

Review of Literature on Fats, Oils and Soaps for 1943*—Part 1

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General

There has been a slight decrease in the volume of literature on fats, oils and soaps during the year 1943. This resulted from the increased difficulty of obtaining journals from the Axis and Axis occupied countries. The literature of Allied and nonbelligerent nations seems to have kept pace with that of former years, although there may have been slight shifts of interest. Physiological subjects such as digestibility, nutritive value, etc. are developing rapidly; the search for new sources continues; there seemed to be a numerical reduction in patents issued on synthetic detergents and the fat economy situation in Allied nations was discussed with favorable comment.

The revision of the well-known American Chemical Society Monograph "Vegetable Fats and Oils" by George S. Jamieson brings the information treated in the first edition up-to-date and describes many new fats and oils which did not appear in the older book. The introduction of new and revised analytical procedures has also added to its popularity. Another monograph entitled "Biochemistry of the Fatty Acids" by W. R. Bloor fills an important need in scientific literature on fats and oils. In addition to its value as a reference book, this publication will encourage interest and development in the food phase of fatty materials. A book "Practical Emulsions" by H. Bennett contains information on production and preservation of emulsions.

Production of fats and oils from domestic material will total about 11.2 billion pounds as compared with 10.6 billion pounds a year earlier. As evident from the accompanying statistical chart this was substantially greater than ever before. Tabular data on production, consumption, imports and stocks for the years 1938-42 were issued by the U. S. Department of Commerce.

Requirements for fats will be larger for the coming year. Military needs are increasing and Lend-Lease shipments will continue at high levels. If the war should end in 1944, the relief requirements for Europe will impose an additional demand on our stocks. A general treatise on the economical outlook of fats and oils in the war was prepared by Brandt (*Food Research Institute War-Peace Pamphlet No. 2*).

The prices of oils have remained near ceiling levels with the exception of linseed oil and lard on which high seasonal supplies reacted by lowering the price.

The soap industry suffered from a peculiar situation. A deficiency in inedible fats led to releasing some lard for soap making. However, since the ceiling price of soap has not been increased it is doubtful whether much of this higher priced raw material will find its way into the soap kettles. This demand for soap oils was the result of present industrial activity. Before the war about 15% of the soap produced was used for industrial purposes. The added industrial activity, the military needs and the requirement for synthetic rubber are utilizing about 50% of the soap produced. Statistical data and an economic survey of the soap industry were prepared by Kozlik and Diebold (*Agr. Expt. Sta. Iowa State College Bull. 311*).

Attempts to fill the soap fat deficit were in the form of regulations on the minimum requirements for addition of rosin and builders, a new domestic fat salvage plan and promotion of industrial salvage. Housewives are now being paid four cents and two meat points per pound of salvage fat turned into meat shops. Trade literature, especially that of the meat packing industry, has been fostering fat salvage, particularly with descriptions of methods and equipment of recovery from waste water, glue liquors and the stockinettes used for hanging special meats during smoking. The California Dairy Council has launched a drive to save the fat residue from individual Babcock milk tests. A simple method for doing this, with the calculations on probable yields was issued by Biendarra (*Natl. Butter & Cheese J. 34, No. 6, 9*). A new source of salvage fat appearing in foreign literature (Chiarabba—*Chim. peintures 4, 280*) was from the lye used to boil broom in order to separate the fibers. Neutralizing this lye with sulfuric acid yielded a precipitate containing 40-50% fat.

Before the war the tin plate and cold reduced sheet steel industries used about 40 million pounds of palm oil annually. Since this oil was unavailable in sufficient quantities, an especially hydrogenated cottonseed oil was developed as a substitute (Bauer and Markley—*Oil & Soap 20, 1*; Skinner—*Mfrs. Record 112, No. 7, 34*). Palm oil economy notes and a new method of refining the oil for the metal industry were prepared by and issued to Dunmire (*Iron Steel Engr. 20, 55; U. S. 2,316,571*).

In the drying oil field several writers proposed means of conserving linseed oil (Gardner—*Paint Ind. Mag. 58, 120, 122*; Allen *et al.*—*Ibid. 118*; Mof-fat—*Can. Paint & Varnish Mag. 17, No. 4, 38*), especially as to commercial treatment and methods of partial substitution with synthetic resins. Good

OUTLINE

- A. General
- B. Technical Treatment
- C. Products (Except Detergents)
- D. Deterioration
- E. Biochemical
- F. Characteristics and Composition
- G. Detergents

* Report of the American Oil Chemists' Society Committee for Review of Literature on Fats, Oils and Soaps. G. R. Greenbank, Geo. S. Jamieson, H. A. Mattill, R. C. Newton, M. M. Piskur, Chairman.

(*Paint Ind. Mag.* 58, 154) advised the use of isomerized linseed oil, "Conjulin," and isomerized soybean oil, "Conjulsoy," as substitutes for the better drying oils. New substitutes for tung oil are the seed oils of *Garcia nutans* and cacahuananche. Their characteristics will appear in tables appended to the section of this review on composition and characteristics of fats and oils. The last named oil is similar to tung oil but dries more slowly. Jamieson and Rose, and Gardner and Westgate who analyzed these new drying oils also recorded useful information on their drying behavior. The chart also contains data on three new drying oils described by Steger and van Loon. Another new oil, from dingili seed, is noteworthy because it contains a high percentage of ricinoleic acid. Industrial use for castor oil and its derivatives was the subject of several monographs (Worsdall—*Paint Varnish Production Mgr.* 22, 255; Good—*Ibid.* 23, 3; Vianna—*Tribuna farm. Brazil* 10, 210; Richelsen—*Soap* 19, No. 1, 24).

