for recovery of fats and oils from waste water by flotation with air bubbles is illustrated and described (Wittmann—*Pharm. Ind. 14*, 352). A patented process of this type for fatty acid recovery includes conversion to insoluble soaps before the air-bubble flotation (Gibbs—*U. S. 2,637,737*). The economy of fat recovery from meat packing house waste waters is discussed (Safonov—*Myasnaya Ind. S.S.S.R. 23*, No. 6, 44).

NEW COMPREHENSIVE LITERATURE. The previous review of this series is published in *J. Am. Oil Chemists' Soc. 30*, 197, 228. A review of the same scope by the Oils and Fats Group of the Society of Chemical Industry (*Repts. Ann. Prog. Applied Chem. 37*, 607) is selective and critical of the progress in the field. Hilditch (*Ann. Rev. Biochem. 22*, 125) prepared a review on the chemistry of lipides. A book on "Progress in the Chemistry of Fats and Other Lipides, Volume I, edited by Holman *et al.* contains information on: molecular structure and polymorphism of fatty acids and their derivatives by Malkin, on sterols by Bergman, on structure and properties of phosphatides by Desnuelle, on chromatography of fatty acids by Holman, and on derivatives of the fatty acids by Harwood.

Other new books of the fat and oil field are:

Practical Butter Making—7th ed. By H. G. Wilster. O.S.C. Cooperative Assoc., Corvallis, Oregon. 497 pp.

Soybean Industry. By R. A. Goldberg. University Minnesota Press, Minneapolis. 186 pp.

Estrazione con solventi e raffinazione degli oli vegetali. By U. Morini. G. Lavagnolo, Torino, Italy. 176 pp.

Refining of Oils and Fats for Edible Purposes. By A. J. C. Anderson. Academic Press, New York. 204 pp.

Die Untersuchung der Fette und Öle. 4th ed. By F. Puschel & A. Dierichs. W. Knapp, Halle, Ger. 322 pp.

Detergent Evaluation and Testing. By J. C. Harris. 208 pp.

Les plantes a matiere grasse. T. IV. Le ricin. By J. Adam. Soc. ed. geographiques, maritimes et coloniales, Paris, Fr. 127 pp.

Possibilidades de los aceites espanoles de animales marinos. By R. Montequi Dias-Plaza. Consejo Superior Invest., Madrid, Spain. 152 pp.

Trattato teorico-pratico del saponiere profumerie moderno. 4th ed. By G. Coatti. U. Hoepli, Milan, Italy. 953 pp.

Moderne Wasch- und Reinigungsmittel. By H. Mannek. M. & S. Moser, Garmisch-Partenkirchen, Ger. 28 pp.

Die Herstellung der Schmierseifen und flüssigen Seifen. By H. Ziilske. Verlag Chem. Ind. H. Ziolkowsky, Aushurg, Ger. 217 pp.

Soap Manufacture. Vol. I. By J. Davidsohn *et al.* Interscience Pubs., New York. 525 pp.

Seifenfabrikation. 3rd ed. Edited by T. Klug. Walter de Gruyter & Co., Berlin, Ger. 116 pp.

Soap Maker's Directory. 55th ed. Newham, Cowell and Gripper, London, Eng. 70 pp.

Seifen-Industrie Kalender. By H. Heller, Delius Klasing & Co., Berlin, Ger. 272 pp.

A series of lectures presented as a short course on engineering aspects of processing of edible oils appeared in a journal (*J. Am. Oil Chemists' Soc. 30*, 473) and is also sold as a separate publication (American Oil Chemists' Society, Chicago, Ill. 105 pp.).

A survey of institutions which offer training in fat and oil technology in the United States (Kummerow—*J. Am. Oil Chemists' Soc. 30*, 313), and a treatise on the functions of "The Instituto de Oleos in Rio de Janeiro" (Martinenghi—*Olearia 6*, 61) are of general interest to fatty oil chemists.

Production Processes

ANIMAL AND FISH FAT EXTRACTION. The cold degreasing of bones and other fat-containing materials by subjection to mechanical impulses transmitted through water was the center of attention for fat rendering as evident from several editorials, news notes, and technical journal items (*See 1952 review; Chem. Eng. 60*, No. 1, 102; No. 2, 118; *Ind. Eng. Chem. 45*, No. 3, 11A; *Soap Perfumery & Cosmetics 25*, 1263; Chayen & Ashworth—*J. Applied Chem. 3*, 529), and a U. S. patent (Chayen—*U. S. 2,635,104*) was issued on the process. The low temperature high speed rendering method of the Kingan firm, also cited last year, was the subject of a new article (Dayen *et al.*—*Food Tech. 7*, 421). Khar'kov (*Rybnoe Khoz. 1952*, No. 9, 21) described the advantages and technique for application of dry rendering in the fish industry.

A Norwegian technical research committee (*Norsk Hvalfanst Tid. 41*, 289) has reported experimental data on processing whales. They designed processes utilizing conical centrifuges which have greater capacity so as to approach the efficiency of separation of fat, grax, and low-sludge glue water by the cylindrical centrifuge processes. A new process designed for fish, but applicable to other fatty materials, comprises electrolyzing ground material in salt solution, filtering, and centrifuging out the fat (Bruzac—*Fr. 885,512*). In two new

processes for converting fish waste and animal carcasses to fertilizer and grease, digestions by acetic acid (Alimentation Equilibree—*Fr.* 979,036) and hydrochloric acid (Salonaise Chem. Products Co.—*Fr.* 881,923) are used to release the fat.

Fish liver processing for vitamin A was investigated by Murata & Suzuki (*Bull. Fac. Fisheries Hokkaido Univ.* 2, 262, 266, 273). In general the potency of the vitamin extract increases and the oil yield decreases when the pH is increased to the range of 11.4-13.4 in the predigestion stage. With flounder liver, digestion at pH 11-12 induces high vitamin A potency in the extract, whereas with codfish liver a pH of 8.0 during digestion is preferred. A comparison of five methods of production of vitamin A concentrates from fish livers by Martin-Perez (*Ion* 13, 61) indicated that digestion of livers with 30% by weight of solid potassium hydroxide, addition of two parts of flour, and extraction with trichloroethylene at 40° gave the highest potency product with 99-100% recovery. An oil-solvent extraction comprises adding two parts of edible oil per each part of liver, vacuum drying, and separating dehydrated solids from the oil (Greenfield—*U. S.* 2,651,647).

Several articles on production of butter and butter oil seem pertinent to this review. An investigation on churning time of milk and cream at various temperatures indicates that there is an optimum relationship between liquid and solid fat composition for churning and that the increase of either extends churning time (Holland & Herrington—*J. Dairy Sci.* 36, 850). From similar work Grishchenko (*Molochnay Prom.* 14, No. 4, 28) converted his data to equations that give the relationship between temperature and churning time. "Vacreator" treatment of cream is alleged to improve the texture of the butter (Dolby—*J. Dairy Res.* 20, 201). Crystallization of butter under super atmospheric pressure improves the texture (Horneman *et al.*—*U. S.* 2,630,338). A paraffin-like body, the so-called gummy butter, results when dairy cows are given rations containing hydraulic- or solvent-extracted cottonseed meals (Moore & Leighton—*Texas Agr. Expt. Sta. Bull.* No. 752, 11 pp.).

Methods of preparation and descriptions were published on two Russian butters, Vologda (Zheltakov & Shersheva—*Molochnaya Prom.* 14, No. 5, 12) and Meleshin method butter (Titov—*Ibid.* No. 4, 13). A patented Russian butter-making process comprises preparing rich cream (70% fat), adding butterfat at 35°, and centrifuging the mixture at 26° (Zheltakov—*U.S.S.R.* 77,182). Blok & Geimberg (*Molochnaya Prom.* 14, No. 6, 17) retard development of tallowy and fishy flavors in butter by adding yeast to the cream prior to churning.

For continuous butter processes, there was one patent (Wilsman—*U. S.* 2,649,377) which demonstrates that it is advantageous to add the salt, color, etc., at a stage when the major portion of the buttermilk is already separated from the "butter corn." A dissertation on the continuous process stressed proper ripening of the cream (Babich—*Molochnaya Prom.* 14, No. 7, 39).

Butter oil made by de-emulsifying cream with surface active agents is practically identical to conventionally made butter oil, except that it contains less lecithin (Stine & Patton—*J. Dairy Sci.* 36, 516). Two butter oil making processes were patented. Bettman (*U. S.* 2,612,454) gradually heats butter while adding relatively small amounts of salts and cools to stratify the butter oil so it may be separated. Lundal & Robichaux (*U. S.* 2,626,217) melts the butter, adds water, neutralizes the acidity, re-emulsifies the fat, and pasteurizes so that on cooling it stratifies.

VEGETABLE OIL EXTRACTION. McKinney *et al.* (*J. Am. Oil Chemists' Soc.* 30, 83) demonstrated that the economy of grove as compared to mill hulling of tung fruit are realizable without loss in quality or quantity of the tung oil. Hulling is best at 15-20% moisture but the kernel moisture should be quickly reduced to less than 10% for storage (Holmes *et al.*—*Ibid.* 137). Cutting (*J. Sci. Food & Agr.* 3, 505) shows that optimum moisture content for oil expelling from tung meats containing 16% shell is 4.5%.

The new high oil content varieties of Russian sunflower seeds are less stable in storage than the common varieties which they have replaced (Astrelin—*Masloboino Zhirovaya Prom.* 18, No. 6, 9). Rice bran, which is very susceptible to development of fatty acidity, is stabilized for storage by heating to 100-150° (Tsujino—*Japan* 5,935[51]). When olive oil pressing residues are stored before extraction of residual oil, there occurs a loss of oil content, and considerable increases in free fatty and oxy acids. These changes during storage, hermetically sealed, in ordinary heaps, or in pressed heaps over several months, were plotted by Montefredine (*Fette u. Seifen* 55, 577).

Several fundamental studies pertaining to the seed treatment and its effect upon the nutritive value of the oil meals. Enzyme inhibitors of soybean products which reduce the nutri-

tive value of the protein are said to be destroyed by autoclaving with 15 lb. pressure of steam for 15 minutes (Viswanatha & De—*Indian J. Physiol. & Allied Sci.* 6, 95). Other workers (Balloun *et al.*—*Poultry Sci.* 32, 517), however, find that such treatment of soybean meal for 20 minutes also sharply decreases the value of the proteins as measured by water solubility. The deleterious effect of over-heating is partially avoided by addition of water prior to heating (Renner *et al.*—*Ibid.* 582). This same group (*Ibid.* 492; *J. Nutr.* 50, 487) found less lysine, arginine, and tryptophan liberated on acid hydrolysis from sunflowerseed oil meal cooked at 240°F. and conditioned at 260°F. than that handled at 200-220°F. and 220-240°F., respectively; similar improvement in nutritive quality of expeller sunflower meals is obtained by opening the "choke" to reduce the heat developed on pressing. The effect of heat treatment in processing of cottonseed on the biological protein value of the proteins was determined in tests on rats (Sure *et al.*—*J. Agr. & Food Chem.* 1, 82).

Statistical data from studies on feeding peanut meals to dairy cows were interpreted to indicate that solvent extracted products are more digestible and their proteins are of greater biological value than those of pressed cakes (Letard *et al.*—*Oleagineux* 7, 495). Similar work with calves evaluated expeller and hydraulic meals, of 0.059 and 0.173% free gossypol content, with regard to toxicity (Hollon *et al.*—*J. Animal Sci.* 11, 765). Good cooking procedures in hydraulic pressing of cottonseed can yield products as low in free gossypol as those of the expeller process (Pons Jr. *et al.*—*J. Am. Oil Chemists' Soc.* 30, 128). This same report indicates how either under or overheating may be injurious to the biological value of the meal proteins. Reuther *et al.* (*Ibid.* 28) produced a hydraulic cottonseed meal of very low gossypol content with little effect on protein quality by flaking the meats at 14% moisture and cooking at less than 225°F. Tung meal can be rendered less toxic by heat and by extraction with organic solvents, but the value of the meal as a feed does not justify the expense (Lee & Watson Jr.—*J. Am. Oil Chemists' Soc.* 30, 32).

A new seed hulling machine designed for castor beans has rubber hulling surfaces of different hardness so that other seeds and nuts can be processed (Hansen—*U. S.* 2,662,570).

Fines in the ultimate extracted meals are reduced by flaking the seeds at 12-20% moisture and at 180-275°F. before extraction; and further improvement is effected by adding phosphatides or carboxymethylcellulose (Ayers & Scott—*U. S.* 2,645,650). Free gossypol content of cottonseed meal is found to be directly related to thinness of the flakes extracted and inversely to the moisture content during flaking (Luedtke & Wamble—*Texas Eng. Expt. Sta. Rept. No.* 39, 7 pp.). High oil yields with good reduction of free gossypol in meals from cottonseed meats occurs on precooking for 7-20 minutes, at 190-215°F. and at 12-20% moisture content (Dunning—*U. S.* 2,629,722). Improvements on the continuous seed cooker having several superposed cooking chambers through which material is dropped in series are patented (Greenbank—*U. S.* 2,653,084).

Test with 56 surface active compounds during pressing indicated that they do not improve the rate of oil extraction (Debruyne—*Bull. mens. inform. ITERG* 7, 56). The addition of 1.5-3.5% salt to crushed ripe olive pulp improves oil yields on pressing (Garoglio—*Olearia* 5, 341). Better maceration of the pulp is also recommended for the same purpose (Castorina—*Olivicoltura* 7, No. 12, 3).

General information on oil pressing is directed to an expeller mill of 50 tons of cottonseed per day capacity (Dunning *et al.*—*Oil Mill Gaz.* 50, No. 3, 13); a 10,000 kg. per day mill (Reutenberger—*Seifen-Öle-Fette-Wachse* 79, 125, 157); and a combination single pressing-solvent continuous cottonseed oil mill (Minasyan & Plyushkina—*Masloboino Zhirovaya Prom.* 18, No. 3, 7). A study of the relationship between the volume of cottonseed meats fed to a press and the volume of resulting oil cake is recorded to point out sources of errors that occur in commercial calculation of processing yields (Goldovskii & Matsuk—*Ibid.* No. 6, 10).

Some simple uncommon oil extraction processes are described. Oil from Palm fruit is obtained by macerating the fruit in a closed mixer at 88°, heating on a perforated plate in an autoclave, and washing with hot water (Inst. recherches huiles palme et oleagineux—*Brit.* 691,266). The oil is skimmed from the drippings and wash water. A decoction process for castor seeds comprises grinding, cooking in brine solution, stirring with lime, then potassium nitrate, settling, and decanting the oil (Verma—*Proc. Sym. Indian Oils Fats Nat'l Chem. Lab. Poona* 1951, 130, 132; Council Sci. & Ind. Res.—*Indian* 45,118). In a similar process for peanuts the lime

treatment is replaced by a digestion with sulfuric acid (Council Sci. & Ind. Res.—*Indian 45,117*). A process similar to that of Chayen for degreasing bones is applied to oil seeds in general (Onchi—*Japan 1929*[52]). A 300-year-old process for sesame seed comprising grinding in water and stirring until oil is displaced by water is described by Lou *et al.* (*Sci. Record China 3, 203*). Use of biochemical action in oil production is evident in a process utilizing proteolytic enzymes to release oil from coconut meats (Anon.—*Food Eng. 24, No. 10, 143*), and in a method for bran mash in which fatty acids are released by natural decomposition (Inaba—*Japan 39*[51]).

Considerable fundamental data useful for designing commercial solvent extraction processes are recorded. Using trichloroethylene as the extraction solvent there has been determined: the boiling point-vapor pressure-composition relations for the cottonseed oil miscellas (Arnold & Liu—*J. Am. Oil Chemists' Soc. 30, 249*), the effect of extraction temperatures from 100 to 240°F. on the color and bleaching characteristics of the cottonseed oil produced (Liu—Iowa State Coll. *J. Sci. 27, 212*), the effect of solvent-feed ratio and temperature on extraction time for soybean flake (Arnold & P'Pool—*J. Am. Oil Chemists' Soc. 30, 611*), and the miscella density-concentration data for extraction of wheat germs, milkweed seeds, and cottonseed (Arnold *et al.*—*J. Am. Oil Chemists' Soc. 30, 393*). Data for specific gravity-concentration curves of chlorinated hydrocarbon-soybean oil miscella show that the extraction rates by the various solvents at 250°F. increased in the order of: 1,2,3-trichloropropane, 1,1,1-trichloroethane, 1,2-dichloroethane, 1,2-dichloropropane and trichloroethylene (Arnold & Carvey—*J. Am. Oil Chemists' Soc. 30, 81*). Comparison on grape, cottonseed, and olive pulp shows that trichloroethylene is superior to carbon disulfide as an oil extraction solvent (Fontana—*Ion 13, 574*). In a comparison of trichloroethylene and hexane under practical plant operating conditions the differences in extraction rates are insignificant, but the rate with the former solvent is affected less by moisture in the system (Arnold & Patel—*J. Am. Oil Chemists' Soc. 30, 216*). The use of difluoro-dichloro-methane as an oil extraction solvent was patented (Kageyama *et al.*—*Japan 7039*[51]).

Solvent seed oil processors who prepare by-product feeds are reminded of the hazard that may be involved in using chlorinated solvents. In a comparison between trichloroethylene and hexane soybean meals, 32 of 41 animals fed rations containing the former died with the following symptoms: morbidity, anorexia, elevated body temperature, bloody nasal discharge and feces, and hematological leucopenia and agranulocytoses (Pickens, Jr. *et al.*—*J. Animal Sci. 11, 799*). Administrations of various antibiotics, vitamins, or other nutrients were neither prophylactic nor therapeutic. No abnormalities were apparent in any of the animals fed hexane meal.

The density-composition-temperature data for refined rice bran oil in commercial hexane at five per cent concentration intervals and 10°F. intervals are tabulated (Magne *et al.*—*J. Am. Chemists' Soc. 30, 8*). The data are useful in commercial processing for design and control of operations. In a patent on rice polishing oil, extraction is with warm benzene and the miscella is left standing overnight to precipitate the wax (Kitsuta—*Japan 5339*[51]). Wax and oil are separate products of the process. In the extraction of various husks with ligroine the presence of moisture initially retards, but ultimately greater amounts of the oils are extracted (Rossetti—*Olii minerali, grassi e saponi, colori e vernici 30, 1*).

