Report of the Literature Review Committee

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

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INTRODUCTION

This review of the 1962 literature was compiled from current, original publications and from abstracts of publications that were not available in the original. Judged from the number of references included in each section, the contributions on the subject under review were about the same as last year. The foundation for the establishment of an ''oleochemical'' industry is being firmly laid as the result of increasing researches on fatty acids and their derivatives to obtain various products for technical and other uses. Studies on the importance of cholesterol and different types of fat in heart disease are receiving considerable attention. The plethora of studies in the area of diet, blood and liver lipids, and lipid transport continues. Gas-liquid chromatography and thin-layer chromatography continue to dominate the fatty acid analytical scene.

SOAPS, SURFACTANTS, AND DETERGENTS

MANUFACTURE

Processes

SOAP. The Mazzoni process for continuous and automatic fine soap production was described (Fock, *Fette Seifen Anstrichmittel 63,* 695) as was the similar ''SC'' process (Zacchi, *Grasas y Aceites 12,* 239).

U.S.S.R. autoclave saponification process using soda ash was described and details of theory given (Maftei, *Ind. Aliment Produse Vegetale 12,* No. 3, 73). A continuous washing process for glycerol removal from kettle soap was produced by reacting carbon dioxide with palm oil fatty acid diethanolamide (Kawakami, *Japan. 11,971 ('61*). White or light-colored soaps were produced from tallow or other dark oils by introducing a combination of a mercaptan and an acid synergist (Copenhauer, *U.S. 3,004,048*).

SULFONATION. Alkylbenzenes were sulfonated with sulfuric acid containing added sulfur trioxide (Ashimov, *Azerb. Khim. Zh. 1961, No. 4,* 23), while continuous sulfonation was achieved with sulfur trioxide from converter gas (Davidsohn, *3rd Congr.* 1,* 113; *Mfg. Chemist 32,* 500). Improved color was claimed by sulfonating dodecylbenzene in two stages (Conwell, *U.S. 3,007,961*).

Organic liquids were continuously and efficiently sulfonated in a venturi flow mixer using gaseous sulfonating agent (Henton, *Brit. 855,774*). Oils were sulfonated by atomizing the oil with a gaseous sulfur trioxide-dioxide mixture (Shekhter, *U.S.S.R. 138,615*).

REFINING. Alkylbenzenesulfonate with low inorganic salt content is claimed through treatment with dilute lower alcohol (Pengilly, *U.S. 3,031,498*). Similarly low inorganic salt content is claimed by salting neutralized alkylbenzenesulfonate with NaCl, Na_2SO_4, etc. (Richter, *Ger. 1,131,662*). Similar salt treatment is claimed for sulfonated alcohols or alkylaromatic hydrocarbons (Bespyatov, *U.S.S.R. 138,247*). Improved properties were obtained with a sulfonated product (Gutman, *Novosti Neft. i Gaz. Tekhn. Neftepererabotka i Neftikhim, 1961, No. 3,* 10).

Sulfonated dimethylheptyl maleate was decolorized by treatment in aqueous alcohol with a poly- or orthophosphate (Anderson *U.S. 3,033,896*). Alkylbenzenesulfonates are improved in color by adding $HCONH_2$ before sulfonation (Schar, *U.S. 3,042,713*). Crude sulfonic acids or their derivatives are bleached by treatment with di- or trichlorocyanuric acid or their salts (Merkel, *Ger. 1,127,352*).

The odor of sulfoalkyl esters of fatty acids was improved by removing the phosphine odor by treatment with hydrogen peroxide (Schenck, *U.S. 2,999,871*).

MISCELLANEOUS. Separation of more effective fractions of alkylbenzenesulfonates was attained by treatment with a methanol solution of urea (Piasek, *Przemysl Chem. 41,* No. 1, 38). Improved surfactants were obtained by fractionation of the alkylbenzene (Muchinskii, *Izvest. Akad. Nauk Turkmen. S.S.R., Ser. Fiz-Tekh, Khim. i Geol. Nauk 1961, No. 4,* 73).

Beta-sulfoethyl esters of fatty acids are continuously produced by reacting a finely divided reactant with liquid fatty acid chloride in a stream of hot gas (Tiedemann, *U.S. 3,040-073*).

Methods for surfactant preparation through oxidation were reviewed and paraffinic kerosene fractions were peroxidized to obtain the sulfonated products directly (Kleng and Brincoveanu, *Rev. Chim.* (Bucharest) *10, 214*).

Hydroxyalkylamides of fatty acids are prepared by direct reaction of hydroxyalkylamines with fatty acids and conversion by alkali metal methylate (Ernst, *U.S. 3,024,260*). Lauric diethanolamide was prepared from methyl laurate and

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H. BOOK REVIEW

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* Vortraege Originalfassung International Kongress Grenzflaechenaktive Stoffe, 3, Cologne, 1960.

diethanolamine by a continuous high temperature process (Monick, *JAOCS 39,* 213). Fatty alkanolamides were prepared by reacting dialkanolamines with fatty triglycerides, then refining by phosphoric acid addition and separation of this phase (Lohr, *U.S. 3,040,075*). A process for salt-free preparation of N-acyltaurines was described (Schenck, *U.S. 2,987,526*).

Improved conditions for ethylene oxide addition to alcohols were achieved by using a tertiary amine catalyst (Oshiro et al., *Kogyo Kagaku Zasshi 64,* 1588). Addition of ethylene oxide under pressure to a fatty alcohol or an alkyl phenol had slightly increased the rate of addition but had no effect upon degree of polymerization (Nagase, *Kogyo Kagaku Zasshi 64,* 1043). Polyoxyethylene derivatives of rosin acids, rosin alcohols, or other derivatives were improved by solubilizing them during the polymerization (Wilson and Knox, *U.S. 3,041,326*).

Metal carbonates and alkali in a wt. ratio of 1–10:1 are used to neutralize detergent sulfuric or sulfonic acids (Taylor and Rao, *Brit. 889,040*).

An alkylbenzenesulfonate mixture with various salts and sodium tripolyphosphate may be spray dried under specified conditions (Monick, *U.S. 3,055,835*).

A process for manufacturing anhydrous ammonium soaps was described (Reiling, *U.S. 3,053,867*).