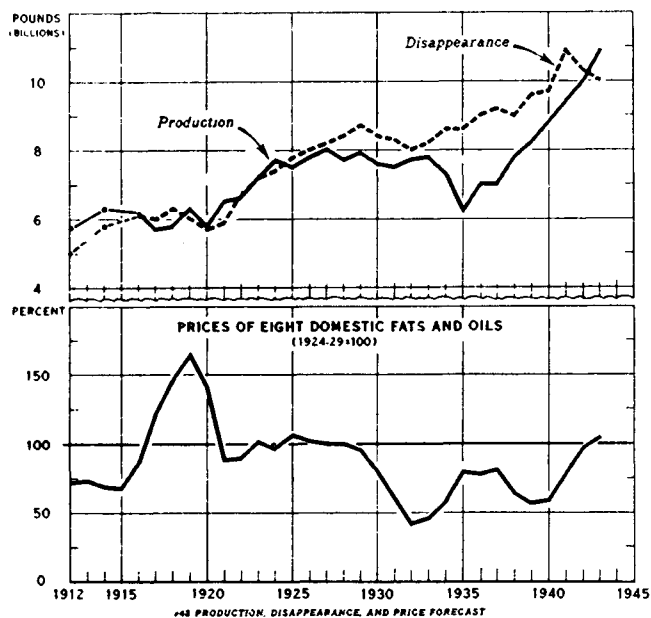
In the food fat field substitution concerned principally olive oil. Musher (*Oil & Soap* 20, 26) recommended blending other vegetable oils with olive oils carrying a strong flavor. Bickford *et al.* (*Ibid.* 85) recorded that peanut and pecan oils appeared capable of being modified to produce a product chemically similar to olive oil. Supplies of olive oil will increase as the war progresses. Some supplies from Algeria and French Morocco were already received in exchange for other fats and oils (Small—*Foreign Commerce Wkly.* 10, No. 4, 8). Angel'skii and Gachechiladze (*Izvest. Batum. Subtropicheskogo Botan. Sado* 1940, No. 5, 161) recorded that seed oil of the camphor tree was a good substitute for coconut oil. Kraemer *et al.* (*Oil & Soap* 20, 235) prepared a substitute for cocoa butter from cottonseed oil by selective hydrogenation followed by fractional distillation.

Other communications on interchangeability of fats and oils were general papers on altering the physical properties of fats so that they would meet definite requirements (Vahlteich—*Chem. Eng. News* 21, 1238; Wittka—*Allgem. Oel- u. Fett-Ztg.* 38, 269, 318, 397). A discussion on fat derivatives was prepared by Ralston (*Chem. Eng. News* 21, 5).

Several other general communications encouraged greater utilization of many known oils. Some of these were: bone fat (Violier and Iselin—*Mitt. Lebensm. Hyg.* 32, 255), grape seed oil (Balbi—*Vernici* 18, 165; Engels—*Allgem. Oel- u. Fett-Ztg.* 39, 271), pecan shell oil (Planters Cotton Oil Co.—*Chem. Met. Eng.* 50, No. 9, 157), penao tree seed oil (Bondar—*Rev. quim. ind. Brazil* 11, No. 128, 15), poppy seed oil (*Allgem. Oel- u. Fett-Ztg.* 39, 127), pumpkin seed oil (Tschermak-Seipenegg—*Milchw. Ztg. Alpen-Sudeten u. Donauraum* 50, 220), squash seed oil (Buchinger—*Wien. Landw. Ztg.* 92, 202), star fish oil (Pottinger—*Atlantic Fisherman* 23, No. 9, 9), tall oil (Jacobson—*Svenski Papperstidn.* 46, 128, 183, 283; Baroni—*Z. Papier, Pappe, Zellulose, Holzstoff* 59, 20) and vegetable oils of Brazil (de Aranjó—*Industria y quim.* 5, No. 2, 46).

A new biochemical method for fat synthesis made use of *Fusarium* (Damm—*Chem.-Ztg.* 67, 47; Henkel & Cie.—*Ital.* 381,767). These bacteria can utilize all commercial nitrogen compounds and fermented pentoses without prolonged adaptation. Reviews on this subject were prepared by Fawns (*Food Manuf.* 18, 333) and Munin (*Fette u. Seifen* 49, 123). Schreiber

FATS AND OILS: PRODUCTION FROM DOMESTIC MATERIALS, APPARENT DOMESTIC DISAPPEARANCE, AND INDEX NUMBERS OF WHOLESALE PRICES, 1912-43



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(*Z. physiol. Chem.* 276, 56) evaluated the fat synthesizing action of several seed lipases. A glycerol concentration of 55-90% was optimum and in some cases synthesis reached 48% of the calculated possible maximum.

Manufacture of fat substitutes from gases, mineral oils and coal are active commercial processes in Europe. Imhausen (*Kolloid-Z.* 103, 105) reported that the oxidation residues from products prepared by hydrogenation of carbon monoxide differed from natural fats in that they contained both even and odd carbon chains. The esters of these fat acids were said to be used as cooking fats and butter substitutes. However, most of the general discussions on this subject pertain to manufacture of soap from the products (Spirk—*Chem. Listy* 35, 99; Profft—*Fette u. Seifen* 49, 868). New patents on synthetic fat acids and oils deal with temperatures and conditions for oxidation of hydrocarbons (Markische-Seifen-Industrie—*Ger.* 706,791 Cl. 12o; Roblee & Thörl G.m.b.H.—*Ger.* 722,617 Cl. 23d), a distillation method for the oxidation residues (Noble & Thörl G.m.b.H.—*Ger.* 723,294 Cl. 23d) manufacture of chlorinated products from the synthetic fat acids (I. G. Farbenind A.-G.—*Ger.* 709,582 Cl. 12o), refining methods for the saponified products using treatment with sulfites (*Ibid.*—*Ger.* 708,124 Cl. 23d) and treatment with salt at pH 5.5-7.5 (*Ibid.*—*Ger.* 708,125 Cl. 23d).

Primary Technical Treatment

One hardly expects great revolutionary changes in equipment and processes during times of stress; alterations would require machinery whose manufacture is restricted. However, many new developments have been described in letters patents, and communications have encouraged rational use of the raw materials and equipment on hand.

Lachle (*U. S.* 2,325,327-8, 2,331,785) designed oil extraction processes for cellular oil-bearing materials having gelatinous constituents. Some of the interesting steps in the processes were removal of starch,

protein, or other gelatinous materials by liquefaction with enzymes or solution in water, the use of ball mills for liberating the oils from cells and the use of centrifuges for liquid concentration and separation. Operating schemes for olive pulp, corn germ, sardine, castor bean, and other raw materials were illustrated and described in the letters patents. Schopmeyer and Sharp's (*U. S. 2,310,184*) process for separating oil from corn gluten comprised finely grinding, converting to a solution or slurry in water and separating with centrifuges.

Some patents featured improvements for pressing equipment. Improvements in the equipment of V. D. Anderson Co. (*U. S. 2,299,784, 2,325,357, 2,331,883, 2,331,910*) were a novel organization of and relation between preliminary press, feed hopper, feed press and main press, an improved valve for regulating flow to expeller and a press designed so that the expressed oil was utilized as a cooling medium for the pressing mechanism. Other mechanical patents dealt with a "cake knocker" for oil press (Sumner and Branch—*U. S. 2,300,182*) and a new centrifuge (Hall—*U. S. 2,313,540-1*). Two communications emphasized that sludge and turbidity should be removed from newly pressed oils to reduce decomposition and prevent reduction of quality. For this purpose Cellin (*Allgem. Oel- u. Fett-Ztg. 38, 272*) recommended centrifuges and Wittka (*Ibid. 37, 409*) preferred small filter presses. An oil filter patented by Halstead (*Brit. 545,733*) was made of cotton impregnated with a sulfonated high molecular weight organic compound.