Another patent has been issued for use of alcohols, acetone-water mixtures, and the like solvents in a continuous system in which extraction and oil separation from miscella are based on great differences in solvency between high and low temperatures, respectively (Westergaard—*U. S. 2,653,957*). Magne & Skau's (*J. Am. Chemists' Soc. 30, 288*) basic phase relationship data for such a process shows isopropyl alcohol is the more desirable solvent since complete miscibility with oil can be attained at temperatures below its normal boiling point even at moisture contents as high as 10% by weight; whereas ethyl alcohol tolerated only about 1.5% of moisture. According to Wen (*Chinese Chem. Ind. Eng. 1, No. 1, 2*) optimum alcohol extraction of soybeans is at 95% alcohol solvent concentration, and moisture content of beans not greater than one percent. Alcohol is shown to be a good extractant for both oil and carotene from palm fibers, and the carotene thus extracted has good stability (Mellier—*Oleagineux 8, 371*). Extraction of oil from oil cakes with alcohols yields meal or flour of a quality suitable for human consumption (Rao—*J. Sci. & Ind. Res. India 11A, 414*). Rice bran oil extracted with methyl alcohol darkens on heating in the presence of iron (Kawai *et al.*—*J. Chem. Soc. Japan Ind. Chem. Sec. 55, 125*).

Extraction of linseed oil with ethyl alcohol, isopropyl alcohol, hexane or heptane, petroleum ether, and hexachloroethane yielded residues containing the following respective amounts of hydrocyanic acid: 0.022, 0.023, 0.025, 0.024 and 0.027% (Khangy—*Bull. mens. inform. ITERG 7, 112*). Treatment of solvent-extracted cottonseed meal containing 10% moisture with sulfur dioxide at 260-350°F. reduces the toxic gossypol content (Bonotto—*U. S. 2,631,099*).

New equipment has been designed for solvent extraction. The principal unit of a filtration-extraction process is a continuous horizontal, rotary vacuum filter (D'Aquin *et al.*—*Ind. Eng. Chem. 45, 247*; Graci *et al.*—*J. Am. Oil Chemists' Soc. 30, 139*). Equipment recommended by Strusi & DeDonno (*Ann. sper. agar, Rome, 7, 241*) for olive oil production is a Sculco pressure apparatus for the whole pulp and a solvent extractor for the pressing residues. The merits and advantages of solvent versus prepressing followed by solvent extraction are discussed by Hutchins (*J. Am. Oil Chemists' Soc. 30, 56*). Descriptive solvent process publications are written on the DeSmet process (van der Voort—*Fette u. Seifen 55, 361*) on the Bollmann, Hildebrandt, Craun, Allis-Chalmers, Kennedy, DeSmet, and Chyoda processes (Tamaoki—*J. Oil Chemists' Soc. Japan 1, 111*), and on solvent extraction in general (Co-field, Jr.—*Seifen-Öle-Fette-Wachse 78, 479*). Continuous extraction equipment using hydrocarbon solvents has been designed to handle powdered dried raisin seeds (Reynaud—*Fr. 801,607*). Several patents on solvent extraction processes came to the writer's attention but details were not available at the time of writing (Metallgesellschaft A.G.—*Brit. 680,728*; Arnaud—*Ger. 317,336*; Takahashi—*Japan 1230*[51]; Miyamoto—*Japan 2323*[51]; Yamazaki—*Japan 2422*[51]; Tamaoki—*Japan 3983*[51]; Maki—*Japan 5628*[51]).

Some new developments dealt with limited phases of solvent extraction equipment. Solvent prewetting of flaked meats before loading a basket type continuous solvent extractor inhibit the packing and poor solvent circulation that occur when dry packed baskets are sprayed with solvent (Richard—*U. S. 2,626,265*). A unique apparatus for continuous filtering of miscella, forces it to rise through a frustum-shaped body having cloth shaking filters (Kichigin—*Masloboino-Zhirovaya Prom. 13, No. 6, 26*). Handling equipment described by Spinov (*Ibid.*, No. 5, 7) comprises a drum filter from which filtered miscella is conducted to an Ershov evaporator and filter sludge is returned to the extraction apparatus. The use and advantages of a continuous Kestner type rising-film evaporator for concentrating vegetable oils from ligroine (boiling point 26.5-62.0) is described by Zancani (*Chimica e ind., Milan 34, 454*); and a flash evaporator for recovery of benzene from oils is described by Yoshizaki & Sakamoto (*J. Chem. Soc. Japan Ind. Chem. Sec. 54, 458*). A system of solvent recovery in which the propane solvent is heated by compression, and the hot gases are used to provide heat for stripping the miscella of solvent was patented (Dickinson—*U. S. 2,616,912*). Two new desolventizers for soybean flakes contain novel means of removing fines and returning them to the solids while the vapors are condensed and recirculated (Coats—*U. S. 2,663,089*; Leslie—*U. S. 2,618,560*).

PRODUCTION OF WOOL AND TALL OILS. A new method of scouring wool and recovery of the wool grease is based on extraction with aqueous solutions containing polar organic compounds, as lower alcohols and ketones, an electrolyte, and an emulsifier (Lundgren *et al.*—*U. S. 2,629,723, 2,655,423*). Mechanical equipment for defatting wool with organic solvents is patented (L'Abate—*Ital. 466,608*). Boyle (*Soap Sanit. Chemicals 29, No. 7, 36*) reviewed commercial methods for scouring raw wool.

Fatty acids are derived from tall oil by various methods. Bergström & Trobeck (*Swed. 137,679*) heat the tall oil with aldehydes which combine with the phenolic acids present and the fatty acids are distilled. Mayer *et al.* (*Ital. 467,994*) added methanol, sulfuric acid, and benzene, heated, added water, separated the upper layer, and recovered the fatty acids by distillation. A process patented by Glynn & Hofferth (*U. S. 2,662,849*) comprises removal of short chain acids by distillation and completing the separation of fatty acids from the still residue by crystallization. Gloyer & Vogel (*U. S. 2,640,823*) selectively esterify the fatty acids of the tall oil with a lower alcohol, extract with polar solvents to leave a raffinate of fatty acids which is purified by distillation. Two reviews on tall oil are about commercial fractionation processes (Wheeler *et al.*—*Am. Paint J. 37, No. 24, 62*; Linder—*Acta Polytech. Chem. Met. Ser. 2, No. 10, 56 pp.*).

REFINING, BLEACHING, AND DEODORIZATION. Clarifying, degumming or desliming of oils are performed as a means of

lowering subsequent refining losses, as a step in production of phosphatides, or simply to produce a clear oil where reacidifying refining is unnecessary. Decoction and expeller process oils are purified by agitating with dilute brine, allowing them to stand so as to stratify into an aqueous residue layer, an emulsion layer containing the phospholipides, and an oil layer (Skipin & Polstyanoi—*Masloboino-Zhirovaya Prom.* 18, No. 6, 27; Aleksandrov *et al.*—*Ibid.* No. 5, 24). Several methods of simple purification are called "emulsion washing." For two such methods, the aqueous washing agents are dilute inorganic acid solutions (Yamakita *et al.*—*Bull. Inst. Chem. Res. Kyoto Univ.* 24, 82; Dini—*Olearia* 6, 293). One method for cottonseed oil which included preparation of gossypol as a by-product, makes use of dilute alkali solution at moderate temperatures for emulsification and the purified oil is obtained on demulsification (Sergeev & Sterlin—*Masloboino Zhirovaya Prom.* 18, No. 4, 6). With rice oil where a wax is a by-product, a mixture of ethylene glycol and gelatine in aqueous solution is the emulsifier used (Hitotsumatsu *et al.*—*Japan 4126* [51]). A process for olive oil comprises agitation with air, a thorough water wash, and a heat treatment (Miranda-Sampedro—*Span.* 199,056).

General information articles on degumming, refining, etc., were prepared by Sergeev (*Masloboino Zhirovaya Prom.* 18, No. 2, 11) Castro-Ramos (*Grasas y aceites* 2, No. 4, 26), and Nosti-Vega & Castro-Ramos (*Ibid.* 3, 147). A new vessel for such processes has liquid phase indicators and a multi-tube draw-off system for drawing off of individual layers after settling (Sawyer—*U. S.* 2,661,093). A settling tank with overflow sleeves is designed for separating sediment from crude cottonseed oil (Baboshin—*Masloboino Zhirovaya Prom.* 18, No. 4, 27). Ultrasonic waves could be used to partially decompose carotenoids and chlorophyll in vegetable oils (Maffei—*Olearia* 7, 79). This shifts the fluorescence from yellow to blue.

As fundamental data for commercial deacidifying lard by steam stripping, the rate of free fatty acid removal with steam is determined and defined by two equations (Walt & Rushton—*J. Am. Oil Chemists' Soc.* 30, 147). A combination of distillation *in vacuo* with alkali refining and clay decolorization is patented for refining chrysalis oils (Kobayashi—*Japan 1535* [52]). Sakurai (*Japan 1630* [52]) esterifies the free acids of rice oil with lower alcohols and removes these esters by distillation under *vacuo* as a method of deacidification.

Highly acid black grease is improved by heating to cause re-esterification (Kirby—*U. S.* 2,547,014). Chrysalis oils of acid numbers as high as 72 are converted to neutral oils by re-esterification with glycerol in the presence of zinc compounds as catalysts (Imada & Shiroshina—*Japan 1536* [52]).

Equipment was designed for refining and fractionating oils with low boiling hydrocarbons based on differences in solubility of the free fatty acids and of fatty materials at certain critical temperatures and pressures (Palmer—*U. S.* 2,658,907). This method is applied for removal of free fatty acids from wool fat (Passino & Meyers—*U. S.* 2,649,466). Rice oil is deacidified and dewaxed by washing with lower alcohols (Koyama—*Japan 7225-6* [51], 3032 [51]; Sumimoto—*Japan 4934* [51]). A modification of this process does the washing with the oil dissolved in trichloroethylene (Yamagiwa & Yoshida—*Japan 2424* [51]). Another variation to the use of alcohol with this oil comprises dissolving the oil in the alcohol and precipitating free fatty acids with urea (Tsuruta & Tamura—*Japan 1292* [52]). Furfural extraction is used for the removal of free fatty acids and color from cottonseed, peanut, and sunflower oils (Gonzalez-Quintana—*Rev. fac. quim. LaPlata* 23, 59).

The innovations in alkali refining are mainly for the purpose of minimizing neutral oil losses. Clayton (*U. S.* 2,641,603; *Reissue 23,680* of *U. S.* 2,412,251) uses just sufficient caustic for neutralization and precipitation of gums so as to inhibit emulsion formation, then, centrifuges foots from the oils; a second refining may be made with sodium carbonate as the refining agent. In a two stage refining process part of the caustic is used in the first step and the refining is completed by retreatment with dilute caustic while vigorously stirring (Aktiebolaget Separator—*Brit.* 634,035, *Swed.* 138,985, *U. S.* 2,657,224, Bhasin & Aggarwal—*J. Sci. Ind. India*, 12A, 193). For the same purpose Dron & Lindsey, Jr. (*U. S.* 2,644,004) use strong lye in first refining at low temperature, add water to dilute the residual lye, and raise the temperature to complete the refining. Taniguchi (*Japan 2785* [52]), dissolves the crude oil in methanol for a caustic refining process and separates a methanol-refined oil layer from foots at 70°. Crude soybean oil is dissolved in a hydrocarbon solution and refined with a methanol-sodium hydroxide solution (Ayers—*U. S.*

2,614,111). The additives patented for reducing oil losses in alkali refining are organic amino-carboxylic acid compounds (James—*U. S.* 2,638,476) and tartaric acid or its salts (Lever Brothers & Unilever Ltd.—*Brit.* 676,574). Newly patented refining reagents are lignin made water soluble by treatment with caustic (Tausky—*U. S.* 2,654,766), and sodium sulfite (Frankel—*U. S.* 2,646,436). The latter is intended for solvent-oil while still in the miscella stage. Fal'kova & Ozernova (*U.S.S.R.* 77,359) recommends using anhydrous sodium carbonate for this purpose.

Some economy in bleaching oils that are to be hydrogenated is attained by partially prebleaching and completing the bleach during hydrogenation by adding bleaching earth and carbon with the hydrogenation catalyst (Bahadur—*J. Sci. & Tech., India*, 9, 63). Vogel (*U. S.* 2,639,289) uses magnesium silicate having active adsorbing surfaces for both bleaching and refining vegetable oils. A two stage bleaching process comprises using acid-activated earth and rebleaching with alkaline treated earth (Takayasu—*Japan 6925* [51]). The use of Fuller's earth and active carbon for bleaching of fats and oils was reviewed (Anon.—*Seifen-Öle-Fette-Wachse* 78, 23). A lower cost method for activating Spanish clays intended for bleaching oils involves treating with sulfuric acid instead of hydrochloric acid (Castro-Ramos & Nosti-Vega—*Grasas y aceites, Spain*, 2, No. 1/2, 7).

The activities on recovery of oil from decolorizing earth pertain to decoction assisted with surfactants (Debruyne—*Bull. mens. inform. ITERG* 7, 109), hydrogenating waste before separating the fatty material (Ueno—*Japan 7040* [51]), and a bibliography of 37 references on the subject (Jaillet-Rouyer—*Bull. mens. inform. ITERG* 7, 67).

Processes utilizing chemicals for bleaching fats and oils use a combination of sulfuric acid and zinc powder (Mochida—*Japan 3317* [51]), and hydrogen peroxide (Koyama—*Japan 2584* [52]) for rice oil; hydrogen peroxide for soap stock (Samarin & Gurevich—*Masloboino Zhirovaya Prom.* 18, No. 4, 25); ozone, chlorates, sulfites and hypochlorites for wool grease (Torres—*Anales fac. farm y bioquim. Univ. San Marcos* 1, 508); and hypochlorites for cottonseed oil (Zharskii—*Masloboino Zhirovaya Prom.* 18, No. 2, 23). Optical bleaching agents which induce a bleached or whitening aspect to solid fats and waxes are listed in a patent (Sandoz Ltd.—*Swiss 285,144 Cl. 38a*).

A method of calculating optimum blowing steam rate for deodorization and steam distillation in oil processing is based on curves derived from various operating data and extrapolating (Chirgwin—*J. Am. Oil Chemists' Soc.* 30, 583). For the same purpose and for distillation, Anders (*Seifen-Öle-Fette-Wachse* 76, 368) recorded the relationships of pressure, quantity of steam, and temperature. In a new deodorizer, steam forces liquid up to the vapor area of the still where it impinges on a baffle plate to present itself as a thin hot film for efficient evaporation of lower-boiling constituents (Metallgesellschaft A.G.—*Brit.* 685,719). A method for determining the degree of completion in commercial deodorization is based on capacity of steam volatile material to reduce potassium permanganate (Kaganowicz—*Przemysl Chem.* 31, 358).

A method of deodorizing chrysalis oil is based on treatment with inorganic acids followed by steam distilling to remove the low-boiling fraction (Nagai—*Japan 179,944* [49]). Fish oil containing a solution of sodium sulfate, sulfonated oil, and reducing iron compounds is electrolyzed at 20-95° to destroy the fishy odor (Itakura—*Japan 3318* [51]). Two articles indicate that improved deodorization of oils is obtained by steaming in the presence of citric acid (Lyubchanksya—*Masloboino Zhirovaya Prom.* 18, No. 6, 28; Zharskii—*Ibid.* 30).

Flavor and odor stability of hydrogenated oils are improved by propane extraction at temperatures critical for the selective removal of the unsaponifiable portions (Mattil—*U. S.* 2,613,215).

BYPRODUCTS OF REFINING: ALCOHOLS, PHOSPHATIDES, STEROLS, ETC. Rossouw & von Rudloff (*J. Appl. Chem. London* 2, 335) studied the saponification step in the lime method of production of alcohols from wool wax and found that twice the required amount of lime, or presence of 20% calcium stearate, or one percent lye does not accelerate saponification; but, the presence of a nonionic surfactant or "cellulose" doubled the rate, or that rates could also be increased by heat but reaction products darken above 140°. A rapid process for wool grease alcohol production comprises saponification with sodium hydroxide, addition of a lower alcohol-water mixture and extraction with naphtha (Christenson & Harpt—*U. S.* 2,619,945). To produce alcohol from spermaceti, Spada & Gavioli (*Farm. sci. e tec. Pavia* 7, 435) saponify with monoethanol amine and extract the alcohol with ether.

The gum and wax foots removed in degumming rice oil are heated and centrifuged to produce aqueous gum solutions and oil-wax products (Strezynski & Lawatsch—*U. S.* 2,663,717). Cousins *et al.* (*J. Am. Oil Chemists' Soc.* 30, 9) demonstrate production of a white wax product from rice bran oil tank settlings by solvent extraction and bleaching the crude wax extract with hydrogen peroxide and chromates.

Utilization of refining wastes was the subject of two reviews. One contains general information (Desikachar *et al.*—*Indian Soap J.* 17, 289), and the other stresses extracting vitamin E, tocopherols, from the wastes and lists the tocopherol content of many oils (Verma—*Ibid.* 18, 55). Vitamin A was concentrated from fish liver oil by selective extraction with furfural and washing the crude extract with 80% methanol (Abe—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 229). Three methods are on production of carotene from palm oil. One deals with distilling the oil as methyl esters and further concentrating the vitamin A fraction by chromatography (Blaizot & Cuvier—*Farmaco, Ed. prat., Pavia* 8, 3); the second involves separation of the unsaponified fraction of palm oil which contains the vitamin A (Blaizot—*U. S.* 2,652,433) and the other method involves the use of powdered rubber for absorbing the carotene from either the oil or unsaponifiable fraction (Boldingh—*U. S.* 2,632,012; *Dutch* 66,316, 68,577). Residual lipides of expressed palm pulp are recommended as good sources of carotene (Blaizot & Cuvier—*J. Am. Oil Chemists' Soc.* 30, 586).

Oat oil extracted with ether-methanol mixed solvent and treated with acetone while still in the solvent yields a phosphatide precipitate that is suitable for use as an emulsifier (Washburn—*U. S.* 2,636,888). The application of phosphatide emulsifiers in food processing was reviewed by Aylward (*Food Manuf.* 27, 311). Two procedures for improving lecithin as an emulsifier are based on mild oxidation with chemicals (Gaver *et al.*—*U. S.* 2,621,133; Julian—*U. S.* 2,629,662).

Crude cholesterol is concentrated by crystallization from alkali metal hydroxide-alcoholic solution in preparation of a U. S. Pharmacopoeia product (van Ness—*U. S.* 2,648,687). Pure gossypol was isolated for nutritional and pharmacological study by dissociating the gossypol-acetic acid complex prepared from the butanone extract of defatted cottonseed flakes (King & Thurber—*J. Am. Oil Chemists' Soc.* 30, 70). Cerebrosides were extracted from spleen and brain tissue lipides, for biochemical investigations, by boiling in chloroform-methanol solvent and isolated by virtue of their property of accumulating at the interphase zone when trichloroacetic acid was added (Uzman—*Arch. Biochem. & Biophys.* 45, 149). Sesamin was isolated and found to have antitubercular activity (Gangadharam *et al.*—*J. Indian Inst. Sci.* 35A, 69).