Raw Material

Alkylbenzene as a raw material for detergent production was reviewed (Garcia, *Ing. Quim.* (Mexico) *6,* No. 65, 34). Isodecylbenzene was produced by treating benzene with diamylene (Lyuter et al., *Tr. Groznausk. Neft. Nauchn.-Issled. Inst. 1959, No. 4,* 218). Alkylarenesulfonates were produced from oil shale and gas-benzene (Faingol'd et al., *Goryuch. Slantsy, Khim. i Tekhnol., Akad. Nauk Est. S.S.R., Inst. Khim. 1961, No. 4,* 107, 115, 126). Alpha-olefins from cracked paraffins were used to produce alkylbenzenesulfonates (Potolovskii et al., *Primenenie Poverkhn-Aktivn. Veshehestv v Neft, Prom. Ufinsk. Neft. Nauchn.-Issled. Inst., Tr. Pervogo Vses. Soveshch. 1961,* 182). Thermally cracked gasoline was used as a raw material for alkylbenzenesulfonate production (Kolesnikova and Kirillov, *Tr. Bashkirsk. Nauchn.-Issled. Inst. po Pererabotke Nefti. 1960, No. 4,* 82). Alkylarenesulfonates and alkyl sulfates were prepared by treating oil products with sulfuric acid and oleum (Ben'kovskii et al., *Trudy Inst. Nefti, Akad. Nauk Kazaki S.S.R. 4,* 179). Alkylbenzenesulfonates and secondary-alcohol sulfates were produced using products based upon the thermal processing of Baltic oil shale (Koll, *Goryuch. Slantsy, Khim. i Tekhnol., Akad. Nauk Est. S.S.R., Inst. Khim. 1961, No. 4,* 91). Pyrolytic dipentene was used in the preparation of sulfonated p-menthane (Pospisil, et al., *Chem. Prumysl 12,* 117).

Acid surfactants from heavy petroleum oils were produced by selective adsorption and subsequent recovery (Groszek and Lodwick, *Brit. 883,491*). Phenols from peat oil were reacted with ethylene oxide (Dmitriev et al., *Forfyanaya Prom. 38,* No. 6, 24). Phenol fractions of shale or coal distillation were treated with ethylene oxide to produce surfactants (Gadaskina and Reidkovskii, *Primenenir Poverkhn.-Aktivn. Veshchestv v Neft. Prom. Ufinsk. Neft. Nauchn.-Issled. Inst., Tr. Pervogo Vses. Soveshch.* 1961, 153). Autothermally cracked heavy petroleum oils were used for the preparation of sulfonated surfactants, or nonionics by further reaction with ethylene oxide (Blouri, *3rd Congr.* 1,* 108).

Microcrystalline waxes may be air oxidized to produce carboxyl containing products (Zalichi and Groszek, *Ger. 1,120,441*). Soybean soapstock is used in making potash soap (Bestraten, *Lipidos, 21,* 23). Glycerol ethers from rat fish liver oil can be used as surfactants (Sulzbacker, *Mfg. Chemist 33,* No. 6, 233). Anacardol from cashew nut-shell liquid was hydrogenated and sulfonated (Sethi et al., *Indian 71,754*). Fatty acids are used in preparing sodium N-tallow fatty acid-N-methyltaurate (Lamberti, *Ger. 1,120,461*), and for manufacturing sodium N-methyl-N-coconut oil fatty acid-taurine (Thomas Hedley & Co. *Brit. 888,323*). The waste filtrate from monochloroacetic acid production was reacted with, for example, stearylamine to form a surfactant (Matsuda et al., *Japan 824* ('62).

Synthesis

AMPHOLYTES. Cycloimidinium compounds with SO_3H instead of COOH groups are described (Mannheimer, *Soap Chem. Specialties 38,* No. 1, 62). Fatty acids may be condensed with aminocarboxylic acids to form surfactants some of which foam more than sarcosides. Other data are given (Graf et al., *3rd Congr.* 1,* 42). Claimed are carboxylic acid amides of N-aminoalkylene heterocyclic amines (Mannheimer, *U.S. 3,001,997*). Pure sodium lauroylsarcosinate may be made by reacting fatty acid anhydride with the salt of an alpha-amino acid (Lorentzen, *Ger. 1,127,359*). Poly-(DL-alanine dodecylamide) was prepared and has surfactant properties (Isemura et al., *Bull. Chem. Soc. Japan 34, 1236*). Ampholytes may be prepared by reacting albumin-fatty acid condensation products with formaldehyde and amines (Stelle and Ruhnau, *Ger. 1,059,912*). An ethylene oxide-containing product is described (Murai, et al., *Japan. 10,078* ('61). An ampholyte was produced by reacting alkylene oxides with protein degradation products (Ulsperger, *Seifen-Oele-Fette-Wachse 87,* 654). Surfactants were prepared by hydrolyzing sulfur containing proteins to give ampholytic products (Bieselaar et al., *Ger. 1,119,292*).

ANIONICS. Alkylarenesulfonates were prepared from polymers formed in the hydration of ethylene (Movsumzade et al., *Azerb. Khim. Zh. 1961, No. 3,* 61). Conversion of straight chain olefins to alkylarene compounds and sulfonation is claimed (Hale et al., *Brit. 874,555*). Alkane sulfonates are prepared from petroleum cracking giving terminal olefins (California Research Corp., *Belg. 612,074*). Alkylarene sulfonate surfactants from olefins, and a process for sulfonation are claimed (California Research Corp., *Ger. 1,098,936*). Homologous series of alkyl sulfates and alkyl ether sulfates were prepared and evaluated (Goette, *3rd Congr.* 1,* 45). The synthesis of aliphatic sulfonic acids was reviewed (Goethols, *Industrie Chimique Belge 27, 663*).

Monoalkyl sulfosuccinates were described as a new series of surfactants (Vallee, *3rd Congr.* 1,* 84). Sodium bis (4-methylhexahydrobenzoyl)sulfosuccinate synthesis was disclosed (Danek and Hell, *Czech. 99,269*). N-(sulfoalkyl)-alpha-alkenylsuccinimide salts were synthesized (Gaertner, *U.S. 3,029,250*). Unsaturated sulfonic acid ester syntheses were described (Friedrichsen and Distler, *Ger. 1,126,860*).