New methods for fish oil production were proposed. Mazza and Migliardi (*Ricerca Sci. 13, 267; Quaderni nutriz. 18, 80*) described apparatus and a procedure for extraction of fish liver oils after digestion of the livers with papain. More oil was extracted, its characteristics barely differed from normal oil, but its vitamin A and vitamin D₂ content were each about 4-6 times greater than the oil produced without proteolysis. It was suggested that these additional vitamins had been bound to the lipoproteids and were released during digestion. Another method of fish liver oil extraction by Bull (*Tek. Ukeblad 87, 309*) comprised mixing the ground livers with anhydrous sodium sulfate, heating for two hours and centrifuging the mass into three portions: oil, viscous organic residue and salt solution. Hempel (*U. S. 2,303,740*) treated fish livers that had to be held for a prolonged time before extraction with ammonium hydroxide and ammonium borate. The chemicals acted as preservatives and aided in the subsequent extraction. Another pre-extraction treating agent, particularly for fish offal, by Henshall (*U. S. 2,309,392*) comprised a mixture of sodium bisulfate with aluminum and iron sulfate. Similarly van Deurs (*Ingenioren 51, No. 19, K20*) recommended acidifying to a pH of 2 with organic acids. A press for production of fish oil and fish meal was designed by Sterner (*Ger. 718,728 Cl. 58b*).

A biochemical process for production of animal fats included fermenting the comminuted raw material with a carbohydrate culture of lactic acid bacteria under aeration (Ekhard—*Ger. 707,226, Cl. 23a*). The bacterial culture was separated and the fat washed.

Freeman, Pack and McKinney (*Ind. Eng. Chem. 35, 1156*) listed hexane, petroleum naphthas, cyclo-

hexane, benzene, *n*-heptane, methylcyclohexane, chloroform, isopropyl ether and ethyl ether as suitable solvents for extraction of tung oil. This investigation included tests on 33 solvents. Measmer (*Iowa State Coll. J. Sci. 17, 100*) recommended alcohol containing 10% trichloroethylene for the hot extraction of soybean oil. After cooling, the solvent separates into 2 layers, the upper layer being pure solvent and the lower containing 50% oil. New equipment for countercurrent solvent extraction of soybeans was designed by Levine and Dent (*U. S. 2,321,923*). A new solvent recovery invention was a still for separating propane from extracted cottonseed oil (Rosenthal—*U. S. 2,290,209*). In 2 papers of general nature there was a review on solvent extraction of cottonseed oil (Harris—*Bull. Agr. & Mech. Coll. Tex. 12, No. 12*) and a description of a solvent extraction plant for the meat packing industry (*Natl. Provisioner 109, No. 18, 10*).

New improvements for continuous fat and oil refining systems were made. Thurman (*U. S. 2,327,569*) invented a device for injecting refining alkali into the flowing oil of a continuous system. Carruthers (*U. S. 2,324,763*) designed new filtering and centrifuging portions of the equipment. Thurman (*U. S. 2,299,603*) arranged and standardized the apparatus so that the foots obtained from cottonseed oil would be a substantially completely saponified mixture of soap and detergent compounds, resulting from the thermal decomposition of proteinaceous material. Special centrifuges for the continuous removal of gums and resins from crude cottonseed oil were patented by Bloomer (*U. S. 2,319,970*).

An alcoholic solution of lye (Kelley and Cornell—*U. S. 2,305,619*) and the reaction product of alkali and yacca gum (Henderson—*U. S. 2,317,056*) were two newly developed alkali types of fat refining agents. Some of the losses from alkali refining were recovered by diluting the foots (Giles and Kelley—*U. S. 2,337,041*); the process releases some of the refined oil taken up by the foots. Spada (*Atti soc. nat. mat. Modena 71, 46*) improved the alkali refining process for the castor oil intended for lubricating uses, by breaking emulsions formed with paraffin oil additions. This not only reduced refining time but reduced the loss through emulsification which is ordinarily considerable with this oil. Spirk (*Chem. Listy 35, 275*) recommended organic acids as lactic, tartaric, etc. for removing metal compounds from oils. Traylor (*U. S. 2,334,850*) improved fats by treating them under ionizing conditions with metals above hydrogen in the electromotive series and an acid salt. Palm oil and wax sludges from steel mills were refined with potassium dichromate and an absorbent in vacuum at about 400°F. (Dunmire and Camelford—*U. S. 2,316,774*). A selective solvent refining method made use of liquefied normally gaseous hydrocarbons for removing free fat acids (Ewing—*U. S. 2,329,889*).

Tall oil was improved by new refining procedures. Ross and Percy (*U. S. 2,296,952*) refined the crude black raw material by saponifying, steaming to remove unsaponifiable and subjecting repeatedly to a salting out process. Phenols were recovered as by-products from the brine solutions. Brandt (*U. S. 2,308,431*) dissolved the unrefined product in hydrocarbon solvent and extracted dark color with sulfur dioxide. According to Mitchell (*U. S. 2,324,012*) the

sulfur dioxide extract could be processed to yield phytosterols. Trimble (*U. S. 2,310,046*) used furfural as an extractant for color. Borglin (*U. S. 2,315,584*) recorded that treatment with oxalic acid removed metal salt impurities and color. Special heat treatments to produce stable and clear tall oil were the subject of some inventions (Oliver and Palmer—*U. S. 2,330,792*; Hasselstrom—*U. S. 2,311,386, 2,334,762*). The new methods for separation of fat acids from the resin acids involved a distillation and a crystallization procedure. Färber (*U. S. 2,337,235*) added 0.1 of the neutralization equivalent of caustic before distillation. Brown and Scott (*U. S. 2,294,446*) selectively esterified the fat acids with a lower alcohol, crystallized out part of the resin acids, saponified the remaining resin acids and washed out the soaps formed. Valentine (*U. S. 2,309,483*) hydrogenated the high boiling constituents of tall oil intended for use as a perfume base.