WINTERIZATION, SPLITTING, AND FRACTIONATION. Phase relations in the solvent winterization of cottonseed oil in 85-15 acetone-hexane mixture were developed to serve to indicate conditions for most economical separation of the stearin fraction (Boucher & Skau—*J. Am. Oil Chemists' Soc.* 30, 455.) In two publications on direct chilling winterization it is pointed out that maintaining a small temperature differential between oil and coolant reduces oil inclusion in the precipitate and induced crystallization desirable for filtration (Greenfield—*U. S.* 2,619,421; Kupchinskii—*Masloboino Zhirovaya Prom.* 18, No. 2, 8). The addition of small amounts of sulfide of a high-molecular alkyl-substituted aryl ether carboxylic acid salt to oil being winterized or fractionally crystallized also induces desirable crystallization (Mattil—*U. S.* 2,631,156). Winterized characteristics are induced in some oils by addition of certain compounds such as zirconium or zirconyl soap (Royce—*U. S.* 2,646,356), and coconut oil, petrolatum, lanolin, ethylene glycol monethyl ether acetate, lactic acid, or linoleic acid dimer (Ayers & Scott—*U. S.* 2,643,260).

In graining fats and oils for the production of olein the addition of naphthenic acid induces improved separation of the stearin fraction (Bezzubov *et al.*—*Masloboino Zhirovaya Prom.* 13, No. 1, 11). A lower fraction is distilled from coconut oil so as to improve the residue fat for edible purposes and get a distillate that is useful as a resin plasticizer (Nakajima—*Japan* 2417 [52]).

General papers on fat splitting are on: alkaline hydrolysis (Jellinek—*Rev. Pure & Appl. Chem.; Australia* 2, 139), autoclave splitting (Radfang—*Seifen-öle-Fette-Wachse* 79, 250, 273), continuous splitting (Ferrandini—*Ingegneria chim.* 1, No. 3, 10); and fat splitting agents (Nishizawa—*Yushi Kagaku Kyokaiishi* 2, 2). The observation that the velocity constants of Twitchell hydrolysis increases about two fold for each 10° rise in temperature is interpreted by Hartman (*J. Am. Oil Chemists' Soc.* 30, 349) to mean that the rate of reaction is governed by hydrolysis already completed rather than by dif-

fusion of water into the fat phase. Loncin (*Fette u. Seifen* 55, 7) describes the splitting mechanism thus: the speed of hydrolysis varies directly with the free fatty acid content; the lower fatty acids are more pronounced accelerators percentage-wise; and the reaction first develops only acids and diglycerides until a fatty acid concentration of 10% is reached. Yonese (*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 50, 51, 53, 55, 498, 500, 503; 55, 268, 311) demonstrates that the process is stepwise, and records data from commercial scale splitting of coconut and palm oils. A comparison of glycerol recovery from Twitchell versus autoclave sweet waters indicates that percentage yields are approximately the same (Nagata *et al.*—*Ibid.* 54, 169). New fat splitting catalysts demonstrated are: an acid regenerated cation exchange resin, "Dowex-50," (Sutton & Moore—*J. Am. Oil Chemists' Soc.* 30, 449), tri- and tetrabutynaphthalenesulfonic acids prepared from naphthalene, butanol, and sulfuric acid (Fukuzumi & Ozaki—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 727; *Japan* 6731 [51]), a reaction product of tung oil, butanol and sulfuric acid (Miyazi & Ueno—*Japan* 3034 [51]), and phthalic anhydride (Takao & Tomiyama—*Japan* 4178 [52]). New patents for autoclave hydrolysis describe the following equipment and processes: hydrolysis in two successive stages with different water to oil ratios (Bamag Ltd.—*Brit.* 679,538); a process giving 75% hydrolysis in which unhydrolyzed fat is separated and returned to the splitting unit (Palmer—*U. S.* 2,654,768); a method in which the novelty pertains to specific ranges of water to fat ratios and to temperatures that induce solvency of split fatty acids in the aqueous phase (Mortenson—*U. S.* 2,645,651); and use of antioxidants to inhibit decomposition or darkening of product (Ross & Trent—*U. S.* 2,619,494).

Wachs (*Z. Lebensm.-Untersuch. u. Forsch.* 96, 168) improved a laboratory molecular distillation apparatus and demonstrated the equipment with data on separations of sesamolin from sesame oil, and monoglyceride from technical mixtures. Application of cylindrical molecular distillation type apparatus to the distillation of rice oil acids gives good fractionation with least polymerization (Yamakito—*Bull. Inst. Chem. Res., Kyoto Univ.* 24, 81). With falling-type molecular stills efficiency of fractionation is equal for plant and laboratory scale models (Komori *et al.*—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 225). Information on production of vitamins A and E, sterols, and other constituents of oil by molecular distillation is reviewed (Carola—*Olii minerali, grassi e saponi, colori, e vernici* 29, 117). Molecularly distilled vitamin A concentrate from whale liver oil had an actual biological potency 65.7% of that determined spectroscopically; whereas for the straight oil the figure was 55.6% (Sabashi—*Bull. Japan Soc. Sci. Fisheries* 16, 46).

A comprehensive article on distillation of fatty acids or their lower alcohol esters by Stage (*Fette u. Seifen* 55, 217, 284, 375) contains information on types of stills, ratios of plate numbers to reflux, influence of pressure, problem of moisture, distillation schemes, distillation curves and tables for many binary mixtures, steam concentration relationships, and other useful information. Stage with others (*Ibid.* 513, 580) also designed a laboratory steam still; plotted the distillation behavior of C₄ to C₂₂ fatty acids, and of the coconut oil acids. The historical development of the continuous fatty acid still is depicted by Potts (*J. Am. Oil Chemists' Soc.* 30, 49), who with Olson (*U. S.* 2,627,500) designed a new still that has two or more distilling zones. Pretreatment of crude fatty acid mixtures with sulfuric acid improves color of the distilled fractions and inhibits development of color (Terry & Warner—*U. S.* 2,652,414).

Several investigators developed fundamental data that is applicable to commercial fractionation of fatty materials by crystallization. Cording, Jr. *et al.* (*J. Am. Oil Chemists' Soc.* 30, 66, 111) determined the crystallization behavior of white grease from several lower ketone and ether solutions under dry and moisture saturated conditions preliminary to development of a continuous process for production of lard oil. The amounts of soybean oil fatty acids that crystallize from 10% acetone solutions at various temperatures are tabulated as basic data for the preparation of a paint oil fraction (Maruta—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 234). Petroselinic acid was isolated from celery seed oil fatty acids (Skellon & Spence—*Chemistry & Industry* 1952, 691) and kamalolenic acid from fatty acids of the seed oil of *Mallotus philippinensis* (Council Sci. & Ind. Res.—*Indian* 44,736) by simple crystallizations from organic solvents. A stepwise crystallization process of repeated cooling and filtering out of crystal fractions of lower fatty esters was patented (Armour & Co.—*Brit.* 690,885). Another patent (Pramuk *et al.*—*U. S.* 2,645,652) was issued covering a similar process for the crystalliza-

tion of oils from solutions of special ratios of acetone, hexane, and oil.

Hixon & Miller (*U. S. 2,631,157*) and Dickinson (*U. S. 2,660,590*) fractionate oils into free fatty acid, relatively saturated glyceride, and relatively unsaturated glycerides with use of liquefied gaseous hydrocarbons. Solutions of oils with these separate into two immiscible phases containing oil fractions respectively more suitable for food and for paint uses.

Abe (*J. Oil Chemists' Soc., Japan, 1, 169, 171; 2, 16, 19*) recorded solubilities of 14 common commercial oils and fats in furfural to serve as basic information for fractionation by liquid-liquid extraction. Nolla *et al.* (*Afinidad 29, 446*) using such a method on rapeseed oil produces unsaturated fractions with iodine numbers as high as 144. Fractions with iodine values as high as 144.51 are obtained from soybean oil in commercial equipment with furfural and petroleum ether as solvents (Sakurai—*J. Oil Chemists' Soc. Japan 1, 115*). Combinations of liquid-liquid fractionations with either partial saponification (Rigamonti—*Olearia 6, 289*) or interesterification (Aktiebolaget Separator—*Brit. 632,797*) of the oils induce greater yields of unsaturated fractions. Phase composition charts were recorded for quaternary systems containing methyl oleate, methyl stearate and various binary mixtures of many commercial solvents to be used as fundamental data for designing liquid-liquid fractionation processes (Ruis & Alicia-Crespi—*Anales real soc. españ. fis. y. quim. 49B, 63*). Metzsch's (*Angew. Chem. 65, 586*) tables on solubilities, critical mixing temperature of solvent pairs, and recommended solvent pairs for 400 materials which include fatty substances are useful for the same purpose. Tower-type equipment was patented for the process (Pittsburgh Plate Glass Co.—*Brit. 685,681*). A means of liquid-liquid contacting and liquid separation in centrifuges is also designed for this process (Gloyer—*U. S. 2,646,439*).

Fractionation of fatty materials by selective precipitation with urea was reviewed (Rigamonti & Riccio—*Fette u. Seifen 55, 162*). This type technique has been demonstrated by: Knafo (*Bull. mens. inform. ITERG 7, 114*) for concentrating linoleic acid from the mixed fatty acids of soybean oil and from linseed oil acids; Swern & Parker (*J. Am. Oil Chemists' Soc. 30, 5*) for concentrating linoleic from corn oil fatty acids and linolenic from perilla oil and linseed oil fatty acids; Ericson & Clegg (*Trans. Kansas Acad. Sci. 55, 493*) for removing pigments and segregating the unsaturated acids from corn oil fatty acids; Knafo (*Bull. mens. inform. ITERG 7, 24*) and Skellon & Taylor (*Nature 171, 266*) for isolation of erucic acid from rape oil fatty acids; Skellon & Taylor (*J. Chem. Soc. 1953, 1433*) in the preparation of brassicid acid from rape oil, Knafo (*Bull. mens. inform. ITERG 7, 291*) for purifying ricinoleic acid; and Catravas & Knafo (*Ibid. 6, 384*) for precipitation of oxidized fatty acids from castor oil. Studies of the application of the urea segregation process to rice oil shows that selective precipitation occurs with the methyl esters but not with the free fatty acids (Sakurai—*J. Chem. Soc. Japan, Ind. Chem. Sect. 55, 16, 80*). The use of this process for fractionation of fatty amides, nitriles, esters, alcohols, aldehydes or ketones was patented (N. V. de Bataafsche Petrol. Maatschappij—*Brit. 665,248*).

HYDROGENATION. Reviews on hydrogenation of fatty oils were published by Ramos-Ayerbe & de la Ossa (*Grasas y aceites, Spain, 2, No. 1/2, 11; No. 3, 71; No. 4, 19; 3, 95*). Ueno (*J. Oil Chemists' Soc. 2, 54*), and Muscari-Tomajoli (*Olii, grassi e saponi, colori e vernici 29, 57*). Also a general review on hydrogenation has a section on fatty oils (Atwood—*Ind. Eng. Chem. 45, 1976*).

Studies were made on the mechanism of hydrogenation. According to Suito & Aida (*J. Chem. Soc. Japan, Ind. Chem. Sect. 54, 765*) the activation energies for hydrogenation of oleic acid is 7.6 kcal. and for the methyl oleate 5.8; and the heat of reaction is 35.6 kcal. per mole in both cases. Oliver & Borrero (*Grasas y aceites, Spain, 2, No. 4, 7*) plotted the changes occurring in density, refractive index, and iodine number of olive oil during hydrogenation and developed equations from these data for control of hydrogenation. Petroselinic acid hydrogenates more rapidly than its trans-isomer, petroselaic acid (Figulevskii & Artamonov—*Zhur. Obshchei Khim. 22, 1140*). The rate of hydrogenation of monounsaturated acids declines with an increase in molecular weight (Artamonov—*Ibid. 23, 216*). Data are recorded to show change in fatty acid composition during hydrogenation of a domestic tung oil (Planek *et al.*—*J. Am. Oil Chemists' Soc. 30, 598*). During hydrogenation, migration of the double bond occurs in both directions but in all cases it is more strongly pronounced in the direction opposite to the ester group (Boelhouwer *et al.*—*Ibid. 59*). Feuge *et al.* (*Ibid. 454*) found that as linoleates are hydrogenated, as much as 17.4% conjugate to diene struc-

tures; that about 89% of the oleate formed in the first stages contains trans bonds; and that after the linoleate disappears the percent of trans isomers on the basis of total amount of oleate tends to remain above the equilibrium concentration of 67% for normally elaidinized methyl oleate. Conditions which favor selective hydrogenation also favor the development of trans isomers (Sims & Hilfman—*Ibid. 410*). Isomerization during hydrogenation is exceptionally high when the nickel catalyst contains some nickel sulfate (Catravas—*Compt. rend. 236, 716*). The rate of hydrogenation in organic solvents is greater than without, toluene is good in this respect (Sokol'skii & Melekhina—*Doklady Akad. Nauk S.S.S.R. 89, 881*). Rice oil which is hydrogenated at high hydrogen pressures in the presence of one percent nickel and one percent trichloroethylene is relatively light colored (Hitotsumatsu & Takeshita—*Japan 5935 [51]*). Hydrogenation of castor oil at 150-60° and at 10 atmospheres saturates the double bonds without loss of hydroxyl groups (N. V. de Bataafsche Petrol. Maatschappij—*Brit. 680,508*); Gupta & Aggarwal—*J. Sci. Ind. Res., India 11B, 303*). In a process in which cycloaliphatic hydrocarbons are used as hydrogen donors, the polyunsaturated fatty acids are selectively converted to monounsaturated acids (Baldwin & Floyd—*U. S. 2,656,371*). A method for measuring the selectivity of a hydrogenation process, the so-called "selectivity index," is based on the amount of hydrogen used until disappearance of double bonds in a sample of linoleic acid in relation to that theoretically required for 100% hydrogenation of one double bond only (Tyutyunnikov & Fraier—*Masloboino Zhirovaya Prom. 18, No. 2, 14*).

Seipioni & Greppi (*Ann. chim., Rome, 41, 785*) experimentally determined the conditions under which both hydrogenation and alcoholysis can take place in a combined operation, and applied the procedure to several oils for making hard methyl, butyl, isopropyl, furfuryl and tetrahydrofurfuryl esters. The presence of considerable water in the hydrogenation process produces hydrolysis so as to yield hard free fatty acids; whereas if caustic is also added the products are hard soaps (Yoshijima & Tsutsumi—*J. Chem. Soc. Japan, Ind. Chem. Sect. 54, 554*; Tsutsumi *et al.*—*Ibid. 235*; Tsutsumi—*Japan 4585 [52]*; Nishimura—*Japan 4630 [51]*; Ueno—*Japan 3079 [51]*, 7227 [51], 128 [52]; Tsuchiya *et al.*—*Japan 1231 [51]*).

Nanavati (*Proc. symposium Indian Oils Fats Nat. Chem. Lab. Poona 1951, 169*) issued instructions for preparation of hydrogenation catalyst. Faulkner (*U. S. 2,609,346*) prepares nickel catalyst dispersed in hard fat and molded into blocks. Catalyst compositions patented by Paterson (*U. S. 2,645,620*) contain specific mixtures of nickel, silicon dioxide, and alumina. Treatment of reduced nickel catalyst with very dilute solutions of hydrogen peroxide is said to improve its stability (Iijima—*Japan 2224 [51]*). Hydrogenation with a copper oxide-barium sulfate catalyst is said to selectively reduce polyunsaturated acids to the monounsaturated stage (Miyake *et al.*—*Japan 3077-8 [51]*). Binary metal catalyst containing nickel and either copper or chromium, or aluminum were prepared and claimed to be superior to ordinary nickel catalyst (Kiryu—*J. Chem. Soc. Japan, Ind. Chem. Soc. 51, 81*; Ueno—*Japan 3319 [51]*; Sanchez-Delgado—*Rev. cienc. apl., Madrid, 7, 39*; Schertel—*Ger. 807,814*). Several articles gave details on recovery of nickel and regenerating for reuse (Artamonov *et al.*—*Masloboino Zhirovaya Prom. 18, No. 6, 17*; Ramaswami—*Indian Soap J. 18, 20*; Bhasin *et al.*—*J. Sci. Ind. Res., India 11B, 248*; Ohashi & Nobori—*J. Chem. Soc. Japan, Ind. Chem. Sect. 54, 287*).

A test for the activity of hydrogenation catalysts is based on the average volume of hydrogen uptake per hour in a standard hydrogenation (Tyutyunnikov & Fraier—*Masloboino Zhirovaya Prom. 18, No. 1, 7; No. 3, 10*).

Panyshv (Ibid. No. 5, 25) described the design and installation of tubular heat-exchangers for the cooling of hydrogenated fat. Hydrogenated fats are washed with dilute solutions of citric, tartaric, or phosphoric acid for removal of metallic contaminants (Dron & Lindsey, Jr.—*U. S. 2,650,931*).

INTERESTERIFICATION, ESTERIFICATION, CRYSTAL MODIFICATION, AND ISOMERIZATION. Experiences on interesterification of various oils were recorded by several investigators. According to Toyama & Yamaguchi—*J. Chem. Soc. Japan, Ind. Chem. Sect. 54, 188*), the process applied to peanut and rice bran oils raises their cold points, whereas with coconut oil the cold point is lowered. With cuttle fish oil the cold point can be increased as high as 17° (Yoda—*Japan 2425 [51]*). Interesterification of cuttle fish and soybean oils followed by destearination yields oils with good drying characteristics (Toyama *et al.*—*J. Oil Chemists' Soc., Japan 2, 66*). With sardine oil the process induces an easily separable stearin fraction and the destear-

minated oil could be bodied sufficiently for paint uses (Toyama *et al.*—*Ibid.* 63). The literature on interesterification was reviewed by Huesa (*Grasas y aceites, Spain*, 2, No. 1/2, 18).

Fundamental information, such as reaction conditions, yield versus time curves, etc., for alcoholysis of various fats and oils with methyl, ethyl, propyl, and butyl alcohols using *p*-toluene sulfonate as the catalyst is recorded by Poré & Verstraete (*Oleagineux* 7, 641). Industrial equipment for the process is also described (Santelli—*Ibid.* 481). Use of lithium as an alcoholysis catalyst was patented (Goldsmith—*U. S.* 2,622,091).