Sulfonated esters of pentaerthrytol or glycerol were described (Slominskii and Kozhevnikova, *Sbornik Statei o Robot. Ukrain. Nauch.-Isseldovatel. Inst. Maslozhir. Prom. 1958, No. 3,* 79). Sulfates of lauryl alcohol-ethylene oxide products may be neutralized with various amines (deJong et al., *U.S. 3,054,820*). A survey of known methods for monoglyceride synthesis was made, and the preparation of sulfated alkyl esters of various fatty acids described (Kaufmann et al., *Fette, Seifen, Anstrichmittel 64,* 245). Alkylglyceryl ether sulfonate synthesis was disclosed (Shyte and Korpi, *U.S. 3,024,273*). The preparation and properties of various fatty acid monoglyceride sulfonates were described (Kluge and Kuczorowski, *Fette, Seifen, Anstrichmittel 63,* 835). Alkyl esters of several fatty acids were prepared and evaluated (Kaufmann et al., *Ibid., 64,* 245). A series of sodium alkyl alpha-sulfopelargonates were synthesized and evaluated (Stirton et al., *JAOCS 39,* 55). The sodium salts of alkyl esters of alpha-sulfo fatty acids were synthesized and evaluated (Stirton et al., *JAOCS 39,* 168).

Carboxylic acid esters of 2-hydroxyalkane sulfonic acids were disclosed (Schenck, *U.S. 3,004,049*). Polyesters prepared by reacting unsaturated dicarboxylic acid or anhydride with a fatty acid, and a glycol, are sulfonated (Brandner and Ford, *U.S. 3,016,393*). Surfactants were synthesized comprising acidic esters of N-hydroxyalkylimides of polybasic carboxylic acids and their salts (Krause and Quaedvbig, *Ger. 1,112,075*).

A complex anionic surfactant was claimed (Mannheimer, *U.S. 2,993,918*). Sulfates and sulfonates of N-tert-amino-alkylcarboxylic acid amides and their synthesis were disclosed (Manheimmer, *U.S. 3,001,996*). The synthesis and properties of perfluoro-carboxylic and sulfonic acids are described (Guenthner and Vietor, *Chem. Res. Develop. 1,* 165).

ANIONIC PHOSPHOROUS COMPOUNDS. The synthesis and properties of complex phosphate ester surfactants were described (Mayhew and Krupin, *Soap Chem. Specialties 38,* No. 4, 55). Such esters, comprised of, for example, nonylphenolethylene oxide condensate reacted with a mole of phophorus pentoxide (Nunn and Hesse, *U.S. 3,004,056*) had improved color by esterifying in the presence of hypophosphites (Nunn, *U.S. 3,004,057*). Phosphate esters of branched chain alcohols (reacted with ethylene oxide) were claimed (Chiddix and Enyeart, *U.S. 3,033,889*). Monoethanolamine and urea were reacted to evolve ammonia, then coconut oil acids were added, subsequently sulfonated or phosphonated (Praetorius and Hiller, *Ger. 1,122,938*).

Phosphonates were prepared by reacting, for example, p-tert-octylphenyl-2 ethylene oxide condensate with triethylphosphite and neutralizing with an alkali (MacMullen and Rockett, *U.S. 3,032,578*). Esters of phosphinic and thiophosphinic acids were synthesized (Moshkina et al., *Tr. Vses. Nauchn.-Issled. Kinofotoinst. 1960, No. 37,* 27). Amidophosphonates such as bis(ethylhexyl)beta-hydroxy-ethylamidophosphate were claimed (Bolle, *Fr. 1,186,826*).

Metallic Soaps. Aluminum soaps of definite Al:fatty acid ratios were produced by metathesis (Tanabe, *U.S. 3,056,819*). Mono-, and dichloride and tri-soaps of aluminum were synthesized and their properties determined (Mahrotra et al., *J. Indian Chem. Soc. 39*, 1). Various cobalt soaps were prepared by metathesis and fusion (Aslam and Mehdi, *Pakistan J. Sci. Ind. Research 4*, No. 2, 57). Catalytic production of multivalent soaps was claimed (Soc. Anon. Ghent, *Brit. 891,858*). Oil-soluble multivalent soaps were prepared and claimed (Soc. Anon. Ghent, *Fr. 1,271,037*). Lead naphthenate was produced in solvent naphtha by metathesis (Shimizer, *Japan, 14,378 ('61)*. Metallic soaps were produced by treating metallic oxides of Groups IVA and VA with mixed anhydrides of fatty acids, with one radical shorter than the other (Considine, *Belg. 612,745*). Zirconium naphthenate soaps are synthesized by metathesis in naphtha using water-dissolved alkalis (Tittle, *U.S. 3,036,101*).

Quaternary Products. Alkylbenzyl chloride may be condensed with a tertiary amine or pyridine (Stefanovich, *U.S.S.R. 139,392*). Crystallizable 2-[2-carboxy (or carbalkoxy) alkyl]trialkylammonium bromides are claimed (Janata and Nemcova, *Ger. 1,124,508*). N-cetyl-alpha or -beta-picolinium iodide synthesis was described (Angelescu et al., *Rev. Chim. Acad. Rep. Populaire Roumaine 6*, 309). Cationic polysoaps may be synthesized by quaternizing the N of poly(alpha-vinyl beta-ethylpyridine) with n-dodecyl bromide (Narasaki et al., *Kogyo Kagaku Zasshi 64*, 1787). Epoxidized fatty acids or their esters contained alkanolamines to which di- or poly-amines may be added. The quarternary compounds of such resins form active surfactants (Forster, *Ger. 1,133,551*). Beta-(hydroxyalkyl)hydrazinium salts were synthesized (Rudner, *Brit. 884,669*).

Nonionics. The preparation and properties of some poly(oxyethylene) glycol monodecyl, monododecyl, and monohexadecyl ethers were described (Mulley, *3rd Congr.* 1*, 31). Various polyoxyethylene glycols and their monoethers were claimed (Lowe and Butler, *Ger. 1,117,097*). Glyceryl higher diethers were synthesized (Kuwaramura, *Kogyo Kagaku Zasshi 64*, 1965). An ether was obtained by reacting terpineol with polyethylene glycol (Noda et al., *Japan 12,351 ('60)*.