For economical decolorization of oil, Glenn (*Can. 409,982*) recommended a partial decolorization with 0.1-1% fuller's earth, followed by heating to 350-400°F. and subjecting to vacuum and steam to complete the decolorization. Buxton (*U. S. 2,306,776*) claimed both deodorization and bleaching were obtained in fish oils by treatment with a mixture of carbon and hydrocarbon solvent. Active magnesium oxide prepared by calcinating magnesium carbonate was patented as a new bleaching agent for fats and oils (Winding—*U. S. 2,322,555*). The bleaching of oils with the aid of enzymes promoting oxidation was also patented (J. R. Short Milling Co.—*Ger. 711,242 Cl. 120*). Two processes for recovering fats adsorbed on bleaching earths depended on addition of water to effect a displacement of adsorbed oil with water (Kelley—*U. S. 2,314,621*; Lachle—*U. S. 2,316,005*). A method for bleaching clay regeneration comprised roasting and rehydrating by treatment with water under pressure and temperature of 275-650°F. (Crowley—*U. S. 2,319,185*). Other communications on bleaching recommended distillation (Erdheim—*Allgem. Oel- u. Fett-Ztg. 38, 360*; Brücke—*Ger. 723,436, Cl. 23a*). Distillation was said to be more economical than the use of earths for highly acid oils.

An experimental laboratory deodorizer for fats and oils was designed by Bailey and Feuge (*Ind. Eng. Chem. Anal. Ed. 15, 280*). Renner (*U. S. 2,316,621*) recorded that commercial deodorization could be accomplished by treatment with the freed cell contents of yeast. Two methods for deodorizing fish oils were based on partial hydrogenation at low temperatures and high pressures (Hennessy—*U. S. 2,321,913*; Blaso—*U. S. 2,311,633*).

A review on hydrogenation plants and processes was prepared by Mitchell (*Chem. Age London 48, 471, 495*). In new hydrogenation apparatus designed by MacLean (*U. S. 2,313,654*), hydrogen gas was discharged into the oil charge by means of a rotating impeller; this device thus served for stirring and dispersing the gas through the oil. A theoretical investigation on hydrogenation demonstrated that with C_{18} fat acids containing one double bond the hydrogenation rates were progressively greater for those acids with the double bond further from the carboxyl radical. In methods proposed for hydrogenation of tall oil, the oil was first treated with heavy metal compounds to remove sulfur (Oliver and Palmer—*U. S. 2,317,797*) or the oil was heated first

with hydrogenation catalyst at 100-130°C. and then hydrogenated while in volatile solvent solution (Dressler and Vivian—*U. S. 2,336,472*). Hydrogenated oils were bleached by treating them with hydrogen at 250°C. in the presence of silver or chromium catalysts (Paterson—*U. S. 2,307,065*). Research on 20% nickel formate-kieselguhr catalyst for hydrogenation showed that the optimum temperature for reduction was 300°C. and the activity was half of that of the carbonate catalyst (Joglekar and Jatkar—*J. Indian Chem. Soc., Ind. & News Ed. 5, 4*). The activity of nickel carbonate-kieselguhr was maximum between 10 and 20% nickel after which it decreased to about half as active at 30% nickel (*Ibid.* 8). Paleni (*Chimica e industria Italy 24, 3*) selectively hydrogenated olive oil to a product free of linoleic acid and consisting of oleic acid and the saturated acids originally present, by the use of a catalyst containing 4.5% copper, 4.5% nickel and 91% kieselguhr and at a working temperature of 220°C. Tang and Chang (*J. Chinese Chem. Soc. 9, 173*) recorded that a nickel-chromic oxide catalyst containing small amounts of tungsten oxide, molybdenum oxide, cobalt oxide and manganese oxide was highly active for hydrogenation of peanut oil. A nickel-copper catalyst prepared by Borkowski and Schille (*U. S. 2,320,063*) by drying the carbonates at 200°C. and heating and reducing the residue in mineral oil at 360°F. was particularly suitable for hydrogenating cottonseed oil.

A patent on hardening fats by isomerization dealt with the use of nitrogen oxides as catalysts and reaction temperatures below 50°F. (Eipper—*U. S. 2,310,225*).

General papers giving reviews or discussions of processes for splitting fats were prepared by Lindner (*Fette u. Seifen 49, 862*) and the editors of "Chem. & Met." (*Chem. & Met. Eng. 50, No. 9, 132*). A comparison of Twitchell and enzyme methods of splitting showed that 89-96% hydrolysis occurred in 14 hours by the former method while with the latter 96 hours treatment was required to yield 90% splitting (Goswami—*India Dept. Com. Rept. Tech. Work Board Sci. Ind. Research 1940-41, 5*). Hoffman and Zeigler (*U. S. 2,319,929*) hydrolyzed low grade fats by saponifying, washed the soaps with brine and released the fat acids by acidulating. A saponification method was also patented for marine animal wax (Adams—*Can. 410,446*). A continuous process comprised means of counter flowing hot oil and water through a series of heating zones (Murphy—*U. S. 2,310,986*). In a similar process, Winer (*U. S. 2,313,692*) added nickel hydrogenation catalyst and hydrogen and obtained simultaneous continuous splitting and hardening. Eisenlohr (*U. S. 2,318,206*) patented a eutectic mixture of biphenyl and diphenyl oxide as a heating medium for the reaction coil of a continuous fat splitting apparatus in which splitting was accomplished at 260-340°C. and 3000-3500 lbs. per square inch pressure.

Means of fractionating fats or fat acids into fractions of different degrees of saturation by liquid-liquid extraction were assigned to Pittsburgh Plate Glass Co. (*U. S. 2,313,636, 2,316,512, 2,320,738*). One of the letters patents describes scheme and arrangement of apparatus for the purpose; the others list solvents with some of their properties as related to the process. Bull and Wheeler (*Oil & Soap 20,*

137) investigated the separation of soybean oil into saturated and unsaturated fractions by solvent crystallization from acetone. It was impossible to obtain fractions above 165 I value with a single crystallization. However, single crystallizations of the mixed free acids yielded as high as 50% of acids with I value of 180. Similar experience was recorded by Kleinsmith and Kraybill (*Ind. Eng. Chem.* 35, 674) who used liquid-liquid extraction as a means of fractionation. Both communications produced evidence for suggestions on the distribution of the fat acids in soybean glycerides. The data and information should be of value for manufacture of better technical or food oils from soybean oil. Experiences on crystallization of cottonseed and peanut oils from petroleum naphtha to produce good winterized products for salad oils and olive oil substitutes were recorded by Bailey, Feuge, Kraemer and Bauer (*Oil & Soap* 20, 129).