Lard is improved in appearance, keeping qualities, baking qualities, and workability by heat treatment with a multivalent metallic catalyst (zinc or lead compounds) or sodium amide, or alkali metal, or alkali metal hydroxide or hydride (Mattil & Norris—*U. S.* 2,625,478-82; Mattil & Nelson—*U. S.* 2,625,483; Dominick *et al.*—*U. S.* 2,625,484-5; Nelson & Mattil—*U. S.* 2,625,486-7).

Conditions for elaidimization of various fatty acids with use of selenium as the catalyst are recorded by Skellon & Spence (*Chemistry & Industry* 1953, 302).

Fatty Products (Except Detergents)

HOUSEHOLD FATS. The new information on margarines deals with its manufacture. One formulation devised for army combat rations that is spreadable in both warm and cold climates consists of about 84% vegetable salad oils and 16% distilled monostearate (Jones *et al.*—*J. Am. Oil Chemists' Soc.* 30, 609). The suitability of coconut oil for margarine manufacture is enhanced by distilling off the lower glycerides until the residue has a saponification value of 229-241 and then hydrogenating this residue (Blum—*U. S.* 2,657,995). The methanol soluble fraction of a vegetable oil phosphatide is added to margarine to eliminate spattering on heating (Mattikow—*U. S.* 2,640,780). When adding phospholipides to improve the keeping qualities and palatability of margarine the amount should be less than 0.2% because larger amounts impart bitterness (Lyubchanskaya & Vol'kovskaya—*Masloboino Zhirovaya Prom.* 18, No. 4, 20). Other margarine developments are a dye emulsion for coloring (Barch—*U. S.* 2,652,334), and packages with color capsules in which dyeing is brought about by kneading (Hensgen *et al.*—*U. S.* 2,646,190, 2,652,336).

A procedure suggested for the control of coloring in butter manufacture is based on separating some butterfat from the cream and determining the amount of artificial color necessary to match the desired color intensity (Reinart & Brown—*Can. Dairy Ice Cream J.* 31, No. 5, 31).

Technique for producing "marbling" of beef by the mechanical injection of fat into the vascular systems of carcasses and cuts is described by Shea *et al.* (*Food Tech.* 7, 437). The process is said to produce the desirable flavor of high grade beef in lower grades.

In a study of palatability of doughnuts fried in various oils of low quality, the first batches had poorer flavor than succeeding batches (Lips & Hamilton—*J. Am. Oil Chemists' Soc.* 30, 43). It is suggested that in the early batches, undesirable flavors and odors are carried off by water vapor. A chemical and physical investigation on frying indicates that in general the heating lowers the melting point and iodine number, and raises the density and acid number of fats; with butter the butyric acid number decreases; and that the fat absorbed by fried potatoes has the same composition as the frying fat (Stoy—*Deut. Lebensm.-Rundschau* 47, 271).

Treatises on inhibiting fat bloom of chocolate goods deal principally with tempering so that the mass stiffens with the cacao butter in the stable β -crystal form (Sachsse & Rosenstein—*Fette u. Seifen* 55, 26, 122, 196; Kleinert—*Rev. intern. chocolat.* 8, 72). A new patent deals with inhibiting the fat bloom by adding partial fatty esters of polyhydric compounds to the chocolate products (Cross—*U. S.* 2,626,216).

Deterioration of fat to free fatty acids on storage of cake mixes is related to moisture content, and such deterioration reduces ability to yield good cakes (McWilliams & Soloski—*Cereal Chem.* 30, 367). Mixes compounded with low moisture (less than 2.8%) flour perform satisfactorily even after one year's storage. Glyceryl monostearate when used to improve baking performance in fermented goods is most effective when added as an aqueous emulsion; whereas for baking "sponges" and cakes the dry powder works better (Harper—*Food Manuf.* 29, 429). Monoglycerides or polyoxyethylene emulsifiers increase white cake volume at most shortening levels and permit the use of higher shortening levels for getting maximum cake volume (Favor *et al.*—*Cereal Chem.* 30, 435). A shortening made by substituting acetate radicals for one-third of the fatty acids in the glycerides has a desirable plasticity over a

wide temperature range (Baur & Lange—*U. S.* 2,614,937). A patented dessert or confectionery topping containing carbohydrate, protein, fat, and partial glyceride emulsifier is designed for reconstitution with water and whipping to a definite volume with air (Diamond—*U. S.* 2,619,423).

A fat base for frozen desserts contains hydrogenated coconut oil, cottonseed oil, sugar, propyl-3,4,5-trihydroxy benzoate, and locust bean gum (Katz—*U. S.* 2,658,831). A fat emulsion to enrich reconstituted milk, for ice cream mixes, and other food uses consists of hydrogenated cottonseed oil, casein, butter flavor, vitamin concentrate, and water (Peebles & Girvin—*U. S.* 2,622,984). Mechanical equipment to produce emulsions resembling milk and cream was patented (Bastoni—*Ital.* 471,233). The fat component of a new synthetic infant milk contains 50% palm oil blended with other fats so as to have an iodine number greater than that of the fat of cows milk (Howard & Muller—*U. S.* 2,659,676). Special waxy triglycerides useful as protective coating for fruit, cheese, candy, etc., and useful as hairdressing compounds, suppositories and medicinal carriers are made by interesterifying fats with triacetin to produce mixed esters with an average of one acetate radical per glyceride (Baur—*U. S.* 2,615,160). A therapeutic fat product intended as a concentrated source of calories contains edible fat, sugar, phosphatides, water and emulsifier, and is emulsified so that the fat particles are about 0.5 μ inches in diameter (Wachtel & Meyer—*U. S.* 2,646,354). Oily vitamins were made into powder products using soybean meal or wheat germ flour and a fat-derived emulsifier as a carrier (NOPCO Chem. Co.—*Brit.* 681,930-1), and as aqueous emulsions using gelatin and partial fatty acid esters of polyhydric alcohols as emulsifiers (Zentner—*U. S.* 2,628,930). Lecithin is prepared in dry powdered form by combining with corn flour (Scharf—*U. S.* 2,632,705).

GENERAL EMULSIFIERS. Several improvements for manufacture of partial ester types were described. Iron, chromium, and cobalt soaps are claimed to be excellent catalysts for reacting glycerol with fatty acids (Voegeli—*U. S.* 2,628,967). A continuous process for producing monoesters comprises passing fatty acids with 4-10 times the necessary amount of glycerol through a heated zone and distilling off water and unreacted materials (Malkemus—*U. S.* 2,655,522). Monoglycerides were prepared from soybean oil by hydrolysis to the extent of 91%, adding the required amount of glycerol and reesterifying (Beal & Ludwig—*U. S. Dept. Agr., Bur. Agr. & Ind. Chem. AIC* 330, 4 pp.). A process of producing substantially pure monoglycerides comprises reacting glycerol with an oxo-carbonyl compound whereby two of the carboxyl groups of the glycerol are blocked, esterifying with an alkyl ester of a fatty acid, and then unblocking the two hydroxyl groups by removing the oxo-carbonyl compound (Norris—*U. S.* 2,619,493). 2-Monoolein, 2-monoelaidin, and 2-monoolein were prepared using benzylidene as the blocking group for the 1,3-positions (Martin—*J. Am. Chem. Soc.* 75, 5482). Fractional crystallization from organic solvents (Young & Black—*U. S.* 2,608,564; Nakamori—*J. Chem. Soc. Japan, Ind. Chem. Sect* 55, 277; Doadrio & Montequi—*Anales real soc. espan. fs. y quim., Madrid*, 48B, 687); liquid-liquid extraction (Goldsmith—*U. S.* 2,651,646), and distillation (Kuhrt—*U. S.* 2,634,278-9, *Brit.* 682,626), are all used to separate substantially pure monoglycerides from mixtures of mono-, di-, and triglycerides. Molten monoglyceryl stearate is sprayed in a cooling atmosphere to convert it to a fine powder for special uses (Allen & Arrowsmith—*U. S.* 2,633,604).

Wachs & Heine (*Fette u. Seifen* 54, 760) prepared monoglyceryl esters of oleic, linoleic, linolenic, and ricinoleic acids of 90% purities and compared their emulsifying capacity with commercial monostearin. The monoricinolein does not absorb water but has good wetting capacity; the others absorb 60-70 times more water than monostearin. In similar work, Kaufmann *et al.* (*Ibid.* 55, 670) prepared the monoglyceryl esters of stearic, pelargonic, 12-hydroxystearic, 9,10-dihydroxystearic, and 9,10,12-trihydroxystearic acids and recorded their effect on the interfacial tension between cottonseed oil and water. The data are plotted for different concentrations, and to show the relationship between number of carbons and the capillary activity coefficient.

A method for preparing substantially pure diglycerides comprises interesterifying a fat with glycerol and cooling to crystallize out solid diglycerides (Lange & Baur—*U. S.* 2,626,952).

Some literature on emulsifiers covered partial ester types in which the polyhydric moiety was other than glycerol. Reviews on their usefulness in the food industry were prepared by Pratt and Hays (*Food Eng.* 24, No. 5, 109) and by Scott (*Ibid.* 112). Monoesters of di- or trihydric alcohols made under pressure without use of catalyst are light colored (Mal-

kemus—*U. S. 2,655,522*). Partial fatty esters of glycerol, polyglycols, sugars, and the like have been patented for the preparation of water-in-oil emulsions (Bertram—*Dutch 70,436*), and for coating inner surfaces of containers to eliminate adhesion to meat packed therein (Edger—*U. S. 2,655,451*).

Partial polyoxyethylene glycol-fatty acid esters were prepared by reaction in toluene and with toluene sulfonate as the catalyst (Dolgopoloff—*Fr. 967,916*). Their use as shortening ingredients to increase volume and permit more sugar in cakes is the subject of a new patent (Griffin—*U. S. 2,662,015*). A mixture of 10% polyoxyethylene sorbitan monostearate and 90% castor oil is recommended as a base for ophthalmic ointments containing antibiotics (Hagiwara & Sugiura—*Acta Soc. Ophthalmol., Japan 57, 1*). Solubilization of hexane, toluene, benzene, and heptyl alcohol by polyoxyethylene emulsifier increases as the number of oxyethylene groups in the emulsifiers increase (Cohen—*Mém. services chim. etat, Paris 37, No. 1, 85*). The sharp boundaries formed between clear aqueous sols. of some fatty materials solubilized by polyethylene oxide esters of stearic acid and water have been studied with the Gouy diffusion apparatus (Saunders—*J. Chem. Soc. 1953, 1310*).

Fatty acid esters of polyoxyethylene sorbitan inhibit the action of pancreatic lipase on corn oil, but this inhibition is reversed by addition of bile salts (Minard—*J. Biol. Chem. 200, 657*). Such an emulsifier at 0.1-0.25% in drinking water showed no toxicity with rats for six months, but at 0.5% causes slight growth retardation (Sweeny & Oster—*J. Am. Pharm. Assoc. 42, 556*). At five percent in a casein basal diet the rats develop diarrhea and growth is retarded, whereas some similar emulsifiers even at 15% in soybean basal diets have no deleterious effect (Chow *et al.*—*J. Nutr. 49, 563*). One such compound, "Tween 80," is 20 times less toxic than another "Triton X-100," when tested on chick embryos (Herrmann *et al.*—*Proc. Soc. Exptl. Biol. & Med. 82, 392*). "Tween 60" and "Span 60" at six grams per day in humans produce no changes in gastric acidity or gastric motility (Steigmann *et al.*—*Am. J. Dig. Dis. 20, 380*). In discussing the use of these compounds in food, Pratt (*Food Technol., 6, 425*) emphasizes that in general use a person is not likely to ingest over 0.5 grams per day. However, a food protection committee of the Food and Nutrition Board of the National Research Council (News note—*Food Technol. 7, No. 10, 28*) concluded that polyoxyethylene stearate has not been demonstrated to be safe for food under all patterns of dietary consumption, and the Supreme Court (News notes—*Food, Drug, Cosmetic Law J. 8, 292; Chem. Eng. News 31, 1503*) has refused to review a decision of the third circuit court banning the emulsifier from bread standards.

A study of the metabolism of C₁₄-labeled sorbitan monostearate shows that 90% is hydrolyzed, that the sorbitan is largely excreted in the urine and that five to seven percent of the labeled carbon appears in the tissues 48 hours after feeding (Wick & Joseph—*Food Res. 18, 79*). Sorbitan monooleate will break kaolin and pectin gels to form suspension that will permit them to flow (Faeges *et al.*—*J. Am. Pharm. Assoc. Pract. Pharm. Ed. 14, 293*). The interfacial viscosity of films of sorbitan sesquioleate at a mineral oil-water interface at 25° increases to a maximum value in about 200 hours, then decreases until a constant value is reached in 400-600 hours (Sherman—*J. Colloid Sci. 8, 35*).

Determination of the emulsifying properties of lanolin constituents show that only the free acids and alcohols, especially the α , β -types are emulsifiers and that presence of isocholesterol reduces the emulsifying capacity (Tiedt & Truter—*J. Applied Chem. 2, 633*). Without emulsifier, whole milk powder disperses best when the water is at 105°F. and powder at 86°F., with emulsifier a temperature of about 120°F. for both gives optimum rate of dispersion (Mather and Hollender—*J. Dairy Sci. 36, 565*). Micellar weights as determined by intensity of scattered light were recorded for emulsions of various organic compounds with various sulfonated emulsifiers (Yurzhenko & Kucher—*Doklady Akad. Nauk S.S.S.R. 85, 1337*). Interfacial tension at a mixed monochlorobenzene-paraffin oil layer was determined for aqueous emulsifier solutions containing various concentrations of inorganic salts (van den Tempel—*Rec. trav. chim. 72, 419, 433, 442*). In this work equations were derived showing relations between emulsion particle concentration with rates of flocculation and time of coagulation. Viscosity of aqueous emulsions of xylene produced with various emulsifiers were measured and the results were discussed with regard to structure, effect of shear, and equations for calculating such data (Neogy & Ghosh—*J. Indian Chem. Soc. 30, 113*). Wesselbaum (*Fette u. Seifen 55, 333*) in a general discussion on emulsions stressed the importance of cavitation. An apparatus for determination of emulsifying

capacity comprised mechanical means of uniformly agitating several sealed samples and observing the emulsions formed (Miller & Jaskowski—*U. S. 2,662,752*).

Helin *et al.* (*Ind. Eng. Chem. 45, 1330*) correlated type and concentration of agents used in emulsion polymerization with rate of polymerization, stability of latex, and properties of the synthetic polymer. The anionic compounds generally are most suitable; cationic emulsifiers accelerate polymerization at high temperatures but the latex was unstable; and among non-ionic compounds amides alone equalled the soaps, but only at certain pH values. In similar work Tsvetkov & Yurzhenko (*Kolloid Zhur. 15, 308*) express the rate of polymerization of styrene with individual emulsifiers by equations. Polymerization rate of styrene is the same with sodium and potassium soaps and slower with the ammonia soap. Alkali salts of organic sulfonates produce the same rate with different alkali metals in the compounds (Fermor & Peizner—*Ibid. 292*). For the new low-temperature emulsion polymerizations, lower molecular weight soaps (as sodium caprylate) are effective emulsifiers (St. John, Jr. & Uraneck—*U. S. 2,615,009*). Rosin soaps on treatment with hydrogen peroxide become suitable for use in emulsion polymerization (Uraneck & Landes—*U. S. 2,630,426*). An article by Naidus (*Ind. Eng. Chem. 45, 712*) contains information on the fundamental principles of emulsion polymerization, the role of emulsifiers, pH, agitation and recipes for making polymers for paints.

The surface active fatty materials patented for breaking petroleum emulsion were a reaction product of tall oil, polyethylene glycol and triethanol amine (Kirkpatrick & Koehler—*U. S. 2,627,514*), fatty partial esters of highly polymerized ethylene glycol (Kirkpatrick—*U. S. 2,654,714*), a reaction product of a fatty amine and an aldehyde (Hughes & Fisher—*U. S. 2,646,404*), and hydroxy fatty acid amines (Hughes—*U. S. 2,646,405-6*).

CERTAIN FATTY ESTERS, ACIDS, AND OTHER DERIVATIVES OF FATS. Substituting the acetate radical for one-third or two-thirds of the acids of normal fat yields glycerides with improved properties for special uses. Acetomonostearin is very flexible over a wide temperature range and its films are impermeable to moisture (Feuge *et al.*—*J. Am. Oil Chemists' Soc. 30, 283*). A margarine fat prepared from aceto-olein and hydrogenated cottonseed oil was softer below room temperature and harder above room temperature than butter (*Ibid. 320*). Aceto glycerides prepared from lard, cottonseed oil, and hydrogenated fats can serve as extenders for commonly used vinyl resin plasticizers (Magne & Mod—*Ibid. 269*). A patented process of substituting lower fatty acid groups in glycerides included methods of cooling the products to waxy crystalline forms suitable for candy, frostings, food coatings, suppositories, etc. (Jackson—*U. S. 2,615,159*).

Toxicity studies have indicated that butyl stearate, methoxyethyl oleate, and similar fat derivatives are safe for application to films used for wrapping food (Smith—*Arch. Ind. Hyg. Occupational Med. 7, 310*).

Newly described methods of alcoholysis of fatty acids or fats make use of alkaline earth bases, as calcium oxide and strontium oxide (Hunn—*U. S. 2,654,767*) and "Amberlite IRA400," a commercial resin (Schlenk & Holman—*J. Am. Oil Chemists' Soc. 30, 103*) as catalysts for the process. According to Perron (*J. recherches centre natl. Labs. Bellevue No. 18, 141*), when *p*-toluene sulfonic acid is used as a catalyst for the process it forms an intermediate ester with the alcohol. Acetol esters of several fatty acids were prepared, converted to oximes, nitrophenylhydrones, and semicarbozones and their characteristics were recorded as fundamental data for identification of fatty acids (Paquot & Hiep—*Ibid. 136*).