Poly(propylene oxide-ethylene oxide)-type products were synthesized and evaluated (Sato et al., *Kogyo Kagaku Zasshi 62*, 1277). Propylene oxide-ethylene oxide mixtures condensed with ethylene oxide were claimed (Lundsted, *U.S. 3,022,335*). Ethylene oxide is condensed with an organic active hydrogen compound to give a polymer, followed by condensation with propylene oxide (Jackson and Lundsted, *U.S. 3,036,118*). Mixtures of conjugated poly(oxyethylene)-poly(oxypropylene) compounds were synthesized (Jackson and Lundsted, *U.S. 3,036,130*). A polyglycol was reacted with butylene oxide to give a surfactant rinse (Crecelius, *U.S. 3,031,510*). One or more alkylene oxides are reacted with various active hydrogen materials (Bihan and Arnould, *Brit. 891,901*).

Complex acetals and formals were synthesized (Rohm & Haas, *Brit. 899,824, -825*). Surfactant products were obtained with polyglycol ethers by condensation of alkylphenolformaldehyde and alkylcresol-formaldehyde resin with ethylene oxide (Voicu et al., *Rev. Chim.* (Bucharest), *13*, No. 3, 146).

Sucrose esters of fatty acids were synthesized (Crampton et al., *Brit. 890,206*). Sucrose was reacted with butylene or propylene oxide then with fatty acid chlorides (Crecelius, *U.S. 3,018,281,-282*). Partial esters of sugar polyols were synthesized (Knafo et al., *U.S. 3,021,324*). Ester salts of di- and trisaccharides were claimed (Harge et al., *U.S. 3,024,229*). Mono- or polynaphthenates of sucrose and/or raffinose are claimed (Prey et al., *Austrian 220,270*). A mixture of sucrose, methyl palmitate, formylmorpholine and potassium carbonate was reacted then ethylene oxide added (Komori and Okhara, *Japan. 23,020 ('61)*. Sucrose was dissolved in formylmorpholine and ethylene oxide added. To this mixture was added methyl stearate to form a surfactant (Komori and Okahara, *Japan. 825 ('62)*. Ethylene oxide derivatives of xylitol monoesters of C_{10-20} fatty acids were prepared and described (Yakovenko et al., *Gidrolizn. i Leso-Khim. Prom. 15*, No. 2, 17). One mole of C_{12-18} fatty acids was esterified with 1.15 moles xylitol and the product then reacted with ethylene oxide (Nobile and Poma, *Ital. 625,327*).

Monofatty acid esters of mono- and di-glucose ureide were claimed (W. R. Grace & Co. *Brit. 876,122*). Fatty acid condensation with monoethanolamides was described (Ranny et al., *Prumysl Potravin 13*, 255), as were similar products from synthetic fatty acids (Verlamov et al., *Masloboino-Zhirovaya Prom. 27*, No. 4, 30). Two moles of mono- or diethanolamine were reacted with one mole methyl laurate (Komatsu, *Japan. 13,622 ('61)*. High molecular weight amides by reacting diethanolamine with fatty triglycerides were claimed (Lohr, *U.S. 3,040,075*). Diethanolamine was reacted with alpha, alpha'-dimethyl carboxylic acid (Poltz, *Ger. 1,116,234*). Claimed were N-(5-hydroxymethyl-2-tetrahydrofurfuryl)amides and derivatives (Reynolds et al., *U.S. 3,014,926*).

Polyhydroxyureas were prepared by condensing mono- or diisocyanates with polyhydroxy amines (Ulsperger, *Ger. 1,125,905*). Long chain alkyl ureas were synthesized and their water solubility increased by adding ethylene oxide (Komori et al., *Kogyo Kagaku Zasshi 64*, 279). Polyhydroxy ureas were synthesized (Ulsperger, *J. Prakt. Chem. 12*, 279).

9,10-Octadecanediols and 9,10-dihydroxy stearonitriles were oxyethylated and the properties described (Wrigley et al., *JAOCS 39*, 80). Surface active hexa- and triacylmelamines were produced (Dambacher and Hoerig, *Ger. 1,124,956*). Improvements in the fatty acid amides of *U.S. 1,989,968* were described in reacting them with urea (Dambacher and Hoerig, *Ger. 1,123,106*). Low molecular weight alkyl polymers of acrylamide were produced (Yamasheta and Ito, *Kogyo Kagaku Zasshi 63*, 1746). The competitive reaction of ethylene oxide in a mixture containing amine, imine and hydroxyl groups was studied. The hydroxyl group catalyzed the reaction, preferential reaction occurred with the amino acid, and other data were shown (Oshiro, *Kogyo Kagaku Zasshi 64*, 2132). Nonionic polyhydroxy carboxamides containing D-gluconic acid were described (Ulsperger, *Ger. 1,124,938*).

Compositions

Antiseptic. Whether bacteriostats should be added to toilet soaps was answered (Linfield and Casely, *J. Soc. Cosmetic Chemists 13*, 81), and antimicrobial action was claimed for a liquid potash soap containing a mixture of hexachlorophene and lecithin (Pamzarella, *Soap Chem. Specialties 37*, No. 12, 73). Dispersed silver alginate was used to render liquid and other detergent products antiseptic (Horowitz and Mendelsohn, *U.S. 3,050,467*). Silver salts of aromatic mono- or polycarboxylic acid in 100–500 ppm amounts was added to soap (Permachem Corp., *Brit. 847,258*). Neat's-foot soap was rendered antiseptic by adding a complex salt of silver and a cyclic thiourea (Sajima et al., *Japan. 430 '61*). A complex of silver with ethylenethiourea was used as a germicide (Permachem Corp., *Brit. 847,257*). Silver salts of aromatic cyanamides and imides were claimed for use with soaps (Permachem Corp., *Brit. 847,256*).

Methylene bis(3,4,5-trichlorophenol) was used in a synthetic detergent composition (Yufera, *Span. 263,687*). A combination of 3,4,5-tribromosalicylanilide and 3,4,4-trichlorocarbanilide was added to soap (Bright, *Ger. 1,126,058*). Liquid soaps were prepared using bis(3,5,6-trichloro-2-hydroxyphenol)urethane (Atterby, *Brit. 887,247*). Though 2,2'-thiobis(4,6-dichlorophenol) is effective, and substantive to cotton, it loses substantivity in the presence of detergents, but its metal salts are substantive and fast (Harvey, *U.S. 3,024,163*). Used in a syndet mixture with soap was 3,4,5-tribromosalidylanilide (Kelly, *U.S. 043,778*).