Among developments for distillation of fat acids, Crawford (*U. S.* 2,328,621) added petroleum wax (m.p. 120°F.) to the still charge to prevent polymerization. Dreyfus (*U. S.* 2,331,965) released fat acids from their salts before distillation by treatment with boric acid, hydrated alumina and silicic acid; and others (Eckey—*U. S.* 2,333,712; Lee—*U. S.* 2,297,004, 2,290,373; Potts—*Can.* 414,679; Williams—*Brit.* 546,414; Mills—*Brit.* 546,416) designed new stills or parts thereof.

The patents relating to molecular distillation during the year were on procedures used for concentrating vitamin D (Hickman—*U. S.* 2,316,068, 2,326,644) and tocopherols (*Ibid.*—*Brit.* 548,731). An annual supplement to a bibliography on molecular distillation has aided in organizing the literature on the subject (Todd—*Oil & Soap* 20, 205). The physical and chemical characteristics of fractions obtained by exhaustive fractional distillation of soybean oil with a molecular still were tabulated by Detwiler *et al.* (*Ibid.* 108).

Some oils are processed particularly for recovery of valuable constituents which appear only in small quantities. The new patents on concentration of fat-soluble vitamins were assigned to National Oil Products Co. (*U. S.* 2,311,517, 2,311,554, 2,318,747-9, 2,324,063). The innovations included removing the free fat acids, saturated glycerides and nitrogenous compounds by crystallization from an organic solution of the oil; in the separation of the unsaponified fraction, water was added to precipitate the soap from a solution of the liver oil soaps in organic solvents; emulsions formed during the process were broken by heating to flocculate the soap; and the unsaponifiable material was further concentrated to a higher vitamin potency fraction by adsorption on calcium oxide and elution with polar and nonpolar solvents. The products were incorporated in an aqueous medium by dissolving in ethyl alcohol, removing insoluble material precipitated at -20°C. and then mixing with the aqueous medium. The ethyl alcohol soluble portion of gum tragacanth also improved solubility.

There was some activity toward recovering sterols during the processing of oils. Aseh (*Brit.* 547,324) and Wolf (*Brit.* 547,153) adjusted their fat acid distillation process so that highly concentrated sterol fractions were recovered. A process by Baxter (*U. S.* 2,317,353) comprised precipitating with sulfuric acid

a fraction from the oils while in alcohol solution at 2°C. This fraction was distilled to concentrate the tocopherols. Baxter with coworkers (*J. Am. Chem. Soc.* 65, 918) also separated the various tocopherols by molecular distillation, prepared certain ester derivatives and described their properties. Phytosterol glucosides were separated from vegetable oil refinery foots by acidulating and heating; after standing the acidulated foots separated into 3 layers, the intermediate contained the phytosterol glucosides (Langlois—*U. S.* 2,366,547). Another method of concentrating sterols from foots depended on selectively extracting with organic solvents (Kruse *et al.*—*U. S.* 2,296,794). An isolation method for cholesterol comprised dissolving in fat-dissolving non-alcoholic solvent and treating the solution with hydrochloric acid to precipitate the acid addition products of the sterol (Yoder—*U. S.* 2,322,906). Schoeller (*U. S.* 2,323,584) oxidized several sterols to yield neutral unsaturated polycyclic ketones that exhibited some sex hormone activity. A review on vegetable sterols was prepared by King (*Univ. Microfilms, Ann Arbor, Mich., No.* 429, 78 pp.).

Various phases of the chemistry of drying oils were treated in several communications. Thorough reviews appeared on the physical chemistry of lacquer (Schäfer—*Fette u. Seifen* 49, 481, 585, 653, 713, 787, 854), molecular growth of coating materials (Kaufmann *et al.*—*Ibid.* 102, 321, 841) and synthetic methods of preparing drying oils (Pegorari—*Ibid.* 733). Publications with general information contained notes on polymerization and drying of oils (Drinberg and Ryashentsev—*J. Applied Chem. U. S. S. R.* 15, 346), a theoretical discussion of the reactions involved in oxidation of linseed oil (Hajo—*Melliand Textilber.* 23, 89) and a description with favorable recommendation, of fractionated oils for the paint industry (Fawcett—*Paint, Oil & Chem. Rev.* 106, No. 6, 9).

Triebes (*Ber.* 75B, 203, 331) studied the autoxidation of drying oils to obtain some insight into the mechanism of the oxidative bodying and the drying of coating oils. Curves on the absorption of oxygen by films of methyl esters of the fat acids were first prepared. The behavior of the methyl esters on glass wool in closed glass bottles filled with oxygen was next studied. Changes in viscosity during oxidation were also recorded. It was suggested that the films formed were of relatively low molecular weight for they dissolved easily in methyl alcohol and acetone to yield solutions of low viscosity. The formation of water during drying could have resulted from (1) the production of an oxide group from two hydroxyl groups, (2) the oxidation of an hydroxyl group to a carbonyl group or (3) the cleavage of an hydroxyl group with formation of a double bond. In the autoxidation of linoleic acid 1 or 0.5 molecule of water per molecule of acid could be eliminated depending on the conditions.

Useful information on paint manufacture was gained from 4 investigations. Low viscosity straight bodied paints were superior to unbodied oil or to those containing heavy bodied and untreated oil (Nelson—*Official Digest Paint and Varnish Production Clubs, No.* 225, 98). Procedures for using esterified tall oil fat acids with linseed, wood, perilla and oiticica oils and several commercial resins in the manufacture of spar and floor varnishes were developed

by Niesen (*Farben-Chem.* 11, 149). Hollihan and Briggs (*J. Phys. Chem.* 47, 30) tabulated viscosity data on dilute carbon tetrachloride solutions of tung oils which had undergone various degrees of non-oxidative heat treatment. Evidence was presented suggesting that the bodying process occurs in successive stages.

Reynolds and Kellog (*U. S.* 2,307,158) discovered that the addition of 0.01-0.5% tri-*p*-cresyl phosphite to coating compositions containing tung oil reduced "skinning" and improved weather resistance. A drying oil mixture suitable for ink, paint or varnish, patented by Allen and Haury (*U. S.* 2,317,663), is a mixture of unsaturated ketone condensation product of acetone and tung oil.

Coatings of oiticica or wood oil were rendered non-wrinkling and gas proof by an ingredient made by reacting a monoglyceride of the oils with a polybasic organic acid (Krumbhaar—*U. S.* 2,319,507). Oiticica oil was rendered non-frosting and quick drying by cooking at 300°F. in the presence of a small amount of boron trifluoride (Pratt and Berger—*U. S.* 2,316,187). Gardner (*U. S.* 2,318,304) proposed that oiticica oil could be used with best results when mixed with dehydroxylated castor oil. The mixtures were heated with a small amount of alkali acid sulfate.