Recent developments in the preparation of natural and synthetic fatty acids were reviewed by Gunstone (*Quart. Rev., London, 7, 175*). Pure oleic acid, methyl oleate (Khan—*J. Am. Oil Chemists' Soc. 30, 40*; Knight *et al.*—*Biochem. Preparations 2, 100*) and methyl linoleate (Swern & Jordon, Jr.—*Ibid. 104*) were prepared for fundamental laboratory studies. Linoleic acid was decarboxylated and reconstituted carboxyl-labeled for the same purpose (Howton *et al.*—*J. Am. Chem. Soc. 74, 1109*). Fatty acids were condensed with polysaccharides and proteins to serve in studies to determine if fatty acids are capable of acting as specific haptens groups in serological reactions (Jones *et al.*—*J. Chem. Soc. 1952, 5016*). Several iso-acids were synthesized and their melting points and long x-ray crystal spacing were recorded for comparison with those of the iso-acids of wool fat (Hougen *et al.*—*Ibid. 1953, 98*). 2,5,9-Trimethyl dec-2-enoic acid was synthesized and the relation to the structural features of tubercle bacilli acid was discussed (Joelyn & Polgar—*Ibid. 132*). Isanic acid was syn-

thesized for comparison with the natural acid (Black & Weedon—*Ibid.* 1785).

Short fatty acids and dicarboxylic acids were prepared for commercial use by oxidizing natural fatty acids and segregating the oxidized material by distillation (Logan—*U. S.* 2,662,908) or fractional crystallization (Richter & Berndtsson—*Swed.* 133,160, 134,616). Polyhydroxy fatty acids were distilled in the presence of oxygen for the commercial preparation of azelaic, pelargonic, sebacic and other acidic fission products (Logan—*U. S.* 2,625,558). Distillation of castor oil in the presence of sodium hydroxide and tricresol yields 2-octanone, 2-octanol, and sebacic acid (Soc. Organico—*Brit.* 675,434). Undecenoic acid obtained in the destructive distillation of castor oil is demonstrated to be effective in control of fungus infections (Lozan—*Anales fac. farm. y bioquim. Univ. nacl., Peru*, 1, 553) and of trichophyta (Kato & Nagano—*Tech. Repts. Kyushu Univ.* 25, 31), and its uses in medicine are discussed (Ultschmid—*Fette u. Seifen* 55, 700).

Mageli *et al.* (*Can. J. Chem.* 31, 23) synthesized ethyl 13,14-dihydroxybehenate, methyl 13,14-oxidoerucate, ethyl 13,14-acetoxybehenate and methyl 13,14-hydroxyacetoxybehenate from rape oil by a method which included hydroxylation and epoxidation with performic and peracetic acids. Some of the compounds were converted to aminohydroxy acids by aminolysis with liquid ammonia and ammonium bromide. Infrared and x-ray diffractions of hydroxy- and epoxy-compounds made from oleic and elaidic acids, respectively were recorded to substantiate previous hypothesis explaining formation of 10-hydroxy acid rather than mixed 9- and 10-isomers (O'Connor *et al.*—*J. Org. Chem.* 18, 693; Mack & Bickford—*Ibid.* 686). Thermal and x-ray characteristics are also recorded for a complete series of octadecenoic acids with monoxo- and monohydroxy groups at different carbon atoms (Bergstrom *et al.*—*Acta Chem. Scand.* 6, 1157). The general process of oxidizing unsaturated acids to hydroxy and epoxy compounds was also used to correlate the stereochemical relations involved in conversion of cis- and trans-2-octadecenoic acid to the three- and erythro-2,3-dihydroxystearic acids (Meyers—*J. Am. Chem. Soc.* 74, 1390). Epoxidized fatty acids were found useful in resin making (Rinse—*U. S.* 2,652,376), and as plasticizers (Greenspan & Gall—*Ind. Eng. Chem.* 45, 2722).

12-Oxo-8-octadecenoic acid was prepared from ricinoleic acid by oxidation with excess chromic acid in acetic acid solution (Nichols—*U. S.* 2,623,888-9).

The new developments in preparation of fatty peroxides pertain to concentrating by partitioning crude preparations between a polar and a non-polar solvent (Lundberg—*U. S.* 2,636,890; Privett *et al.*—*J. Am. Oil Chemists' Soc.* 30, 17), and by precipitation as urea complexes (Coleman *et al.*—*J. Am. Chem. Soc.* 74, 4886). Such peroxides are very efficient initiators of copolymerization (Swern—*J. Polymer Sci.* 11, 487).

Traynard (*Bull. soc. chim. France* 1952, 323) issued directions for the preparation of dihydroxystearic acid by oxidation of oleic acid with potassium permanganate. A process using hydrogen peroxide as the oxidizing agent was patented (Yoritachi—*Japan* 4534 [1951]). This process when applied to linoleates selectively hydroxylates at the 12,13-double bond leaving the 9,10-unsaturated (Arimune—*J. Chem. Soc., Japan Ind. Chem. Sect.* 54, 739, 760). Some hydroxy groups are produced by electrolyzing halogen saturated fatty acids (Terai *et al.*—*Ibid.* 47, 48).

In the preparation of mono-acetylenic fatty acids from the corresponding monounsaturated acids, dehydrobromination of the dibromides of the latter with sodamide in liquid ammonia is superior to the alcoholic-potassium hydroxide method (Khan—*J. Am. Oil Chemists' Soc.* 30, 355). 9(10)-Keto-stearic acid is obtained by dechlorination of methyl alkoxychlorostearates with alcoholic-potassium hydroxide followed by acidification to decompose soaps and to hydrolyze the intermediate vinyl ether (Gast *et al.*—*J. Am. Chem. Soc.* 74, 6280). Products useful as plasticizers, and intermediates in the preparation of amino acids or lactones are prepared from fatty acids containing halogen in the α -position and in at least one other position by selectively hydrogenating to replace only the α -halogen (Ladd & Sargent—*U. S.* 2,655,521). New patented methods for halogenation of fatty materials make use of chlorine gas in presence of alkali (Kobayashi & Miyazaki—*Japan* 2680 [1952]) and mixtures of chlorine gas with catalyst such as Cl_2S , SCl_2 , SO_2Cl_2 , SOCl_2 , etc. (Morghen & Chalupny—*Austrian* 173,230). For biological studies fat was labeled with radioactive iodine by halogenation in organic solvent (Hoffman—*J. Lab. & Clin. Med.* 4, 521). Reviews were written on halogenation (Pierce & McBee—*Ind. Eng. Chem.* 45, 1969) and on reactions of fatty acid chlorides (Sonntag—*Chem. Revs.* 52, 237).

Commercial methods of reduction of fats to fatty alcohols were reviewed by de Bock (*Tech. Wetenschap. Tijdschr.* 22, 99). In procedures published for the sodium reduction method use of butanol (de Ursinos & Saenz—*Grasas y aceites* 3, 180) and amyl alcohol (Soc. anon. *d'innovations chim. S. ou Sadie—Fr.* 973,161) as the reducing alcohols, and excess alcohol for breaking emulsions (Binka & Peddicord—*U. S.* 2,647,932) are emphasized. The articles on higher alcohol production by high pressure hydrogenation of fats give details for processes using the following catalysts: copper-chromium-barium and copper-chromium-zinc mixtures (Kiryu & Muto—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 51, 81), copper-chromite (Nodzu *et al.*—*Ibid.* 55, 78), cupric salts (Kunz & Fiore—*U. S.* 2,590,105), and mixtures of zinc and other metallic oxides (Kobashi *et al.*—*J. Chem. Soc. Japan Ind. Chem. Sect.* 54, 145). Fatty alcohols are also produced by hydrogenating lead soaps (Komori *et al.*—*Ibid.* 55, 103, 237) and by reduction of fat with hydrogen developed from the action of sodium or powder aluminum on lower alcohols (chim.tech. union chim. nord et Rhone—*Fr.* 978,994-5). A report on conversion of the N-methyl anilides of organic acids to good yields of aldehydes by reduction with lithium aluminum hydrides covers several fatty acids (Weygaud—*Angew. Chem.* 65, 525).

An aluminum chloride catalyzed condensation of glycerides with benzene to give aralkyl ketones is reported as a new reaction of glycerides (Silverstein *et al.*—*J. Am. Oil Chemists' Soc.* 30, 350). Japanese acid clays were used to catalyze the addition of naphthene to fatty acids (Kuwata—*J. Chem. Soc. Japan Ind. Chem. Sect.* 54, 101).

Werner-type complexes were prepared from fatty acids and chromic chloride for use in water proofing paper (Pavlin—*Tappi* 36, No. 7, 107A). Complexes prepared from fatty acids and copper and cobalt acetates are analyzed with regard to properties and structures (Kaufmann & Lüssling—*Fette u. Seifen* 55, 90). A covalent chelate structure proposed for copper laurate is based on the properties of its co-ordination compounds with ammonia and pyridine (Gilmour & Pink—*J. Chem. Soc.* 1953, 2198). A patented oil soluble cobalt oleate is intended for intramuscular injection in therapeutics (Klement—*Ger.* 812,575). Derivatives of fatty acids containing sulfur and gold were synthesized to study their physiological activity and toxicity (Kundu—*J. Indian Chem. Soc.* 29, 523). Reaction products of polytitanic acids with fatty alcohols and acids are prepared for use as water-repellent agents, paper and textile sizes, and surface active agents for use in organic solvents (Boyd—*U. S.* 2,614,112; Balthis—*U. S.* 2,621,194; Haslam—*U. S.* 2,621,195). Several silicon-containing fatty acids were synthesized and described (Bunnell & Shirley—*J. Org. Chem.* 17, 1545).

Secondary amines with one long and one short fatty chain were prepared for utilization in manufacture of wetting agents, insecticides, waterproofing agents, etc. (duBrow & Harwood—*U. S.* 2,627,526). Electrolysis of fatty nitriles in ammonium chloride solution yields the corresponding amine (Janardhan—*J. Sci. Ind. Res., India* 12B, 183). The physical properties of films of α -aminolauric acid polymers of 172,000 mole weight in dilute acid, base, and salt solutions have been recorded (Eda & Masuda—*Bull. Chem. Soc. Japan* 24, 140). Fatty acid amides of secondary amines can be oxylated with polyoxyethylene adducts to yield useful emulsifiers (DeGroot—*U. S.* 2,652,409). Polyamine materials ranging from hard waxlike substances to viscous oily liquids are prepared by reacting mixtures containing fatty acids, acrolein, and ammonia (Harman & Sommer—*U. S.* 2,640,822). Decomposition of hydrogenated sardine oil by liquid ammonia yields fatty amides which are useful as substitutes for stearic acid in rubber manufacture (Tengasa—*J. Soc. Rubber Ind. Japan* 23, 276). Fatty polyamides can be used as gelling agents for making incendiary bombs, flame throwing compounds, etc. (Floyd—*U. S.* 2,662,068). Silica gel may be used as a catalyst for such amide manufacturing processes (Yoshizaki—*J. Chem. Soc. Japan Ind. Chem. Sect.* 54, 186). Acetonitrile, propionitrile, benzonitrile, and other nitriles will add to the double bond of oleic acid in the presence of sulfuric acid to yield the corresponding substituted amidostearic acids (Ree & Swern—*J. Am. Chem. Soc.* 75, 5479). Condensation of 9,10-dibromostearic acid and 2-bromostearic acid, respectively, with thiourea in butanol yields dithiouronium salt and pseudothiohydantoin (Suzuki & Oda—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 55, 178).

FAT IN THE METAL, LEATHER, WAX, AND WATERPROOFING INDUSTRIES. Replacements for palm oil in cold-rolling steel (Williams *et al.*—*Iron Steel Eng.* 30, No. 8, 65) and in hot-dip tinning (Fochtman *et al.*—*U. S. Dept. Agr. Bur. Agr. & Ind. Chem. Mimeo. Circ. Ser. AIC354*, 14 pp.) are made from better grades of animal fats by hydrogenating to iodine values

of 48-54 and adding antioxidants. Polymerization technique for the preparation of core binders from fish oils is described Michalowska & Kossakowski—*Przemysł Chem.* 31, [8], 360).

The flotation collecting effects of palmitic, oleic, linoleic, linolenic, and tall oil acids as determined on ilmenite, magnetite, pyrite, hornblende, hematite, and rutile show they are efficient for the metal oxides but poor for silicates (Hukki & Vartiainen—*Suomen Kemistilehti* 25B, No. 11, 65; *Trans. Am. Inst. Mining Met. Eng.* 196, *Tech. Pub. No. 3542-B*). Tests to associate efficiency of oleic acid during flotation on oxidizability of the double bond did not confirm this alleged hypothesis (Gaudin & Cole—*Mining Eng.* 5, No. 4, 418).

New rust inhibitor coatings for metals contain: a heated mixture of fatty acids, sulfur and lead oxide in thinner (Cophthorne—*U. S.* 2,609,302), a mixture of α -hydroxylauric, its lactide and self esterified derivative, mineral oil, and calcium mahogany sulfate (Kleinholz—*U. S.* 2,610,919), and a paraffin-wax-silicone-lead or calcium soap mixture (Romberg—*U. S.* 2,627,474). Corrosion of ferrous metals by hydrogen sulfide in oil wells is inhibited by adding alkylamines made from vegetable oils to the well fluids (Lytle—*U. S.* 2,614,980). The rusting of metals in contact with gasoline, naphthas, and boring oils can be inhibited by adding dimeric fatty acid to these fluids (Landis & Backensto—*U. S.* 2,632,695). Lanolin films for the protection of mild steel should be made from light mineral oils solutions so as to have 0.004-0.006 oz. of lanolin per sq. ft. of film (Stroud & Rhoades-Brown—*J. Applied Chem.* 3, 287).

Fat derived brake fluids are rice oil slightly oxidized with hydrogen peroxide (Yukida—*Japan* 1569 [1951]), or with potassium permanganate (Koyama—*Japan* 2325 [1951]), and condensation products of ethylene oxide and tall oil (Esposito—*U. S.* 2,610,966).

Special mixtures of aluminum stearate and sulfated fatty alcohols were prepared for waterproofing textiles, cement, etc. (Cunder & Licata—*U. S.* 2,660,568). Waterproofing of base materials was also done by preparing water-insoluble soaps *in situ* (Lolkema—*U. S.* 2,657,155). A wax-like coating for the same purpose was an emulsion of paraffin, stearic acid, triethanolamine, and water (Figdor—*U. S.* 2,635,055). A review on making fibers water-repellent and for determining water-repellency was written by Serehi & Chini (*Chimica, Milan*, 7, 194, 233).

A new wax composition for candles contains paraffin and a eutectic mixture of palmitic, stearic and oleic acids (Thompson *et al.*—*U. S.* 2,638,411). An aqueous wax polish was an emulsion of waxes, fatty acids, and resin acids completely saponified with ammonia (Tüchy—*Austrian* 173,522). A cream for protecting hands comprised a solution of stearic acid, lanolin or beeswax, glycerol, casein ammonia, and water (Bevilacqua & Porro—*Ital.* 467,137).

COATINGS, RESINS, AND PLASTICS. The review and general information articles dealing with drying oils and fatty resins were on: fat science and surface coating research (Scheiber—*Fette u. Seifen* 55, 81), symposium on oxidation of fats and oils (Bawn—*Nature* 171, 1057), mechanisms of combination of oxygen with fatty acids (Meier—*Chem.-Ztg.* 76, 218), oxidation, dimerization, etc. (Skellon—*Chemistry & Industry* 1953, 1047), rates of oxidation of fatty acids (Toyama—*Separate. Review of Toyama and coworkers papers*; Maruta—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 55, 303), kinetics of polymerization (Berger—*Bull. soc. chim. France* 1953, 511; Ashar & Champetier—*Compt. rend.* 234, 1555), theories of heat polymerization and oxidation of oils (Balbi—*Olearia* 6, 335), reactions and properties of oils and resins in varnish cooking (de Wilde—*Finska Kemistsamfundets Medd.* 66, 81), factors affecting the heat thickening of linseed oil (Seavell & Sleightholme—*Offic. Dig. Federation Paint & Varnish Prod. Clubs.* 334, 789; *J. Oil & Colour Chemists' Assoc.* 36, 119), polymerization of oleic acid in the presence of boron fluoride and phosphoric acids (Topchiev & Vishnyakova—*J. Gen. Chem. U.S.S.R.* 21, 1775), isomerization of oils (von Mikusch—*Schweiz. ver. Lack- u. Farben-Chem. u. Tech. Bull.* No. 20, 1; Balbi—*Olearia* 7, 125; Narayan & Kulkarni—*Paintindia* 2, No. 5, 15), reactions of conjugated double bonds and diene synthesis (Kaufmann—*Grasas y aceites* 3, 63), methods of improving drying oils (Terrill—*Offic. Dig. Federation Paint & Varnish Prod. Clubs* 344, 536), metallic soap driers (Oddifreddi—*Ind. vernice, Milan*, 6, 184, 211, 229), tall oil utilization in the paint industry (Ijak—*Chim. peintures* 15, 372), dehydroxylated oils (Terrill—*Paint, Oil Chem. Rev.* 116, No. 4, 13; Bryson—*Ibid.* No. 3, 12; da Rosa—*Olearia* 5, 232; Balbi—*Ibid.* 7, 15; Sivsamban *et al.*—*Proc. Sym. Indian Oils Fats Natl. Chem. Lab.* 1951, 197), drying properties of mannitol and sorbitol esters of sardine oil fatty acids (Shimo & Kamei—*Sci. Repts. Res.*

Insts. Tohoku Univ. 3A, 234), vegetable oils in floor coverings (O'Hare—*Seifen-Öle-Fette-Wachse* 78, 541), influence of various oils on the properties of alkyl resins (Stieger—*Fette u. Seifen* 54, 639; Prakash—*Proc. Sym. Indian Oils, Fats, Natl. Chem. Lab.* 1951, 217), advantages of fatty acids over whole oils in alkyls (Reigler—*Paint Varnish Production* 43, No. 7, 26), gelation points of alkyl resin-fatty acid combinations (Imoto & Horiuchi—*Chem. High Polymers, Japan* 8, 198, 205), synthesis and properties of vinyl esters of fatty acids and their polymers (Asahara & Tomita—*J. Oil Chem. Soc., Japan*, 1, 76), wrinkle finishes from tobacco seed oil alkyls (Kapur & Joshi—*Paint Manuf.* 23, 185), copolymerized oils and resins (Daniels, Jr.—*Offic. Dig. Federation Paint & Varnish Prod. Clubs* 332, 611), analytical methods applied to coatings, polymers, and plasticizers (Stafford & Shay—*Anal. Chem.* 25, 8), apparatus for determining gelation time of tung oil samples (Pack—*A.S.T.M. Bull.* 191, 49), and lacquer testing for drying and porosity (Gorback & Bukowiecki—*Fette u. Seifen* 55, 511).