N-alkanoyltaurate is used to complex iodine to form a series of germicidal compositions (Neracher, *U.S. 3,039,916*). Cationic carriers were claimed as superior to nonionic products in complexing iodine (Winicov and Schmidt, *U.S. 3,028,299*). A liquid antiseptic is produced using a complex ampholyte (Schmitz and Cramer, *U.S. 3,039,917*). Long chain quaternary salts increase solution viscosity and provide germicidal power (Diehn et al., *Belg. 616,019*). A biocidal ingredient for soap is comprised of a basic anion exchange resin such as urea-guanidine-formaldehyde condensation product (Schmitz et al., *U.S. 3,016,327*). Bacteriostatic and fungistatic properties are imparted using 2-mercaptobenzothiazoles and their salts (Weight and Seiler, *Brit. 873,602*). The sodium salt of sulfonated alkyl chlorodiphenyl oxide proved occasionally useful for disinfection (Miedler et al., *J. Invest. Dermatol. 35, 269*). Cleaning and disinfection of garments was reviewed (Kiger, *Prod. Pharm. 17*, 341), four hospital detergent-sanitizers were evaluated (Vinson, *Soap Chem. Specialties 37*, No. 11, 61), and various disinfectants for home laundering were described (McNeil and Choper, *Soap Chem. Specialties 38*. No. 8, 51).

Bars and Tablets. The details of preparing a high quality toilet soap were reviewed (Charlton, *Mfg. Chemist 33*, No. 5, 176). Comparison was made of two tests for evaluating syndet bars (Manneck, *3rd Congr.* 3*, 184). An extensive investigation of efflorescence in concentrated translucent soap was made by examination with gas chromatography. Several explanations based on the findings were given (Prevot and Cabeza, *3rd Congr.* 3*, 199). Epidermal tests of the effect of soap were made and it was postulated that increase in permeability with time resulted from damage to the barrier zone caused by soap (Bettley, *Brit. J. Dermatol. 73*, 448). Excised normal human skin prevented the penetration of six sodium alkyl sulfates and four sodium alkylbenzenesulfonates through combination with the skin protein (Blank and Gould, *J. Invest. Dermatol. 37,* 311). Hypersensitivity to soap and synthetic detergents was most marked in those subjects sensitive to certain allergens (Nilzen et al., *Dermatologica 124,* 42).

Cakes or bars are prepared from the saponification of soap-forming acids and alkali silicates (Menon, *Indian 70,851*). Soaps containing inorganic salts are formed more effectively by adding a water-soluble glycol ether (Glynn et al., *U.S. 3,032,505*). Soaps are improved by adding an alkanolamine trisalt of ethylenediamine tetraacetate (Campbell and Howard, *U.S. 3,026,265*). Mixtures for bars are formed by combining sodium alkylisethionates and alkylarenesulfonates (Wittwer, *U.S. 3,055,837*). Mixtures of sodium stearol isethionate and alkylarylsulfonate are used for cake making (Wittwer, *U.S. 3,057,805*). Alkali metal C_{8-18}alkyl sulfates are used for toilet bars (Huyser et al., *Ger. 1,111,761*). Sulfonated long straight chain olefins from cracking petroleum wax form bars (California Research Corp., *Brit. 890,696*). Combinations of sodium n-alkanesulfonates and sodium alkylbenzenesulfonates are used to prepare bars (Bohrer, *Ger. 1,118,384*). Either a mixture of alkyl sodium sulfonates or an alkylbenzenesulfonate may be used in cake manufacture (California Research Corp., *Belg. 611,781*). A nonionic-fatty alcohol sulfate mixture with Aerosil is used in formed products (Manneck, *Ger. 1,083,469*). A mixture of soap and alkali metal fatty acylaminomethane sulfonate is used to prepare tablets (Parke and Penketh, *U.S. 3,043,779*).

Skin irritation caused by detergents is said to be reduced by adding N-acylsarcosine and its water-soluble salts (Dvorkovitz et al., *U.S. 2,962,448*).

A detergent bar is claimed comprising a fatty acid monoglyceride sulfate, sodium dodecylbenzenesulfonate and soap (Colgate-Palmolive Co. *Brit. 885,332*). A bar containing sodium monoglyceride sulfate and zinc soap is claimed as superior (Turck, *U.S. 3,030,310*). A nonbrittle product is claimed made from soap and pentaerythritol monostearate, or other mixtures (Dehydag Deutsche Hydrierwerke G.m.b.H., *Brit. 886,583*). A solid cleaning composition is described made of a water soluble primary alkane sulfonate and soap (Levine at al., *Ger. 1,108,367*).

A scum-dispersing product is comprised of a fatty acid methyltaurate, soap, fatty acid, and a bacteriostatic agent (Kelly, *U.S. 3,043,778*). Soap for hard water usage is prepared by combining soap and sodium dodecylbenzenesulfonate (Sheely and Glynn, *U.S. 2,991,253*).

Bleach Compositions. The proper use of hypochlorite in commercial laundry work was described and the manner in which fabric degradation occurs is noted (Nieuwenhuis, *Lavoirs-Laveries-Blanchisseries No. 176*). Ampholytes were used in preparing perfumed aqueous hypochlorite solutions (Gibbons et al., *Brit. 886,084*). A process for potassium dichloroisocyanurate is described (Symes et al., *U.S. 3,035,054*), as are both the sodium and potassium dichloroisocyanurates (Symes, *U.S. 3,035,056*). Stabilization of compositions containing trichlorocyanurate salts is achieved (Symes, *U.S. 3,002,931*).

Concentrated solutions of acidic hydrogen peroxides, and organic and inorganic perhydrates are produced by adding certain alkylene oxide adducts (Henkel & Cie., *Ger. 1,121,594*). Sodium perborate is added to an alkaline liquid soap (Chala, *Span. 258,861*). Pelleted alkali metal pyrophosphate perhydrate is used in detergents and bleaching agents (Lindner, *Ger. 1,118,167*). Perhydrates for use in detergent compositions are made from acylation products of phosphorous acid (Schiefer et al., *Ger. 1,133,726*). Spherical or granular forms of perborax as prepared may be used in detergent compositions (Bretschneider, *Ger. 1,112,502*).