Two patents dealt with preparation of varnish oil from linseed oil or other non-conjugated oils. Rust (*U. S.* 2,306,281) heated the oil with maleic anhydride in the presence of an aromatic sulfonic acid. Novak (*U. S.* 2,318,009) changed the oils to conjugated types by controlled heating. The treatment produced a product with substantial absence of frothing or color development characteristics. Oils having conjugated systems were produced from soybean oil by removing some of the oleates, oxidizing to produce hydroxyl groups and eliminating water by pyrolysis (Boone—*U. S.* 2,308,152).

Several means of improving semi-drying oils or deriving drying oils therefrom were investigated. Cowan and Falkenberg (*Oil & Soap* 20, 153) separated dimers from dimerized acids of such oils and synthesized drying oils from these by esterification with polyalcohols with or without polybasic organic acids. The oil films prepared from these dried faster and were more water and alkali resistant than linseed oil films. Morris, Rusoff, Miller and Burr (*J. Biol. Chem.* 147, 273) charted possible changes in unsaturated fat acids during the heat treatment involved in vacuum fractional distillation. The drying character of bodied menhaden oil was improved by molecular distillation until 10% of the oil was distilled (Morse—*U. S.* 2,311,681). Linseed and soybean oil could be bodied without "break" formation by running the unbodied oil into an oil preheated to bodying temperature (Jenkins—*U. S.* 2,317,915). Drying and semi-drying oils were improved by addition of 20-50% of non-drying isano oil (Priester—*Ger.* 712,179 *Cl.* 22h).

New methods of dehydrating castor oil to yield a drying oil substitute were invented. Priester (*U. S.* 2,309,273) heated the oil with a small percent of alkali metal pyrosulfate. Colbeth (*U. S.* 2,317,361-2) reacted the castor oil with boron compounds and decomposed these by rapidly heating to yield unsaturated linkages. Nessler (*U. S.* 2,336,186) acylated the hydroxyl groups and pyrolyzed off acyl acids to yield a dehydrated fatty oil. Walton and Jordan (*U. S.* 2-

308,222) found dehydration occurred by heating the castor oil with fat acids having no conjugated double bonds. The free acids were then esterified with polyhydric alcohol.

Products (Except Detergents)

A report on an investigation of the mechanism of shortening action of commercial fats was issued by the Bureau of Home Economics of the U. S. Department of Agriculture (Hornstein, King and Benedict—*Food Res.* 8, 1). There was no correlation between shortening power and I value or congeal points. It was suggested that the shortening power of a fat may depend either upon the amount of liquid glycerides present or upon the ratio of liquid to solid glycerides. A commercial prime steam lard ranked highest in shortening power. A vegetable oil mixed type shortening consisting of a small part of fully hydrogenated cottonseed oil and a much larger proportion of oil ranked next. A hydrogenated mixture of vegetable oils was slightly lower than the mixed type, while a hydrogenated cottonseed shortening was considerably lower and only slightly better than leaf lard. Hydrogenated lard was superior to the hydrogenated cottonseed oil, but not as good as the prime steam lard, the mixed type vegetable shortening or the hydrogenated mixture of vegetable oils. Butter oils showed higher shortening power at higher temperature than at lower, but were inferior to prime steam lard, the mixed type vegetable, and the hydrogenated mixture of vegetable oils. Margarines showed the poorest shortening power.

Blaso (*U. S.* 2,307,756) hydrogenated fish oils at 60°C. and 200-500 lbs. per sq. in. pressure to a final I value of 60 to produce a shortening rich in vitamins. An apparatus for improving the texture of shortening by incorporating air into the fat was designed by Harrington (*U. S.* 2,306,601). An improved shortening patented by Griffith (*U. S.* 2,332,074) contained equal parts of shortening fats melting at 115-120°F. and 140-160°F. and dispersed therein dry disrupted starch grains. A new dry shortening comprised special mixtures of flour, corn syrup and shortening (Heyman—*U. S.* 2,332,513). A patented baker's utensil coating contained hydrogenated soybean oil, olive oil, almond oil and beeswax (Wassermann—*U. S.* 2,309,488).

Developments on shortening improving agents dealt with lecithin products. A dry lecithinated baking ingredient was made by grinding a mixture of lecithin and granulated sugar (Fitzpatrick and Wagner—*U. S.* 2,334,401). Christlieb (*Ger.* 708,805 *Cl.* 20) prepared lecithin for baking uses by dissolving it in lactic acid. Rosenthal (*Ger.* 720,110 *Cl.* 12a) dispersed lecithin in water by treatment with sulfur dioxide. Glabau (*Bakers Wkly.* 118, No. 19, 47) demonstrated that lecithin was a good shortening extending agent in sugar cookie mixtures.

Two new processes for emulsification in margarine manufacture were patented. Miller *et al.* (*U. S.* 2,330,986) devised means of emulsifying the aqueous ingredients with fat while the latter was in a supercooled liquid condition. Green (*U. S.* 2,325,393) worked the aqueous constituents into hardened oils and then worked liquid glycerides into the mixture.

The deep fat frying qualities of several oils were investigated. Arenson and Heyl (*Oil & Soap* 20,

149) recorded that neither peroxide value nor active oxygen hour rating was indicative of the quality of the frying fat for doughnut manufacture. When 0.5% free fat acids was added to the frying oil there was practically no prequality frying period; that is, the doughnuts began to develop normally almost immediately. In potato chips, shoe-string and French-frying tests by Grim and Eheart (*J. Am. Dietetic Assoc.* 19, 618), those fried in peanut oil showed significantly higher average palatability scores than those fried in cottonseed oil. The potatoes fried in peanut oil absorbed less fat and the oil showed a greater resistance to rancidity than cottonseed oil. Vail and Hilton (*J. Home Econ.* 35, 43) tabulated the smoke point and development of free fat acids during heating. The smoke point of 17 vegetable oils ranged from 190 to 245°C. and for 8 animal fats the figures were from 174 to 234°C. Percentage of free fat acids before and after heating showed a wide range. Means of improving the resistance of glycerides to foam when heated to high temperatures were devised by Black (*U. S.* 2,320,319, 2,322,186-7). These comprised adding calcium or magnesium soaps, or an alkali salt of a sulfonated fat or treating the fat with phosphoric acid prior to the removal of nickel hydrogenation catalyst.