Reactions that occur during polymerization of oils were studied by several groups of investigators. Privett *et al.* (*J. Am. Oil Chemists' Soc.* 30, 61) and Sephton & Sutton (*Chemistry & Industry* 1953, 667) reported that during the autoxidation of methyl linoleate more trans-, trans- isomer forms at 23-4° than at 0°. Williamson (*J. Applied Chem.* 3, 301) and Chang & Kummerow (*J. Am. Oil Chemists' Soc.* 30, 403) propose mechanisms for the oxygenated polymerization based on linkages occurring between carbon atoms which present evidence supports. Monocarboxylic acids of 30 and 32 carbon atoms, some with two double bonds were isolated from polymerized linseed oil (Petit—*Compt. rend.* 234, 1690). Their formation is attributed to cracking of dimer acids which are formed during the polymerization or combination of monoacids with hydrocarbon fission products. When oleate hydroperoxide is thermally decomposed at 100° it gives an oxygen bonded dimer containing two double bonds, plus small amounts of hydroxy unsaturated monomers and, possibly, undecanal aldehyde (Williamson—*J. Oil & Colour Chemists' Assoc.* 34, No. 369, 119). In the polymerization of tung oil below 240°, the α -eleostearic acid is slowly converted to the β -acid; above this temperature the isomerization takes place instantly (van Loon—*Verfkronek* 26, 61). However, a tung oil artificially isomerized into β -form bodies as low as 170°.

Lips *et al.* (*J. Am. Oil Chemists' Soc.* 30, 315) submitted linseed, weedseed screening, and rapeseed oils to deodorization-polymerization at 280° for up to 20 hours and recorded the changes in iodine value, color, viscosity, and mean molecular weight. Another study of rates of autoxidation shows that although the more unsaturated acids have the higher rates in autoxidation, the reverse seems to be the case in oxidation with benzoyl peroxide (Toyama & Yamamoto—*J. Chem. Soc. Japan Ind. Chem. Sect.* 55, 176).

In a polemic note Sutton (*J. Am. Oil Chemists' Soc.* 30, 168) points out that it is unlikely that film formation necessitates a domination of intrapolymerization over intermolecular reaction. Harrison & Wheeler (*Minnesota Chemist* 4, No. 5, 7) interpreted a lack of spectral absorption maximum at 230.5 μ in linoleate dimers as rendering the free radical theory of dimerization highly questionable.

Kaufmann & Korfhage (*Fette u. Seifen* 55, 281) studies on acceleration of drying by free radicals such as triphenyl methyl, diphenyl (*p*-biphenylmethyl), etc., indicate that these react with natural antioxidant thus permitting rapid autoxidation. Other reports from the same laboratory demonstrate that natural phospholipides slow autoxidation or drying (Kaufmann & Schmidt—*Ibid.* 54, 346, 399), and that the prooxidant effect of carotene and vitamin A is abolished by presence of tocopherol (Kaufmann & Gulinsky—*Ibid.* 55, 593). An abnormally long drying time of a sample of linseed oil is attributed to the presence of acidic phosphatides which appear to inhibit both oxidation and polymerization (Desnuelle & Massoni—*Peintures, pigments vernis* 29, 390). Tung oils from *Aleurites montana* have longer gelation times and are more seriously affected by free fatty acids than are oils from *A. fordii* (Cutting—*J. Sci. Food Agr.* 3, 510). Blowing tung oil with air at room temperature causes practically no change in refractive index whereas at higher temperature the change is rapid (Fritz—*Deut. Farben-Z.* 7, 90). Polymerization of drying oils in an atmosphere of hydrogen at room temperature causes polymerization without cyclization whereas at 300° both occur (Boelhouwer *et al.*—*Rev. trav. chim.* 72, 716). Gels produced by oxidizing linseed oils at low temperatures (below 90°) are very suitable for linoleum manufacture (Walker & Mackay—*J. Appl. Chem., London*, 2, 344). Tung oil films dried in the light of a carbon-arc lamp become frosted where temperature

is over 40°, and also in the cold when cobalt linoleate drier is present (Fritz—*Farbe u. Lack* 59, 228).

Break-free linseed oil samples were isomerized to various degrees. Physical and chemical characteristics of these oils and films formed with and without driers were studied (v. Mikusch & Mebes—*Ibid.* 223). Since the films had very little tendency to check or crack and had outstanding weather resistance it is suggested that brittleness in tung oil films is not the result of isomerization. Tests with reduced nickel and nickel in combination with several other metals as isomerization catalyst for grapeseed oil indicate that nickel-cobalt catalyst is most efficient (Rigamonti—*Olearia* 6, 139). Newly patented catalysts for the process are reduced nickel containing some sulfur (Sana, narodni podnik—*Austrian* 170,613; N. V. Mij, tot Exploitatie Vereenide Oliefabrieken Zwijndrecht—*Dutch* 71,883) and phosphotungstic acid (Vorlamov & Slozina—*U.S.S.R.* 77,549). A study of polymerization of oils with sulfur dioxide catalyst demonstrates that it catalyzes isomerization of double bonds whereas oxygen activates the double bonds (Boelhower—*Chem. Weekblad* 49, 197).

Saletore *et al.* (*Proc. Sym. Indian Oils Fats Natl. Chem. Lab. India* 1951, 202) illustrated the advantages of using driers and benzoyl peroxide in production of linseed oil films with data on the drying rate and acetone solubility of the dried film. The trans forms of uranium, thorium, cerium, and cobalt oleates are less active prooxidants initially, but in overall oxidation the cis-forms are less active (Skellon & Spence—*J. Appl. Chem.* 3, 10). Organic compounds of orthotitanic and metatitanic acids have been patented as oil gelation accelerators (Boyd & Green—*U. S.* 2,620,318). Benzofuroxan and benzofurazan are added to drying oils to act as antiskinning agents (Rosenwald—*U. S.* 2,630,438). Use of the mixed naphthenates of cerium, lanthanum, praseodymium and neodymium as a substitute for cobalt drier permits production of baked films of improved marproofness, toughness and color (Gardner—*Paint Varnish Production* 43, No. 3, 38). Baked wrinkle finishes are made from benzene solutions of condensed mixtures of phthalic anhydride, glycerol, and fatty acids of tobacco seed oil (Council Sci. & Ind. Res., India—*Indian* 43,979).

Many methods were studied for converting nondrying oils to drying oils or improving the drying properties of semidrying oils. A continuous system for converting castor oil to a drying oil by dehydration consists of passing it with one percent sulfuric acid over a series of tubes heated to 300° and at six millimeters pressure for 2-10 seconds (Mouchiroud—*Fr.* 873,750). Other articles on dehydration of castor oil pertain to the use of catalysts such as oil-maleic anhydride compounds (Grummitt & Marsh—*J. Am. Oil Chemists' Soc.* 30, 21), emulsion of sulfuric acid (Moser—*Brit.* 691,484), aluminum, zinc or calcium chloride (Bertraut & Kahneman—*Fr.* 975,050), and a mixture of sodium bisulfite and sodium bisulfate (Sivasamban *et al.*—*Indian* 46,457). The methods described for processing tall oil for use in film-forming compositions include: conversion to calcium soaps and mixing with drying oils (Mahler—*U. S.* 2,616,814); condensation with an α,β -unsaturated polycarboxylic acid, then with a polyamino compound and finally with formaldehyde (Schmutzler—*U. S.* 2,640,814); esterification with either glycerol, pentaerythritol, sorbitol-pentaerythritol, polyallyl alcohol, or an epichlorohydrin-2,2-bis-(*p*-hydroxyphenyl) propane (Tess & May—*Offic. Dig. Federation Paint Varnish Production Clubs* No. 311, 1114); and selective esterification with pentaerythritol (Kirjakka & Turunen—*Teknillisen Kemian Aikakauslehti* 9, 161). Drying oils are prepared by removal of saturated fatty acids from semidrying oil fatty acids by urea precipitation and esterifying the unsaturated fraction with pentaerythritol (Asahara *et al.*—*J. Oil Chemists' Soc. Japan*, 1, 175). Another method involves condensation of the oils with cyclic polycarboxylic acids (Elwell—*U. S.* 2,623,056). Still residues from distillation of the polymerized acids of fish or rice oils are mixed with shark liver oil and esterified with glycerol to yield a rapid drying oil (Katsura—*Japan* 3737 [51]). The fatty acids of linseed oil esterified with mannitol yields an oil that dries faster than linseed oil (Maruto—*Japan* 3763 [52]; Takahashi & Takeshita—*J. Chem. Soc. Japan Ind. Chem. Sect.* 55, 281). The ability of drying constituents segregated from pilchard oils by solvents to give good varnishes has been demonstrated (Chatfield—*Paint Oil & Colour J.* 123, 27). Drying constituents are concentrated from fatty acid mixtures by precipitation of non-drying material with urea (Knafo—*Bull. mens. ITERG.* 7, 114).

Rice oil is dewaxed and heated with benzoyl peroxide for paint use (Koyama—*Japan* 3033 [51]). Stand oil prepared from cameline oil is particularly suitable for manufacture of rust-inhibiting paints (v. Mikusch—*Deut. Farben Z.* 6, 391,

392). Kamala oil is mixed with other semidrying oils (Forest Res. Inst. and Colleges—*Indian* 46,216; 46,339) or is extracted from the seeds with another oil as the solvent (*Ibid.*—*Indian* 46,338) to serve as a paint oil. Chrysalis oil is converted to a quick drying oil by heating with manganese dioxide and manganese soap of linseed oil (Nakajima & Kosuge—*Japan* 4733 [52]), and to a factice by vulcanization in the presence of thiazole or guanidine (Maruta & Iwama—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 679; 55, 127).

The manufacture of synthetic drying oils that form suitable dried films from commercial stearic acid by chlorination and dechlorination has been demonstrated (Wekua & Bergmann—*Farbe u. Lack* 59, 267). Reaction with di-tert-butyl peroxide can also be used and the dehydropolymers made from methyl stearate and linoleate in this manner are described (Clingman & Sutton—*J. Am. Oil Chemists' Soc.* 30, 53). Improved drying oils are also prepared by reaction of unsaturated oils with cyclopentadiene and terpene hydrocarbons (Gerhart—*U. S.* 2,630,415), with nonaromatic cyclic hydrocarbons (Geiser—*U. S.* 2,624,717), with acyclic monoolefins or their polymers (Verley—*U. S.* 2,623,890), and with decarboxylated cashew-nutshell liquid (Harvey—*U. S.* 2,637,709).

Products ranging from improved drying oils to resins solids suitable for protective coating uses are made by copolymerizing fatty materials with: vinyl compounds (Port *et al.*—*J. Polym. Sci.* 9, 493; Schertz—*U. S.* 2,601,561; Griess & Teot—*U. S.* 2,639,772; Evans *et al.*—*U. S.* 2,647,376; *Brit.* 691,042; Kanning & Hart—*U. S.* 2,655,488), styrene (Harrison & Tolberg—*J. Am. Oil Chemists' Soc.* 30, 114; Boelhower *et al.*—*Chemistry & Industry* 1953, 1287; Wakeford & Hewitt—*U. S.* 2,607,749; Cass—*U. S.* 2,609,349; Rubens & Boyer—*U. S.* 2,609,353; Block & Geiser—*U. S.* 2,616,863; L. Berger & Sons, Ltd.—*Brit.* 675,761, 688,755; *Brit. Resin Products, Ltd.*—*Brit.* 680,441; Dow Chem. Co.—*Brit.* 679,143-4), allyl and polyallyl alcohols (Cox *et al.*—*J. Am. Oil Chemists' Soc.* 30, 360; Gould—*U. S.* 2,639,273), ethylene (Block—*U. S.* 2,611,788), and various unsaturated hydrocarbons (Geiser—*U. S.* 2,624,717).

The new information on alkyl resins pertains to: their thermal behavior (Skraup & Hedler—*Chem. Ber.* 85, 1161), production from pentaerythritol and degummed soybean oil (Marling & Hempel—*U. S.* 2,637,708), products containing silicone (Hatcher & Bunnell—*U. S.* 2,624,720), use of tetrachlorophthalic anhydride in their manufacture (Mehta—*Proc. Sym. Indian Oils Fats Natl. Chem. Lab. India* 1951, 210; Keyl & Geils—*U. S.* 2,655,486-7), manufacture from free fatty acids, polyhydric alcohols and polycarboxylic acids (Cadwell & Frazier—*U. S.* 2,618,617), modification with an aromatic monocarboxylic acid (Tess & Mika—*U. S.* 2,618,616; N. V. Bataafsche Petrol. Maatschappij—*Dutch* 71,805), and modification with methyl methacrylate polymers (Opp & Werner—*U. S.* 2,647,093).

Thermoplastic masses are prepared by condensing fatty acids, phenols, and formaldehyde (Smith *et al.*—*U. S.* 2,632,745). Adhesive compositions comprising ricinoleate esters, phenol-formaldehyde resins, and polyvinyl acetal resins were patented (Bergstedt & Lamason—*U. S.* 2,637,706). Flexible, elastic polymers, suitable for coating paper, textiles, and metal products are prepared by the condensation of polymerized unsaturated fatty amines with polyunsaturated fatty acids (Ralston *et al.*—*U. S.* 2,617,813). A new sealing compound contains rubbery polymerized vegetable oil, resin, and filler (Fischer & Walker—*U. S.* 2,625,522). Rice oil is converted to a rubber-like product by treatment with sulfur tetrachloride and heating with trichloroethylene (Koyama—*Japan* 3039 [51]).

The fatty derived plasticizers recently demonstrated are laurates and pelargonates of lactic acid esters (Fein *et al.*—*Indian Rubber World* 126, 783) and alkoxyhydroxystearates (Swern—*U. S.* 2,624,680).

Tin ricinoleate (Furter—*Kunststoffe* 43, 189) and a combination of disodium phosphate dodecahydrate with bodied vegetable oil (Grummitt *et al.*—*U. S.* 2,654,718) stabilizes polyvinyl resins against destructive effects of heat and light.

A method of determining polymerized fatty acids in mixtures is based on precipitating nonpolymer components with urea (Catravas & Knafo—*Oleagineux* 8, 139). The acetone tolerance test is recommended for evaluating the performance of bodied oils (Rheineck—*Offic. Dig. Federation Paint & Varnish Production Clubs* No. 340, 281). The water and vapor permeability of thin unsupported films of pure drying oils, pentaerythritol esters, and alkyds has been recorded (Harris & Bialecki—*Am. Paint J.* 37, No. 12, 68). In all cases, the more liquid like the surface, the greater is the permeability. A blue tarnish defect of coating films is discussed in relation to oil, nonpolymerized components, phosphatides, solvents, driers, moisture, etc. (Kaufmann—*Fette u. Seifen* 55, 681).

FATTY MATERIALS IN LUBRICATION, INSOLUBLE SOAPS, TEXTILE OILING, AND METAL WORKING. Articles containing general information on lubrication were on: present problems and future trends in lubrication (Lisman—*Ind. Eng. Chem.* 45, 1406), lubrication for engines and industrial equipment (Davis—*Petroleum*, London 16, 183), manufacture and properties of lithium greases (Meyer, Jr.—*Petroleum Engr.* 25, No. 5, C15), pour point depression of lubricating oils (Gavlin—*Ind. Eng. Chem.* 45, 2327), and comparison of the serviceability of grease consistometers (Göttner & Schultze—*Erdöl u. Kohle* 6, 200).

The literature on the properties of metallic soaps principally concern the manufacture of lubricant greases. Preliminary x-ray studies of soap structures in grease-like systems showed that calcium stearate hydrate orientated to the same extent in fluids ranging in viscosity from cetene to vaseline; but of six commercial greases two had no orientation whereas the remaining displayed varying degrees of orientation (Vold—*Inst. Spokesman* 16, No. 8, 8). In these the major plane of orientation is aligned parallel to the surface of the ribbon-like particles (Vold—*J. Phys. Chem.* 57, 26). Mathews (*J. Inst. Petroleum* 39, 265) suggests that the soap crystal phase can be controlled during fiber growth in the manufacture of lithium grease so as to control the length-diameter ratio of fibers, and hence control the properties of the grease. The oleogels of naphthenic acid aluminum soaps of pH 7 have a maximum viscosity when the precipitation temperature is 60°; with pH 5 the best temperature is 80°; heat used in drying also affects the viscosity of the gels (Trapeznikov & Belugina—*Doklady Akad. Nauk. S.S.S.R.* 37, 825). A study of soap greases in a torsional coaxial-cylinder strain-recording elastometer has indicated transitions from elastic deformation to viscous flow and these were discussed with regards to grease making (Vinogradov & Gvozdev—*Ibid.* 86, 341). The effect of shear, particle shape, composition, and temperature on the thixotropy of sodium stearate grease was determined (Moses et al.—*Can. J. Technol.* 31, 121). An increase in osmotic pressure of aluminum stearate in benzene with increase in temperatures is interpreted to indicate that the association of this soap is due to hydrogen bonding (Inoue & Iida—*J. Chem. Soc. Japan, Pure Chem. Sect.* 74, 145). The observation that aluminum monochlorodilaurate does not thicken hydrocarbons, whereas the dilaurate hydroxide does also may be interpreted to indicate that association is due to hydrogen bonding (Mysels & May—*J. Am. Chem. Soc.* 75, 1750). The maximum amount of lowering of surface activity attainable in calcium soaps is not affected by the amount of unsaturation; but the maximum temperature of activity is related to the melting point of the fatty acid of the soap (Kaufmann & Rackers—*Fette u. Seifen* 55, 497). If the oil portion of a grease is displaced, at a temperature below the first transition point of the soap, by liquid butylene, the latter by ethylene, and the ethylene released, the residual porous soap skeleton has the dimension of the original grease gel and can be studied by electron micrographs (Peterson & Bondi—*J. Phys. Chem.* 57, 30). Sato (*J. Oil Chemists' Soc., Japan*, 1, 119) demonstrated the existing equilibrium relations between concentration of soaps and polar compounds of gels in spindle oil and discussed these equilibria with regard to intermolecular attraction of the soaps and their dipole moments. Metallic ricinoleates in contrast to stearates are more soluble in organic liquids and give clear hard gel structures with limited swelling (Patton & Lindlaw—*J. Am. Oil Chemists' Soc.* 30, 331).