Stabilized liquid detergents containing bleaching organic peroxides were claimed (Schiefer et al., *Ger. 1,131,347*). Liquid detergents were made incorporating glycol derivatives containing peroxide groups (Henkel & Cie. *Brit. 889,652*). Alkali metal monopersulfates can be added to detergents for fibers or porcelain to bleach them to whiteness (du Pont & Co., *Brit. 889,079*). Solid detergents with bleaching action were prepared containing a surfactant and peroxybenzoic acid (Brocklehurst et al., *Ger. 1,105,092*). Detergent bleaching compositions were prepared containing inclusion compounds such as urea treated with percarboxylic acids (Van Embden et al., *Brit. 847,702*).

Corrosion and Inhibition. Reviewed were the properties of modern detergents in their effect on the enamelled surfaces of washing machines (Lloyd, *Prod. Finishing* (*London*) *15,* No. 1, 80). Using copper as the metal, the effect of detergent solution was monitored and the laboratory results were confirmed by similar tests in commercial washing machines (Mosle and Wolf, *Werkstoffe Korrosion 13,* 544). Estimated was the corrosiveness of various detergents on a number of metals encountered in dairy industries (Whittlestone, *Australian J. Dairy Technol. 17,* 101).

Effective for mild steel protection were quaternary mixed polyalkyl pyridines (Jones, *U.S. 3,033,784*). Corrosion inhibiting surfactants are prepared by ethoxylating a phenolic amine (Bruson et al., *U.S. 2,998,452*). Oil and gas ferrous equipment was protected from corrosion using cyclic amidine esters (Stromberg, *U.S. 3,017,356*). Prevention of copper alloy tarnishing was claimed using a complex imidazole (Harris, *U.S. 2,992,996*) and a complex imidazoline (Harris, *U.S. 3,037,937*). Sodium citrate was claimed as a corrosion inhibitor when added to a detergent containing an alkylarenesulfonate, a polyphosphate, and a per salt (Koninklijke Dobbelman N.S. *Neth. 98,553*). Salts of Group IVB have anticorrosive activity in detergent solutions, examples being titanium, zirconium and thallium compounds (Wolhoff, *Brit. 849,747*).

Builders and Adjuvants. In discussing the role of builders in detergency, such action was said to occur only when flocculating cations were present in the wash system. Other characteristics of builders were discussed (Jones et al., *3rd Congr.* 4,* 188). The latest developments with builders were reviewed (Raphael, *Mfg. Chemist 33,* No. 8, 323). The critical concentration at which building first occurs was shown as directly proportional to water hardness (Jones and Parke, *3rd Congr.* 4,* 178). Sequestration of calcium ion by polyphosphates in the presence of precipitating oxalate agent increased with temperature and decreased with increase in polyphosphate chain length. Tripolyphosphate and pyrophosphate were most effective Ca and Mg ion sequestrants on a weight basis (Irani and Callis, *JAOCS 39, 156*). Essentially the findings of Irani and Callis were verified (Oldenroth, *Fette, Seifen, Anstrichmittel 64,* 486). Sodium tripolyphosphate was manufactured in such a way as to minimize grit formation (Cassidy et al., *U.S. 3,054,656*). Form II tripolyphosphate in detergent slurries is preferred over Form I for less degradation and lesser acidity, hence less silica formation during slurry drying (Ryer, *U.S. 3,956,652*). Anhydrous tripolyphosphate Form I exhibited lower viscosity in slurries, and the dried particles are free from sandiness and stickiness (Martin, *U.S. 2,961,410*). Alkali metal salts of imidodiphosphates are claimed as effective detergent builders (Nielsen, *U.S. 3,018,165*). The properties of organic amino type chelating agents and inorganic phosphates were reviewed (Singer, *Chem. Specialties Mfrs. Assoc. Proc. 47th Mid-Year Meeting, 1961,* 152). Both the trisodium salt of ethylenediamine tetraacetate and sodium hexametaphosphate were used in a milking machine detergent (Whittlestone, *Australian J. Dairy Technol. 17,* 43). Partial hydrolysis of gluconates used in caustic compositions resulted in improved sequestration (Bonewitz, *U.S. 3,046,232*). Hard water soap sludge was reduced by adding a sulfoxide to the composition (Wilson and Rudy, *Ger. 1, 130,104*). Redeposition of soil was minimized by adding water soluble salts of copolymers of acrylic acid and/or methacrylic acid with vinylsulfonic acid (Herrle et al., *Ger. 1,134,786*). Sodium carboxymethyl cellulose and other water soluble polymers when added to detergent compositions, improved detergency (Matsukawa, *Kogyo Kagaku Zasshi 64,* 1643). Sodium carboxymethyl cellulose was beneficial in commercial scale tests in removing soil during the soak (Nieuwenhuis, Lavoirs-Laveries-Blanchisseries No. 176). Various polymers as assistants in soil removal were evaluated, proposed for use, and mechanism discussed (Matsukawa, *Kogyo Kagaku Zasshi 62,* 1892). Poly(vinylpyrollidinone) was used as a soil suspending agent (Fong et al., *U.S. 3,000,830*). Carboxymethyl cellulose in liquid detergents was discussed and a new type described (Batdorf, *Soap Chem. Specialties 38,* No. 1, 58). Corn-steep water is said to be superior to carboxymethyl cellulose as a soil suspending agent (Katzbeck and Hach, *Brit. 889,041*). Fluorescent dyes in detergent compositions were discussed (Goldwasser, *Soap Chem. Specialties 38,* No. 4, 47).