Several emulsifier developments dealt with fat acid esters containing free hydroxyl groups. Gooding (*U. S.* 2,309,949) produced such esters in a fat by heating to 200-275°C. in the presence of 0.05-1.0% soap and about .05 to 2% by weight of glycerol. Such esters were also produced by heating fat acids and polyhydric alcohols with superheated steam (Jordan—*U. S.* 2,307,794). Black (*U. S.* 2,320,844) produced monoglycerides by heating fats and glycerol to 200-300°C. in the presence of 0.5-10% activated carbon. An emulsifier patented by Katzman *et al.* (*U. S.* 2,334,709) was a mixture of monostearin and the hydrochloride of a fat acid ester of monoethanolamine. A new compound prepared by Tucker (*U. S.* 2,329,166) was a fat acid ester, the alcohol part of which was an ester of glycolic acid and glycerol. There was at least one free hydroxyl group for each fat acid in the compound. Mannide, hexide and hexitan mono-esters of fat acids were patented as emulsifiers (Brown—*U. S.* 2,322,820-2). Polyalcohol fat acid esters containing free hydroxyl groups were also patented as ore flotation agents (Erickson and Jayne—*U. S.* 2,312,466).

Lotzkar and Maclay (*Ind. Eng. Chem.* 35, 1294) fostered the use of pectin as a substitute for such imported products as tragacanth, karaya, acacia and carob gums which are difficult to obtain at the present time. Pertinent characteristics of emulsions prepared from pectin were compared with those made from the above named gums. Shelton and Huyek (*U. S.* 2,303,236) found that precipitated magnesium trisilicate was a good emulsifying agent for cod-liver oil. Marriot (*Brit.* 546,998) used a mixture of fat acids, organic bases and benzyl alcohol as an emulsifier. Foaming was suppressed in petroleum oil-in-water emulsions by addition of chromium oleate (Liberthson—*U. S.* 2,307,744).

Swift, Rose and Jamieson (*Oil & Soap* 20, 249) prepared purified methyl linoleate by chromatography. An "Alorca" alumina with the most efficiency for recovery of the desired esters was selected from samples of varying activities prepared by exposing the alumina to moist air and using Brockmann's dye

test method to evaluate activity. In work with the methyl esters of cottonseed oil fat acids a 200:1 ratio of alumina:methyl linoleate was preferred; and 1400 cc. of solvent was necessary to remove fractions of I value less than 142. A final fraction had an I value of 172 and was 98.6% pure methyl linoleate. Sebacic and hydroxydecanoic acids were prepared by Davis and Dombrow (*U. S.* 2,318,762) from ricinoleic acid by heating with alkali in a high boiling point inert hydrocarbon oil. Polymethylene dicarboxylic acids of 11 and 12 carbon atoms were derived from 12-hydroxystearic acid by oxidizing with nitric acid in the presence of vanadium. According to Myer (*Soap* 19, No. 11, 37) azelaic and pelargonic are 2 dibasic acids derived from fats which are already available in commercial quantities. Their use is now restricted, but post-war applications will probably be numerous.

Swern, Stirton, Turer and Wells (*Oil & Soap* 20, 224) prepared the lauric, myristic, palmitic and stearic acid esters of *l*-ascorbic and *d*-isoascorbic acids. The antioxidant properties of these are now being studied. Beta monoglycerides of caproic and caprylic acids were prepared by synthesis of the benzylidene intermediates and reduction to the monoesters (Daubert, Fricke and Longenecker—*J. Am. Chem. Soc.* 65, 1718). The melting point and index of refraction of these derivatives were determined. Several new derivatives of chaulmoogra acids were prepared and some of their physical constants recorded (Buu-Hoi and Cagniant—*Bull. soc. chim.* 9, 99, 104, 107, 357). Paranjpe (*J. Univ. Bombay* 11, Pt. 3, 120) applied the Nencki reaction for synthesis of acylphenols to the preparation of many fat acid: phenol derivatives. Some physical constants of these derivatives were also recorded.

Schuckmann (*U. S.* 2,332,834) reduced fat acids to alcohols by hydrogenation in presence of copper and cadmium catalysts. Davis and Hodgson (*J. Soc. Chem. Ind.* 62, 128) improved the Blaise procedure for reduction of acids to aldehydes, to the extent that they obtained a 62.3% yield of undecaldehyde from lauric acid.

Apparatus for the commercial production of nitriles by the reaction of fat acids or their esters with ammonia was designed by Potts and Christensen (*U. S.* 2,314,894). A commercial process of converting oleic acid into mixtures of nonylamine and *w*-octylamino acid amides involved dissolving oleic acid in benzene, treating with ammonia to convert the acid into oleamide and treating the solution with ammonia under pressure with a hydrogenating catalyst (Bersworth—*U. S.* 2,294,442). The use of fat acid amines in froth flotation processes for concentrating non-metalliferous ores was represented by 2 new patents on the subject (Ralston and Segebrecht—*U. S.* 2,313,360; Christmann *et al.*—*U. S.* 2,321,186).

Many new fat derivatives were patented for use as antiseptics, disinfectants, insecticides, and mothproofers. Harris' (*U. S.* 2,321,594-5) and Katzman and Epstein's (*U. S.* 2,322,783) products were hydrochlorides of fat acid amine partial esters of polyhydroxy alcohols. The use of esters of *N*-thiomethylamide or similar amides and fat acids was claimed by Austin and Frank (*U. S.* 2,323,111). Leuchs' (*U. S.* 2,317,999) compounds for this purpose were quaternary ammonium preparations containing both fat acid and aromatic radicals. Similar compounds but in addition carrying a sulfonyl radical were said

to be suitable for the above uses and were softeners for lacquers (Hentrich and Schirm—*U. S. 2,292,998*). A mothproofing compound was produced by action of fat acid chlorides on amino diphenyl-ether-sulfonic acids (Martin and Zaeslin—*U. S. 2,309,969*).

The petroleum industry showed considerable activity towards development of petroleum demulsifiers (Alien Property Custodian—*U. S. 2,307,058, 2,317,726, 2,323,344*; Petrolite Corp.—*U. S. 2,307,424-5, 2,309,243, 2,309,935, 2,310,679, 2,324,488-94, 2,329,699-703, 2,330,472-4, 2,333,769, 2,334,389-91, 2,335,262, 2,335,489*; Standard Oil Development Co.—*U. S. 2,312,731-5*; Socony Vacuum Oil Co.—*U. S. 2,317,050*; Wayne—*U. S. 2,318,034-5, 2,321,056*). The compounds used were a variety of surface active derivatives of fats and oils. The direct use for them was to break petroleum emulsions in calcareous oil-bearing strata; thus releasing the oil for recovery. Amine derivatives of fat acids were also used to clarify turbid waters (Ralston and Pool—*U. S. 2,315,734*).