Pure aluminum tri-soaps are made for fundamental studies by refluxing aluminum isopropoxide with stearic acid in dry benzene and evaporating the solvent under reduced pressure (Mehrotra—*Nature* 172, 74). Water-insoluble metal soaps for general use as medical, pharmaceutical, lubricating, etc., are dissolved in liquid hydrocarbons containing a little alkali (Woudhuysen & Bajol—*Fr.* 890,508). Aluminum soap made from mixtures of normal fatty, dimerized linoleic acid, and naphthenic acid are intended for manufacturing gel hydrocarbon fuels (Dean—*U. S.* 2,620,345). Among various metallic soaps, the stearates are superior as gelling agents for preparation of plastigels (Parker & Tritsch—*Modern Plastics* 30, No. 6, 129). Metallic soaps are rendered water-dispersible for special uses by admixture with a fatty amide (Cunder & Licata—*U. S.* 2,660,567-8). Soaps for use as paint driers or in lubricants are prepared by adding molten fatty acids to an aqueous suspension of metal hydroxides containing a small amount of an alkyloamine (Kebrieh & Pitrot—*U. S.* 2,650,932). Aluminum soap prepared from tall oil and coconut fatty acids is said to be suitable for making grease, coatings, gelled fuels, etc. (Minich & Nowak—*U. S.* 2,618,596).

A continuous method of making lithium greases is based on blending in a votator at 375° F., chilling in a second votator

unit, and milling (Baker & Joyner—*Inst. Spokesman* 17, No. 3, 18). A special apparatus has been designed for deaerating and milling greases (Morehouse—*U. S.* 2,660,259).

A new method of manufacturing comprises adding a small amount of petroleum oil and beeswax to the metal soap, dehydrating, grinding, and then dissolving in sufficient mineral oil to give a gel of grease consistency (Orno-Ornfeldt—*U. S.* 2,658,037). New grease gels are also manufactured by saponification of the soap ingredient *in situ* in the presence of emulsifiers (Lieve—*U. S.* 2,629,692; Faust—*U. S.* 2,659,695). Another method of getting good soap gels is to prepare the soap in a solvent conducive to formation of good gels, remove the solvent, and add the desired petroleum oil (Browning—*U. S.* 2,616,850; Browning & Webb—*U. S.* 2,636,001; N. V. Bataafsche Petrol, Maatschappij—*Dutch* 71,729). The innovation in several patented manufacturing processes deal with procedures for tempering, chilling, and milling to induce suitable physical structures of the soap, consistency of the grease, and stability of the consistency (Matthews et al.—*U. S.* 2,629,695; O'Halloran—*U. S.* 2,646,401; Moore—*U. S.* 2,648,634; Bryant et al.—*U. S.* 2,651,616).

Two new greases are thickened with specific ratios of, respectively, lithium and calcium soaps (O'Halloran—*U. S.* 2,641,577) and lithium and potassium soaps (Whitney—*U. S.* 2,623,017). Greases of butter consistency with a high drop point are made with mixed sodium soaps of oxidized petroleum, oxidized fish oil, naphthenic, and fatty acids (Dilworth et al.—*U. S.* 2,626,896, 2,626,898; McKinley et al.—*U. S.* 2,637,695; N. V. Bataafsche Petrol. Maatschappij—*Dutch* 71,739). Improved physical structure is obtained by including some soaps of branched chained acids (Morway & Smith, Jr.—*U. S.* 2,612,472-3, 2,623,354; Allison & Blalock—*U. S.* 2,628,195-202; Young & Zaborski—*U. S.* 2,626,897). Greases of improved shear stability are made from specific petroleum oils and mixed sodium and lithium or aluminum soaps (Ford—*U. S.* 2,619,461), and from lithium hydroxystearic acid and a soap of a fatty acid having a branched radical at the 9-position (Moore & Saarni—*U. S.* 2,614,076). Greases are also manufactured by thickening petroleum oils with certain fat derived plastic or resinous compounds (Dilworth & Culnane—*U. S.* 2,639,266; Morway et al.—*U. S.* 2,642,397; Matthews & Grimshaw—*U. S.* 2,647,873), or with silica type gels (Stoss—*U. S.* 2,625,508; Sparks & Morway—*U. S.* 2,626,241; Abrams & Stross—*U. S.* 2,626,899; Bryant—*U. S.* 2,640,812; Peterson—*U. S.* 2,647,872; Eckert—*U. S.* 2,656,315; Stross et al.—*U. S.* 2,658,869). Polysiloxanes have been substituted for petroleum oil in aluminum soap base greases (Hotten—*U. S.* 2,654,710).

Greases stable to high temperatures are prepared from special gels made from soaps of zinc, aluminum, beryllium and some rare earths (Passelecq—*Fr.* 889,619). High temperature stability is also induced by adding thio ether acids (Morway & Smith—*U. S.* 2,618,598), and a combination of sodium myristate and N,N'-diphenyl *p*-phenylene diamine (Eckert—*U. S.* 2,663,690).

Special working, tempering, and chilling conditions are used to induce resistance to bleeding in greases (Moore—*U. S.* 2,625,510, 2,648,634; Matthews et al.—*U. S.* 2,629,695). An additive made by condensing fatty acids with an α -hydroxy fatty acid is used for the same purpose (Whitney—*U. S.* 2,628,938).

Resistance to water uptake or emulsification with water is induced in greases with additives such as guanidine carbonate (McLeod—*U. S.* 2,620,301), alkali salt of sulfonated castor oil (Beretvas—*U. S.* 2,640,811), salts of hydroxy acids with a maximum of five carbon atoms (Lamel—*Austrian* 173,327), and various amino compounds (Peterson—*U. S.* 2,623,852, 2,629,691; King & Pattenden—*U. S.* 2,618,599). Greases made from soaps of 12-ketostearic acids are resistant to both water and high temperatures (Sproule et al.—*U. S.* 2,613,182).

The corrosion inhibiting additives for both greases and lubricant oils, derived from fats, are: phosphorus pentasulfide treated fatty amides (Bartleson—*U. S.* 2,614,075), orthotitanates of fatty alcohols (Laugkammerer—*U. S.* 2,621,193), fatty alcohol esters of thiodiacetic acid (Richter & Fuller—*U. S.* 2,649,416), fatty mercaptides (Rudel & Gargisa—*U. S.* 2,644,793; Jones & Smith—*U. S.* 2,627,511), dithiophosphoric acid esters of fatty alcohols (*Am. Cyanamid Co.—Brit.* 672,663). Polymerized free fatty acids of linseed oil (McDermott—*U. S.* 2,631,979), and fatty mono- and diglycerides improve lubricant viscosities (Vitex S. A.—*Fr.* 878,418).

The fat derivatives patented for depressing the pour point of lubricant oils are: copolymer of maleic anhydride and fatty alcohols (Lippincott & Mikeska—*U. S.* 2,615,845), copolymers of alkyl aryl compounds with maleic anhydride esterified with

fatty alcohols or fatty amines (Giammaria—*U. S.* 2,615,843, 2,616,853; Bartlett—*U. S.* 2,612,475), polymers of vinyl acetate and fatty alcohol esters of sorbic acid (Bartlett—*U. S.* 2,637,697), condensation products of ethylene oxide and fatty mercaptans (Wolf—*U. S.* 2,619,466), product prepared by heating stearic acid at 500-700° F. for 4-10 hours in the presence of steel wool (Lieber & Cashman—*U. S.* 2,633,471).

Condensation products of unsaturated fatty oils with aromatic radicals increase the viscosity and oiliness, and decrease the pour point of mineral oil lubricants (Champagnat—*Fr.* 893,202).

The new lubricant additives used to increase extreme-pressure characteristics are: boron trifluoride derivatives of animal or vegetable oils (Kipp—*U. S.* 2,611,742), phosphorous pentachloride treated partial esters of polyhydric alcohols and fatty acids (Matuszak & Weisel—*U. S.* 2,643,261), sulfurized fats and waxes (Cyphers & Waddey—*U. S.* 2,628,940; Sunde—*U. S.* 2,629,721; Fischl & Hill—*U. S.* 2,640,030), and metal sulfides dispersed with sodium soap (White *et al.*—*U. S.* 2,609,342).

Various means are used for converting fatty oils to suitable lubricant oils. Champagnat (*Fr.* 893,179) condensed the fatty oils with aromatic compounds using the Friedel-Crafts reaction. Condensation with resin oils also yields suitable lubricant oils (Vitex S. A.—*Fr.* 883,509). Lubricant oils made by esterification of the hydroxyl and carboxyl groups of hydrostearic acid with various lower aliphatic acids and alcohols, respectively, had pour points ranging from -20 to -53° (Teeter *et al.*—*Ind. Eng. Chem.* 45, 1777). Products developed in the decarboxylation of tall oil with ferric chloride catalyst include lubricating oils, gases, burning oils, phenols, and pitches (Enkvist—*Swed.* 134,809). Another process of decarboxylating tall oil included isomerization and polymerization of the various products to increase the lubricant oil yield (Assoc. Inst. du pin—*Fr.* 980,300). A new process for vegetable and animal fats comprised saponification, catalytic conversion of the acids to ketones, and reducing these to hydrocarbons by hydrogenation or by condensation with aromatic hydrocarbons (Menu—*Fr.* 885,865). Hydrogenated squalene is converted to viscous oil suitable for lubricant use by treatment with hydrogen peroxide in the presence of acetic acid (Matsumoto—*Japan* 4125 [51]). A watch oil is made by condensing *m*-xylylstearic acid with diethylene glycol monethyl ether in the presence of a small amount of *p*-toluene sulfonic acid (Martin, Jr.—*U. S.* 2,636,005). Methyl esters of the fatty acids of bone or apricot oil are recommended for lubricating precision instruments (Minasyan & Fuks—*Masloboino Zhirovaya Prom.* 18, No. 6, 15). A belting grease is made by heating whale oil foets and proteins with lye and then mixing with whale oil soap, water, bentonite and pine oil tar (Imai—*Japan* 1570 [51]).

General papers on wire drawing lubricants containing information on function, choice, etc., of oils, fatty acids, and soaps for the purpose were written by Salz (*Wire & Wire Products* 28, 1056), Pollack (*Seifen-Öle-Fette-Wachse* 79, 225), and Brown (*NLGI Spokesman* 17, No. 11, 8). Soaps for the purpose are improved by the addition of disodium dicalcium salt of ethylene diamine tetro-acetic acid (Bersworth *et al.*—*U. S.* 2,631,978). A lubricant for cold-rolling steel, brass, and aluminum contains mineral oil, fatty material, water, and emulsifier (Nunn *et al.*—*U. S.* 2,632,734). A product for use in rolling, drawing, or forging comprises soft petroleum wax, polybutene, and methyl dichlorostearate (Perry & Talley—*U. S.* 2,621,159). Mineral and fatty oils are emulsified in aqueous solution with fat derived emulsifiers in the production of metal cutting and cooling oils (Killingsworth—*U. S.* 2,628,197; Garner & Winsor—*U. S.* 2,606,874; Badische Anilin- & Soda Fabrik—*Ger.* 830,953; Laug—*U. S.* 2,625,509). Some patents on cutting oils deal with using sulfurized fatty materials in them (Churchill—*U. S.* 2,614,078; Waugh—*U. S.* 2,631,129; 2,631,131; Masanosaki & Hasegawa—*Japan* 535 [52]). Other cutting oil compositions are a mixture of soybean oil, spindle oil, barium soap, and oleic acid (Sumiki—*Japan* 4535 [51]), and a combination of rosin oil, fatty oil, and various hydrocarbons (Compagnie française raffinage—*Fr.* 977,025).

The recent patents on textile lubricants pertain to aqueous emulsion types. The patented aqueous emulsion products contain: animal and vegetable oils, and sulfonated castor oil (Fujii—*Japan* 3799 [51], 7684 [51]), tallow, whale oil, sulfonated sperm oil, and aluminum soap (Matsumoto—*Japan* 3984 [51]); and fatty pentaerythritol esters, fats, oils, fatty acids, fatty alcohol, sulfonated oil or alcohol, and hydrocarbons (Takahama—*Japan* 3800 [51]). Reaction products of fatty acids and polyalkylenepolyamines are recommended as emulsifiers in manufacture of emulsion type textile lubricants

(White & Landis—*U. S.* 2,622,067). Use of an emulsifying agent containing oleyl alcohol and aluminum trichloride also induces antistatic properties in use (Harrington—*U. S.* 2,615,032).

Fats partially polymerized, hydrogenated, and converted to lower alkyl esters are patented for use as spindle oils (Compagnie française raffinage—*Fr.* 980,328).

Records of fifty years of developments in the use of fatty materials in the leather industry is the text of two reviews (Faust—*J. Am. Leather Chemists' Assoc.* 48, 621; Meo, Jr. & Folsom—*Ibid.* 720). Glavind & Pedersen (*Acta Chem. Scand.* 6, 451) interpreted data on the changes in characteristics of leather oils in tanning to indicate that peroxide formation has a decisive role. They suggest that hydroperoxides form bridges between carboxyl groups of collagen peptide chains, and that one mole of unsaturated fatty acids combines with two carboxyl groups. In the manufacture of a new fatting liquor, crude wool oil is mixed with fish oil, submitted to oxidizing and/or polymerizing conditions, and partially sulfonated (Lighthipe *et al.*—*U. S.* 2,630,408). Use of sardine oil containing iron and free fatty acids for fat liquoring causes sticky leather (Czepelak—*Österr. Leder-Ztg.* 7, 113).

Deterioration of Fats and Oils

REVIEWS. General information or review articles pertaining to deterioration of fats and oils were written on mechanisms and theories (Lea—*Chemistry & Ind.* 1953, 1303), autoxidation and its prevention (Tamura—*J. Japan Chem.* 7, 72), contributions of K. Täufel to the chemistry of fat spoilage (Pohloudek-Farini & Rauscher—*Pharmazie* 7, 780), summary of a Scandinavian symposium on fat spoilage (Marcuse & Bergstrom—*Fette u. Seifen* 55, 545), hydrolysis, autoxidation, synergetics, and reversions (Paquot—*Rev. fermentations & Inds. aliment.* 8, 71, 119), metal deactivators for vegetable oil systems (Schwab—*Univ. Microfilms, Ann Arbor, Mich., Publ. No.* 4900, 73 pp.), application of antioxidants (Widaly—*Seifen-Öle-Fette-Wachse* 79, 1; Dubouloz—*Oleagineaux* 7, 465; Funabashi—*Koryo, Aromatics, No.* 22, 38; Tollenaar—*Olearia* 7, 5), free radical and infrared studies of some tocopherol and other 6-hydroxychroman antioxidants (Inglett—*Univ. Microfilms, Ann Arbor, Mich., Pub. No.* 4073, 93 pp.), antioxidants in home preservation of foods (Hanning *et al.*—*J. Home Econ.* 45, 660), tests for deterioration of fats (Drozdov & Materanskaya—*Myasnaya Ind. S.S.S.R.* 24, No. 2, 69), organoleptic study and rancidity tests (Balavoine—*Mitt. Gebiete Lebensm. u. Hyg.* 43, 521), oxidative changes in lard (Drozdov *et al.*—*Myasnaya Ind. S.S.S.R.* 24, No. 4, 82), reversion of soybean oil (Smith—*Am. Perfumer & Essential Oil Rev.* 61, 72), flavor reversion (Mestreit—*Bull. mens. inform. ITEG* 7, 64; Daubert & O'Connell—*Advances in Food Res.* 4, 185), Northern Regional Res. Lab. work on the flavor problems of soybean oil (Dutton *et al.*—*Trans. Am. Assoc. Cereal Chemists* 11, 116), and oxidative defects of stored butter (Petersen—*Fette u. Seifen* 55, 712).

TESTS FOR DETERIORATION, STABILITY, AND ANTIOXIDANTS. A new test for rancidity employs reduced phenolphthalein in the presence of an oxidation-reduction buffer, the degree being indicated by the intensity of red color development (Kehren—*Anais farm. e quim. Sao Paulo* 6, No. 2, 5). In studies on determination of carbonyl compounds as a measure of deterioration, the application of semicarbazide precipitation failed on highly autoxidized oils because of presence of reducing compounds; whereas a procedure based on color reaction with hydroxylamine was useful for the purpose (Feuell & Skellon—*Analyst* 78, 135). Neumer & Dugan, Jr. (*Food Tech.* 7, 191) used the rate of formation of volatile carbonyl compounds under accelerated conditions for appraising the stability of fats in dog food.

A comprehensive study of deterioration and stability of fats was recorded by Purr (*Fette u. Seifen* 55, 239, 317, 389). He reviewed the literature; developed a semimicro peroxide determination; showed how keeping quality could be evaluated by color development with butanolic dichlorodihydroxydiphenylamine during accelerated aging; recorded the temperature and time relations for various fats to attain peroxide values of 10 and of 20; and on the basis of the data showed how rancidity and stability may be defined and the inhibiting value of antioxidants determined. Bose & Subrahmanyam (*Bull. Central Food Tech., Res. Inst. Mysore* 2, 151) plotted the development of peroxide values of peanut and coconut oils during storage at 20-95° to demonstrate that the peroxide test is not reliable as an indication of extent of deterioration. Iodometric determinations of peroxide values in the presence of a sulfuric acid-pyridine mixture or sulfuric acid-dimethylaniline mix-

ture agree, whereas with glacial acetic acid results are lower (Starikova—*Myasnaya Ind. S.S.S.R.* 24, No. 2, 72).

Several modifications were suggested for determining the stability of fats and oils by the Swift accelerated oxidizability method. Determination of peroxide value with ferrous thiocyanate reagent instead of iodometrically and a temperature increase to 125° in the procedure gives a 75-90% saving of time (Golden—*J. Am. Pharm. Assoc.* 42, 545). Accelerating the test at 110° rather than the usual 97.8° reduced the time required about 40% (Morris & Freeman—*Food Tech.* 7, 227). Replacing the glass tubes in the apparatus with iron tubes is said to give more realistic values for stabilities of fats and for the evaluation of antioxidants (Luckmann—*J. Am. Oil Chemists' Soc.* 30, 602). This modification was devised to bring into practice the factor of iron contamination; since the industry transports, stores, and processes fats and oils in iron equipment. A study of accelerated oxidative deterioration tests for olive oils indicated that the Schaal oven test most nearly simulates actual commercial storage conditions (Acala—*Anales real soc. espan. fis. y quim., Madrid*, 49B, 255). A method of detecting the presence of antioxidants or synergists is based on rate of peroxide development during the accelerated oxidizability test (Kantha—*J. Sci. Ind. Res. India* 12B, 35, 37).