Dishwashing. Discussed in a review are the factors involved in both hand and automatic washing of dishes and a discussion of recent developments (Price, *Soap Chem. Specialties 37,* No. 11, 51). Nonfoaming compositions for automatic washing comprise mixtures of nonylphenol ethenoxy ethers and a naphthalene sulfonic acid-formaldehyde adduct (Frank et al., *Ger. 1,122,199*). Another composition comprises a nonylphenol ethylene oxide adduct with builders (Grifo and Mayhew, *U.S. 3,022,250*). A liquid composition consists of mixed ethylene oxide-propylene oxide high molecular weight polymer with, in some cases, addition products of alkylated aromatic hydroxy compounds (Goette, *Ger. 1,106,907*). A dishwashing detergent may be defoamed by adding a polyoxyalkylene glycol mixture of specified properties (Martin and Temple, *U.S. 3,048,548*). A machine rinse to reduce spotting is comprised of an alkylarenesulfonate, an alkyl polyglycol ether, and tartaric acid in water (Pils, *Ger. 1,109,-819*). Described was a substantially alkaline mixture containing 0.05 to 5% of an alkylphenyl ethylene surfactant and a defoamer (Carroll, *U.S. 3,010,907*). A liquid composition comprised of the potassium salts of poly- and pyrophosphates, orthophosphate, silicate, KOH and water (Lintner *Ger. 1,110,798*). For hand dishwashing with a stable high sudsing composition used was N-dodecylurea N'-glucoside, in combination with sodium alkylbenzenesulfonate and sodium tripolyphosphate (Thos. Hedley & Co., *Brit. 811,902*). For metal, glass or plastic surfaces the following have been claimed: A tertiary hydrocarbylamine poly(ethenoxy) sulfate (Dupre and Boettner, *Ger. 1,103,502*); the same compound with a controlled degree of sulfonation (Boettner and Dupre, *Ger. 1,112,602*); ethylene oxide-propylene oxide with nonyl phenol and builders especially good for steel cleaning (Goette et al., *Ger. 1,114,605*). An acid cleaner was comprised of phosphoric acid, tertiary isobutanol, alkylbenzenesulfonic acid, and water (Ohtsuka, *Japan. 3482* ('61). Milk bottles are cleaned with a mixture containing globular caustic soda, dry alkylarenesulfonate and trisodium phosphate hexahydrate (Hellsten, *Swed. 176,270*). A glass cleaning mixture was comprised of sulfonated fatty alcohols, condensates of fatty acids with polypeptides or methyltaurine and organic solvents (Sikorski and Skarzynska, *Pol. 45,045*).

DRYCLEANING. A lecture concerning drycleaning was published (Fogel, *Z. Ges. Textil-Ind. 64,* 794), and the subject was reviewed (Ishiwata, *Koroido To Kaimen-Kasseizai 2,* 226). The influence of surfactants on artificially soiled wool in drycleaning was examined. Various materials were evaluated (Kajl, *3rd Congr.* 4,* 184). The vapor pressure over drycleaning detergent baths was measured (Wedell, *3rd Congr.* 4,* 220). Isopropyl alcohol added to drycleaning solvents aided in maintaining sufficient water to remove water soluble stains (Hess, *Ger. 1,112,240*). Soil redeposition in drycleaning baths was prevented by adding a mixture of an anionic surfactant and an ethylene oxide condensate (Mayhew et al., *U.S. 3,018,251*). Long chain amines are claimed to improve the water dispersion qualities of baths (Boehme Fettchemie G.m.b.H., *Brit. 847,017*). An isopropylamine salt of an alkylarenesulfonic acid acts as an emulsifier for water in the drycleaning bath (Chimiotechnic, Union Chimique du Nord et du Rhone, *Fr. 1,271,170*). A drycleaning bath is comprised of solvent, water, and any of a number of different surfactants of varied types (Wedell, *U.S. 3,057,676*). A disinfecting drycleaning solution is claimed consisting of an organic solvent, formaldehyde in alcohol, and a detergent adjuvant (Chem. Fab. Stockhausen & Cie., *Belg. 611,744*). Spot removing compositions contain organic solvents and especially prepared silicic acid (Pinkert, *Ger. 1,108,837*). Solutions of triethanolamine or triisopropanolamine sulfite or sodium alkylarylpoly(oxyethylene)sulfite are used for spot removing (Pichlmayr, *Ger. 1,111,760*).

FOAM CONTROLLED COMPOSITIONS. A lecture on low foaming products was published (Tschakert, *Soap, Perfumery & Cosmetics 34,* 856), and foaming and foam removal were reviewed (Manegold, *3rd. Congr.* 2, 522*). Combinations of high molecular weight nonionic surfactants with high molecular weight anionics act synergistically to depress sudsing (Schwoeppe, *U.S. 2,954,348*). Low foam surfactants are claimed such as alkylaryl oxyethylene-oxypropylene glycols (Dupre and Wolfrom, *Ger. 1,112,279*). Low foamers are comprised of propylene oxide treated with a condensation product of ethylene oxide with an aliphatic compound with a reactive hydrogen (Kirstahler and Goette, *Ger. 1,126,140*). Mixtures of metal carboxylates and ethylene oxide condensates are claimed (Wedell, *Ger. 1,089,905*). An alkylphenol-formaldehyde condensate is combined with a low foaming benzyl ether of alkylphenoxy-poly(ethoxy)ethanol with enhanced detergency (Rohm & Haas, *Brit. 895,596*). Heavy duty detergents with controlled sudsing action are comprised of a mixture of alkylbenzenesulfonate and a nonionic condensation product of ethylene oxide with a base formed by condensing propylene oxide with propylene glycol (Clarke and Hester, *U.S. 3,009,882*). Controlled foaming of alkaline compositions is attained by adding small amounts of an alkylphenol ethylene oxide condensate and 2-ethylhexylpoly-(oxyethylene)phosphate ester (Carroll, *U.S. 3,010,907*). Excessive foaming of anionic detergents is claimed to be controlled by adding a mixture of high molecular weight fatty acids (St. John et al., *U.S. 2,954,347*). Antifoam agents of an aliphatic, olefinic, hydrocarbon-substituted succinic anhydride are claimed (Jurisch, *U.S. 3,004,923*). An antifoamer is comprised of a poly(oxyethylene) poly(oxybutylene)-ether of glycerol (Teot et al., *Belg. 611,104*).