In the field of waterproofing fabrics Sauer (*U. S. 2,310,873*) patented the use of reaction products of formaldehyde, anhydrous hydrogen chloride and hexanebis-fat acid amide; Nothum and Licata (*U. S. 2,307,852*) found aluminum ricinoleate suitable, and Doser *et al.* (*U. S. 2,328,431*) impregnated the fabric with an emulsion containing wax, fat acids, zirconium oxychloride and a condensation product of oleyl alcohol and ethylene oxide. Kestenbaum (*U. S. 2,309,907*) improved furs by applying a liquid containing fat acids and formaldehyde and thereafter applying heat to form a water-insoluble coating on the hairs.

Many fat derivatives were manufactured for use in protective coatings and plastics. A product manufactured by Pollack (*U. S. 2,306,139*) was polymerized fat alcohol esters of crotonic acid. Kester (*U. S. 2,337,051*) produced a resin by reacting a naphthalene formaldehyde condensation product with a fatty oil in the presence of polystyrene. Chittick and Schlandt (*U. S. 2,330,798*) prepared a mastic from petroleum polymers by reaction with sulfur in the presence of fatty oil. Hanford (*U. S. 2,312,966-7, 2,323,806, 2,320,232*) synthesized fat acid amides and polyamides for use as intermediates for the manufacture of fabric coatings, threads, films, etc. The manufacture of special alkyd resins from castor oil was patented by Waldie (*U. S. 2,319,022*). The plasticizers, softeners or modifiers for resins and plastics were: the reaction product of alkyl urea and a saturated fat acid (Stage and Harvey—*U. S. 2,330,087*); ricinoleic acid derivatives of alkylene glycol monoalkyl (Carruthers—*U. S. 2,310,395*) and aliphatic esters of diacetoxystearic acid (Gruber and Machemer—*U. S. 2,332,849*).

The literature also yielded a group of miscellaneous uses for fat derivatives. Fat acids or metallic soaps were used to disperse ultramarine coating for trade marking coal (Dieterle—*U. S. 2,323,748-9*). Condensation products of pyrenes, having hydroxyl groups attached to the nucleus, and fat acid halides were fluorescence dyes for fats and waxes (Neresheimer and Vilsmeier—*U. S. 2,295,963*). A therapeutic epinephrine derivative contained a lauric acid radical (Gustus—*U. S. 2,332,075*). A lauryl sulfate-tryptose broth was recommended for use in the detection of coliform organisms in water (McCrady—*Am. J. Pub. Health 33, 1199*). Concrete

structures were rendered water-repellent by treatment with fat acid amines (Ralston and Vander Wal—*U. S. 2,317,301*). Fat acid amines were also used to improve the plasticity of plaster and other clay mixtures (Ralston and Hoffman—*U. S. 2,320,009-10*). In the cosmetic field there appeared a solid perfume, the carrier being a mixture of saturated fat alcohols (Berry—*U. S. 2,300,769*) and a hair tonic contained quaternary ammonia salts derived from fat acid amines (Deutsche Hydrierwerke A-G.—*Ger. 726,665 Cl. 30h*). Three wax compositions contained fat derivatives among their ingredients (Hill—*U. S. 2,320,644-5*; Olson—*U. S. 2,331,925*). A patented core oil formula contained naphthenic extracts, drying oil and a plasticizer (Crawford—*U. S. 2,328,622*). Kreitser and Tsvetova (*Liteino Delo 12, No. 2, 31*) fostered the use of cottonseed oil for hardening cores.

Several fatty oil lubricant products were developed. A textile oil comprised petroleum oil and 5-20% of a di-ester of glycerol with fat acid and naphthenic acid (Goodings and Marshall—*U. S. 2,336,087*); another continued petroleum oil, mahogany sulfonates, rosin soap and triethanolamine (Langer—*U. S. 2,328,727*). Colbeth (*U. S. 2,308,355*) patented the use of synthetic oleic acid with the double bond on the 12th carbon atom as a textile lubricant. A wire drawing lubricant comprised sulfonated tall oil and sulfonated sperm oil (Nil—*U. S. 2,326,387*). The cutting oil patents mostly concerned reacting sulfur with the oils. Pfestorf (*Ger. 709,815 Cl. 12a*) heated unsaturated fat acids with sulfur at 250°C. and reacted the product with alkali sulfides. Abramowitz and Beach (*U. S. 2,313,611*) reacted the fatty oils with a mixture of sulfur chloride and ammonia. Evers *et al.* (*Ger. 707,660, Cl. 23c*) prepared a solution of sulfur in suitable solvent having a boiling point lower than the oil to be sulfurated; this solution was mixed with the oil and the solvent was distilled off. Crawford's patent (*U. S. 2,828,620*) on sulfured cutting oil dealt with the proportion of constituents used. Mixtures of train oil, bone oil, petroleum oils and wood tars were recommended as belt lubricants (Micksch—*Allgem. Oel- u. Fett-Ztg. 38, 454*).

The use of nonalkali metallic soaps and other fat derivatives in lubricating oils and greases was described in many letters patents (Am. Cyanamid Co.—*U. S. 2,325,040*; Colgate-Palmolive-Peet Co.—*U. S. 2,319,405*; Internatl. Lub. Corp.—*U. S. 2,308,599*; Lubri-Zol Development Corp.—*U. S. 2,308,622*; Standard Oil Development Co.—*U. S. 2,296,315, 2,307,891, 2,318,668, 2,334,565*; Standard Oil Co.—*U. S. 2,323,670*; Socony-Vacuum Oil Co.—*U. S. 2,335,261*; Texas Co.—*U. S. 2,334,239*; Union Oil Co.—*U. S. 2,291,384*). The use of soybean lecithin as an oxidation inhibitor for lubricating oils was recommended from 2 sources (Armfield—*U. S. 2,316,852*; Jacobs and Othmer—*Ind. Eng. Chem. 35, 883*).

Activities on the pyrolysis of fats and oils were the invention of apparatus for the process (Stalman—*Ger. 723,437, Cl. 23d*), reports on experiences in making motor fuels from cottonseed oil (Lo and Tsai—*J. Chinese Chem. Soc. 9, 164*) and coconut oil (Venkataraman and Parekh—*India Dept. Com. Rept. Tech. Work Brd. Sci. Ind. Res. 1940-41, 6*), and an analysis of the products formed on heating tristearin and triolein at low pressure to a temperature of about 410°C.