Some studies on tests were restricted to butter. Interference of butter color on the colorimetric Schibsted aldehyde value is eliminated by use of photoelectric colorimeter (Dolby—*J. Dairy Res.* 20, 198). Extraction of color and photoelectric measurement are modifications recommended for applying the thiobarbituric acid test for butterfat oxidation (Biggs & Bryant—*Can. J. Tech.* 31, 138). A new deterioration test is based on the amount of potassium permanganate needed to oxidize the steam volatiles obtained from 25 grams of butter (Zaikovskii & Chizhkova—*Molochnaya Prom.* 14, No. 4, 32). An index of stability for butterfat is based on a measure of time required by a sample exposed to air on a glass plate to rapidly gain weight (Zaikovskii—*Ibid.* No. 2, 29). Excessive amounts of water insoluble free fatty acids and/or butyric acid may indicate that the butter was made from cream that has undergone bacterial deterioration (Hillig—*J. Assoc. Off. Agr. Chemists' 36*, 1077; Peters *et al.*—*J. Dairy Sci.* 36, 799). However, good butter may become unsatisfactory if lipolytic bacteria are permitted to develop in the product (Kester—*Ibid.* 794). Running reductase and catalase tests on cream is recommended for predicting the keeping quality of the butters produced therefrom (Valik—*Intern. Dairy Congr. Proc.* 13th 2, 717).

Free fatty acidity in fats is quickly approximated with the pH indicators, neutral red and Nile blue (Keller—*Lebensmittelrzt* 3, 105).

The kinetics of oxidation of free fatty acids may be measured by area changes of monomolecular layers on dilute solutions of sulfuric acid and on potassium permanganate at constant surface pressures (Eda—*J. Chem. Soc. Japan, Chem. Sect.* 73, 260).

MECHANISMS OF DETERIORATIONS. Prior workers have postulated that, on autoxidation, initial oxygen absorbed is used in hydroperoxide formation and later oxygen is consumed to form non-hydroperoxide materials. Such mechanism has been confirmed by Willets *et al.* (*J. Am. Oil Chemists' Soc.* 30, 420) using polarographic analytical technique and by Khan (*Ibid.* 273) who demonstrated the phenomena with oleic acid labeled with deuterium at the double bond carbons. Swern *et al.* (*J. Am. Chem. Soc.* 75, 3135) demonstrated that the hydroperoxides are predominately of trans configuration, and that the non-hydroperoxide portion probably consists of cyclic isomers. Skellon and coworkers (*J. Chem. Soc.* 1952, 1813; 1953, 138), using elaidic and erucic acids, showed that, like oleic acid, they first develop isomeric hydroperoxides and then break down into unsaturated ketones, acidic fission compounds, and neutral saturated compounds. Toyama & Yamamoto (*J. Chem. Soc. Japan Ind. Chem. Sect.* 55, 235) identified many fission products in ethyl oleate which had undergone autoxidation during storage.

In studies of mechanism of autoxidation of animal tissue fat, Tappel (*Arch. Biochem. Biophys.* 44, 378; *Food Res.* 18, 560) records that the catalytic activity of some associated material in decreasing order is cytochrome C, hemin, and hemoglobin. He postulates that the mechanism for linoleate oxidation involves a direct reaction of the peroxide with hematin catalyst; propagation in a manner similar to autocatalytic oxidation of linoleate; and the hematin is co-oxidized with release of iron.

FACTORS AFFECTING STABILITY. In two studies on fat deterioration in stored pork, Privet (*Rept. Hormel Inst.* 1951-52, 10) pointed out that the rancidity is mainly localized in the surface

fat and Palmer *et al.* (*Food Tech.* 7, 90) demonstrated how low temperature storage and superior packaging improves stability. Klose *et al.* (*Poultry Sci.* 32, 82) showed that two percent sardine oil in turkey rations induces an intense fishiness in the cooked carcass and that two percent linseed oil results in moderate fishiness. Feeding tocopherol to dairy cattle increases the tocopherol content of the butter produced, but the alleged increase in stability of the butter was not evident (Nielsen *et al.*—*J. Dairy Res.* 20, 333). A study of the composition of oils from different varieties of peanuts in relation to stabilities, indicated that the linoleic acid content is the major factor, and it is possible that the higher tocopherol content of Runner peanut oil tends to enhance its stability (Fore *et al.*—*J. Am. Oil Chemists' Soc.* 30, 298).

Whole egg sponge cake batter of 4.9% lipide content became off-flavored in four weeks, and inedible after six months' freezer storage (Mayer *et al.*—*Food Res.* 18, 70). There was no off-flavor in samples baked before freezer storage. In these tests flavor deterioration and lipide peroxides were proportional.

In ice cream, rancid flavor was related to the amount of free fatty acids (Shotwell *et al.*—*J. Dairy Sci.* 36, 948). In these studies it was revealed that the free fatty acid content of fresh ice cream is considerably higher than that of the fresh milk and cream constituents. Spontaneous development of considerable amounts of free fatty acids in palm oils during transportation or storage is inhibited by removal and exclusion of moisture (Loncin—*Oleagineux* 7, 695; *Fette u. Seifen* 55, 7; Thuriaux—*Bull. inform. inst. natl. etudes agr. congo* 1, 287).

Acceleration of rancidification is still a major problem when foods are sterilized for storage by means of high-intensity cathode rays (Proctor—*Certified Milk* 27, No. 5, 15; Astrack *et al.*—*Food Res.* 17, 571). Autoxidation of certain fats is also accelerated by supersonic treatments (Sakurai—*J. Chem. Soc. Japan, Ind. Chem. Sect.* 54, 246). Acceleration of the rate of rancidification by bactericidal lamps should be considered against advantages of their sterilizing effects (Hinko—*Fette u. Seifen* 55, 113).

Copper salts form complexes with choline which strongly accelerate autoxidation and may account for development of rancidity in high copper experimental diets (White *et al.*—*J. Am. Chem. Soc.* 75, 2352).

Some investigations on stability pertain to vitamin A and carotene. Reduction of vitamin A in margarine is not stimulated by the synthetic food dye, whereas addition of biacetal or presence of nickel ions is detrimental (Naito & Mori—*Vitamins, Japan*, 5, 191). Vitamin A and carotene are very stable in margarines stored at 45° F., and fairly so even when held at room temperature (Melnick *et al.*—*Food Res.* 18, 504). Although vitamin A is stable in sesame oil for only 13-26 weeks, margarine from good stock containing five percent sesame oil, as required by law in some countries, can be stored for 42 weeks at room temperature without appreciable loss of the vitamin content (Murray *et al.*—*J. Am. Oil Chem. Soc.* 30, 329). Vitamin A content of 10 of 13 commercial samples of Canadian margarines after 12 months' cold storage still surpassed label claims (Murray & Campbell—*Can. J. Tech.* 31, 193). Unsaturated oxidases have been found responsible for loss of vitamin A in butter, and rancidity in fish, lard, and other animal fatty materials (Blain *et al.*—*J. Sci. Food & Agr.* 4, 530).

ANTIOXIDANTS. Studies with eight antioxidants by Sahasrabudhe (*J. Sci. Ind. Res., India*, 12B, 63) indicates that butylated hydroxyanisole and propyl gallate are best for natural peanut oils and hydroquinone for hydrogenated oils. Sato *et al.* (*J. Oil Chem. Soc., Japan* 1, 157) find nordihydroguaiaretic an excellent antioxidant for hardened whale and cottonseed oils, and butyl-*p*-hydroxybenzoate best for coconut oil. Fat stabilization for bacon is provided by dipping the slices in lard containing 5.5-10% butylated hydroxyanisole, or by injecting 0.2% aqueous solutions into the slabs (Hanley—*Food Tech.* 7, 429). Rancidity is inhibited in refined olive oil by adding five percent of virgin oil or any of the above mentioned antioxidants (Alcalá—*Grasas y aceites* 7, 173). Among C₁ to C₁₁ alkyl esters of gallic acid the C₂ to C₄ esters are the best antioxidants (Oshiwa—*J. Agr. Chem. Soc. Japan* 25, 53). At 0.0001 molar concentrations alkyl esters of protocatechuic acid are as effective as the gallic acid esters, at higher concentrations the latter are superior (*Ibid.* 344). At pH 5.1 and in the presence of water the antioxidant effect of gallic acid, pyrogallol, and propyl gallate is less than that of lauryl gallate but pyrocatechol and nordihydroguaiaretic acid are considered most effective (Spetsig—*Svensk Kem. Tidskr.* 65, 65). In tests on the effect of temperatures from -6 to 30° upon

the lipoxidase-catalyzed oxidation of sodium linoleate the best inhibitive effect among several antioxidants was with nordihydroguaiaretic acid, followed closely by propyl gallate (Tappel *et al.*—*Arch. Biochem. Biophys.* 42, 293). These are also demonstrated to be good antioxidants for general use, especially when in combination with synergists (Galindo—*Testis quim. Univ. Chile* 3, 23). Siegfried & Schneider (*Pharm. Acta Helv.* 28, 131) find that "Progallin P," nordihydroguaiaretic acid, and ascorbyl palmitate are only slightly effective for olive or almond oil but very effective for lard. The effectiveness of 43 chemical compounds and substances for shark-liver oil are recorded (Nair & Ramakrishnan—*Bull. central Res. Inst. Univ. Travancore Ser. A2*, 77, 86, 100). Phenolic type antioxidants are found most effective in providing stability in dry dog foods containing added fat (Neumer & Dugan, Jr.—*Food Tech.* 7, 189).

Natural antioxidant concentrates were extracted from spices (Chipault—*Ann. Rpt. Hormel Inst. 1951-52*, 25) and from osage orange fruit (Clopton—*J. Am. Oil Chemists' Soc.* 30, 156). The suitability of quercetin for inhibiting autoxidation of triolein and several alkyl esters of unsaturated fatty acids was demonstrated (Heimann *et al.*—*Fette u. Seifen* 55, 394). A cheap source of this antioxidant is the bisulfite liquors from fir and pine bark (Kurth—*Ind. Eng. Chem.* 45, 2096). Alpha-tocopherol inhibits the hematin-catalyzed oxidation of vitamin A (Tappel—*Arch. Biochem. & Biophys.* 47, 223). Amino-4-hydroxyflavonol (Katsui—*Vitamins, Japan*, 6, 69) and nordihydroguaiaretic acid (Suzuki *et al.*—*Ibid.* 5, 337) are recommended for inhibiting losses of vitamin A in medicinal oils.

New biphenyltetrool compounds (Chiang & Gisvold—*J. Am. Pharm. Assoc. Sci. Ed.* 41, 348), and several compounds with structures like nordihydroguaiaretic acid and *p*-hydroxyanisole (Shishido & Kuyama—*Bull. Inst. Chem. Res. Kyoto Univ.* 25, 73; 26, 87; 30, 49) were prepared and some were found to have good antioxidant activity. Pure alkyl gallate antioxidants were prepared by heating gallic acid and aliphatic alcohols in the presence of alkyl sulfate (von Brammer & Clemens, Jr.—*U. S.* 2,615,042).

Certain oil soluble organic phosphates and sulfates possess antioxidant powers that are manifested directly by a mechanism not involving polyphenols (Desnuelle & Benoit-Micaelli—*Bull. soc. chim. France* 1953, 595). The oil stabilizing properties of phytic acid (Evans *et al.*—*J. Am. Oil Chem. Soc.* 30, 143) and esters of citric acid (Vahlteich *et al.*—*Food Tech.* 8, 6) are attributed to a deactivating action on pro-oxidant metals. Hartman (*J. Sci. Food Agr.* 4, 430) has recorded experimental data for stabilizing lard with citric or tartaric acids. Sorbitan monostearate and polyoxyethylene sorbitan monostearate when tested on butter oil show slight antioxidant capacity (Hollender—*J. Dairy Sci.* 36, 561).

Patents have been issued on using the following as antioxidants: polyhydroxybenzoic acid esters of alcohols containing six or more carbon atoms (Ault *et al.*—*U. S.* 2,595,221), phosphorylated starch containing 10% phosphorus (Cowan & Evans—*U. S.* 2,626,951), a mixture of a phenolic antioxidant and dehydrated alkali phosphate (Watts & Moss—*U. S.* 2,629,664), a mixture of natural tocopherol and methyl silicone (Martin—*U. S.* 2,634,213), compounds formed by esterifying monocarboxylic acid at the hydroxyl group of citric acid (Schwab & Dutton—*U. S.* 2,636,887), certain alkyl phenols (Ross—*U. S.* 2,638,475), 2,5-dihydroxyphenyldimethylcarbinol and its dimer (Bell & Shields—*U. S.* 2,639,288), oxydialkanic acid derivatives (Cowan & Schwab—*U. S.* 2,645,648), a combination of butylated hydroquinone, propyl gallate and an acidic material (Beadle & Kraybill—*U. S.* 2,648,608), β -thiaketo sulfoxide or β -thiaketo sulfone derivatives (Thompson—*U. S.* 2,648,691), 2-amino-3-hydroxybenzoic acid or its alkyl esters (Olcott & Campbell—*U. S.* 2,652,332), alkyl-*p*-phenylene diamine and a mercaptan (Hill & Welp—*U. S.* 2,657,982), carboxymethyl mercaptosuccinic acid (Schwab *et al.*—*U. S.* 2,662,905), certain alkylated hydroxyphenyl ethers (Magoffin—*Brit.* 679,192), 2,4-dimethyl-6-alkylphenols (Jones & Cowie—*Brit.* 679,504), and the alkali treated pressed juice from citrus fruits (Hirano & Kokura—*Japan* 2976 [51]). Nitrogen coordination compounds, as chelidonic or chelidamic acid, inhibit the pro-oxidant influence of certain metals that may contaminate fatty oils (Schwab *et al.*—*U. S.* 2,661,353; *J. Am. Oil Chemists' Soc.* 30, 177); the same effect is alleged for sulfur compounds of the tridentate class having two carboxy groups in α - or β -position to the coordinating atom (Schwab *et al.*—*Ibid.* 413). Dialkyldithiocarbamic acids and tetraalkylthiuram disulfides were patented for preventing off-flavor and fishiness in butter (Tollenaar—*U. S.* 2,638,420; *Dutch* 70,854, 71,606; *Centraal Inst. Voedingsonderzoek T. N.O. Utrecht No. 148*, 16 pp.; 166,

148 pp.). The stability of butter is improved by adding a small amount of lactose (Gintersdorfer—*Austrian* 174,276). Salt coated with glycerol monostearate and an antioxidant is intended for salting and for stabilizing fat-containing foods against rancidity (Robison—*U. S.* 2,645,581).

A blue color defect of lard treated with butylated hydroxyanisole, propyl gallate and citric acid results from reaction of the antioxidants with iron contaminants, but only occurs when lecithin is also present (Dugan, Jr. & Marx—*J. Am. Oil Chemists' Soc.* 30, 452). A study of destruction of propyl gallate and butylated hydroxyanisole in lard indicates that the rate is constant for the former; in two stages for the latter. The rapid stage starts with disappearance of the former; and the lard also rapidly deteriorates after complete disappearance of the propyl gallate (Mahon & Chapman—*Ibid.* 34).

Washing red oils with 20% solutions of urea removes the metallic contaminants which adversely affect the stability as measured by the Mackey test (Knafo & Juillard—*Bull. mens. inform. ITERG* 7, 22). Results from the Mackey tests on methyl oleate and on methyl linoleate with and without antioxidants were tabulated (Francois & Juillard—*Ibid.* 6, 483). These permit evaluation of the approximate composition of red oil and stabilizing activity of diphenylamine, β -naphthol, and butyl cresol.

FLAVOR AND ODOR DETERIORATION. Onoc and coworkers (*J. Chem. Soc., Japan, Pure Chem. Sect.* 73, 193) in studies on odor of whale oil, distilled the volatiles and identified therein formic, acetic, propionic, and isovaleric and other acids; formaldehyde, ethanol, and higher aldehydes; carbonyl, and peroxide compounds. Similar work done on ethyl linoleate (Chang & Kummerow—*J. Am. Oil Chemists' Soc.* 30, 251; Johnson *et al.*—*Ibid.* 317), and on methyl isolinoleate (Harrison & Daubert—*Ibid.* 371) resulted in conclusions that linoleates and isolinoleates are precursors to the flavor reversion compounds.

Several methods for inhibiting reversion were described. For soybean oil, Gooding *et al.* (*U. S.* 2,627,467-9) partially hydrogenated and then winterized the oil; Mattil (*U. S.* 2,613,215) partially hydrogenated and then removed the unsaponifiable substances with selective solvents; and Thurman (*U. S.* 2,621,191, 2,621,196) steam distilled to recover vitamins as by-products, then refined, and bleached the oil. Contact with magnesium oxide is said to remove impurities that cause reversion of oils (Distillers Co. Ltd.—*Brit.* 685,044).

Tollenaar (*Fette u. Seifen* 55, 3) suggested that fishiness in butter is caused by the oxidation of the fatty acids of the phospholipides. However, van Haften & Pette (*Neth. Milk Dairy J.* 7, 41) associated fishiness with presence of copper, season produced, and salting. They could not induce fishiness with egg phospholipides and discovered that fishiness occurs in absence of phospholipides. Munroe & Barincoat (*J. Dairy Res.* 20, 274) failed in deliberate attempts to produce fishiness by low pH, high salt content, traces of copper, and other factors reputed to cause the defect.

BACTERIAL DECOMPOSITION. A yeasty taste in butter is attributed to microorganisms and methods for its control were issued by Petersen (*Fette u. Seifen* 55, 124). In the incubation of cream, free butyric acid arises from the fat and from the fermentation of milk serum constituents, most probably lactose (Peters—*J. Dairy Sci.* 38, 268).

Enzymes of *penicillium chrysogenum* that are capable of oxidizing lard may be present in relatively low concentrations in the young mycelium and be synthesized as growth takes place (Rolinson & Lumb—*J. Gen. Microbiol.* 9, 385).

BIOLOGICAL ASPECT OF SPOILED FATS. A diet containing 10% rancid soybean oil supports almost normal growth in rats when all known essential nutrients are supplied in large amounts and protein constitutes 30% of the diet. This is interpreted to indicate that the protein requirements are increased by the rancidity (Greenberg & Frazer—*J. Nutr.* 50, 421). Abortions in rats fed excessively rancid fat in the diet are said to be due to effects different than when abortion is due to vitamin E deficiency (Curto—*Acta Vitaminol* 6, 241). Fats that develop high peroxides and epihydrinaldehyde contents on rancidification also acquire microbicidal qualities (Torre & Silvestri—*Igiene mod.* 46, 46).

Heating rancid codliver oil reduces its deleterious effect on growth of animals (Groot & Obbink—*Experimentia* 9, 189). Linseed and soybean oils become toxic when excessively overheated or polymerized. The effect is ascribed tentatively to the cyclization of monomeric esters as such, for the segregated polymers are unabsorbed and only traces of hydroxy compounds are present in toxic fractions (Wiseblatt *et al.*—*J. Sci. Food & Agr.* 4, 227; Wells & Common—*Ibid.* 233; Crampton *et al.*—*J. Nutr.* 49, 333).