FOAM IMPROVED COMPOSITIONS. Exceptional foam stability is claimed for a 1:1 weight mixture of tetrapropylene- and pentapropylenebenzenesulfonates (Wilson, *U.S. 3,017,363*). Compositions containing a sulfate ester of poly (oxyethylated) organic compound and/or alkylarenesulfonate have synergistic foaming quality (Jelinek and Mayhew, *U.S. 2,941,951*). The foaming properties of an alkylene oxide addition product are improved by adding a high molecular weight difficultly soluble carboxylate (Wedell, *U.S. 3,008,905*). An alkylbenzenesulfonate composition produces improved foam by addition of a water soluble n-alkyl o-carboxybenzyl sulfide salt (Toland and Schlatter, *U.S. 3,053,771*). A strongly foaming sulfosuccinic ester detergent is produced by reacting the half ester of stearylethanolamide and maleic anhydride with an aq. solution of sodium sulfite (Praetorius, *Ger. 1,115,398*). Foam stability is improved by adding small amounts of a hydrolyzed polyacrylonitrile to an alkylarenesulfonate (Brown, *U.S. 3,032,506*).

FOOD INDUSTRIES. When the trisodium salt of ethylenediamine tetraacetic acid was added to an alkaline milking machine detergent, increased corrosion resulted (Whittlestone, *Australian J. Dairy Technol. 17,* 43). Comparison was made between a hypochlorite detergent system and an iodophor system with decrease in milkstone formation for the iodophor system and no marked change in corrosivity (Scott et al., *Agr. Gaz. N. S. Wales 73,* 227). A brewery detergent comprising an inhibited mineral acid had bactericidal properties (Loncin, *Brauwelt 101,* 1061). Laurylpyridinium bromide was used successfully to disinfect margarine factory plants and rooms (Kajl et al., *Prace Inst. Lab. Badawczych Progemslu Spozywczego 11,* No. 2, 55).

LIQUIDS. Various hydrotropes and their effect upon polyphosphate-containing compositions were discussed (Mausner and Sosis, *Soap Chem. Specialties 38,* No. 2, 47). A liquid containing alkylphenoxypolyethoxyehtanol and alkylbenzenesulfonate for kitchen utensil cleaning was patented (Rowland, *Ger. 1,098,660*). A light-duty liquid contained a salt-free phenylpolypropylene sulfonate (Henderson and Carroll, *U.S. 3,029,205*). Another liquid contained nongelling triethanolamine alkylbenzenesulfonate by adding a low molecular weight amine sulfate (Terry, *U.S. 3,005,777*). Clear solutions are obtained using mixtures of two alkylarenesulfonate and one nonionic surfactant (Shell Internationale, *Belg. 610,211*).

Liquids are prepared from an amine oxide and a salt such as potassium ethylenediamine tetraacetate with a hydrotrope (Drew et al., *U.S. 3,001,945*). Fatty acid bis(hydroxyalkyl)amides are mixed with dodecylbenzenesulfonate (Colgate-Palmolive Co., *Brit. 883,733*). A formulation comprises a mixture of an ampholyte and a water insoluble fatty acid alkylolamide (Th. Goldschmidt A.-G., *Brit. 848,654*).

Light duty liquids were covered comprising polypropylenebenzene sulfonate and a variety of nonionic surfactants (Henderson and Carroll, *U.S. 3,029,205*). Clear detergent solutions containing lanolin oil are described, employing a lanolin alcohol poly(ethylene oxide)ether and a fatty acid alkanolamide (Russell and Hoch, *Drug Cosmetic Ind. 90,* 294). Lanolin was prepared in a transparent emulsion by using (polyoxyethylene)sorbitan monostearate (Hirsh, *U.S. 3,052,608*). Dextro-sorbitol was used to maintain the fluidity of solutions of polyoxyethylene, polyoxypropylene, or their combined polymers (Lafon, *Fr. 1,292,930*).

Compatibility studies of liquids containing silicates, polyphosphates, and alkylarenesulfonate were made, giving tables and graphs showing solution stability (Getty and Stericker, *Soap Chem. Specialties 38,* No. 1, 54; No. 5, 76). Use of phosphates in liquids was reviewed and high concentration composition described (Liss and Tuvell, *Soap Chem. Specialties, 38,* No. 3, 47). Methods for incorporating potassium polyphosphates in liquid detergents were discussed (Pfrengle and Pietruck, *Fette, Seifen, Anstrichmittel 64,* 321). Heavy duty liquids generally contain silicates and phosphates as builders as well as hydrotropes and other usual ingredients. A clear high foam liquid contained in addition to such builders propylene glycol, sodium dodecylbenzenesulfonate and a hydrotrope (Tidridge and Cohen, *U.S. 3,037,935*). An opaque, opalescent liquid contained a tertiary amine oxide, an anionic surfactant and an acrylamide or high molecular weight ethylene oxide product (Pilcher and Eaton, *U.S. 2,999,068*). Viscous opaque liquids contained an alkali metal borate, tertiary aliphatic amine oxide and alkylarenesulfonate (Procter & Gamble Co., *Belg. 610,313*). Creamy low-foam liquids contain propylene glycol and alkylaryl polyoxyethylene alcohol, ester, or ether (Tidridge and Cohen, *U.S. 3,037,936*).

Another heavy duty liquid contains as active ingredient a mixture of alkylarenesulfonate, alkylaryl sulfate, or alkyl phenoxy(polyethoxy) sulfate (Colgate-Palmolive Co., *Belg. 613,165*). A stable aqueous suspension is claimed containing sodium and potassium tripolyphosphate, triethanolamine, C_9-C_{15} alkylbenzenesulfonate, laurylethanolamide and other normal additives (Doan, *U.S. 3,023,168*). Morpholine or morpholine oleate is added to a solution of tripolyphosphate, alkylbenzenesulfonate and laurolyethanolamine (Doan, *U.S. 3,024,204*). A liquid heavy duty composition contains amounts of carboxylalkyl cellulose derivatives with builders and alkylarenesulfonates as active ingredients (Batdorf and Ellis, *Ger. 1,122,658*). Another liquid composition comprises as active ingredients, water soluble organic sulfate, a fatty alkylolamide, and a hydrotrope (Wixon, *U.S. 3,052,635*). A general purpose heavy-duty liquid was claimed containing sulfonated mixtures of *alpha-* and *beta-methyl* and/or dimethyl naphthalene sulfonate and an alkylarenesulfonate (Greene, *U.S. 3,042,623*). A high foaming heavy-duty liquid was described containing sodium dodecylbenzenesulfonate as the active ingredient and propylene glycol among other ingredients (Tidridge and Cohen, *U.S. 3,037,935*). A low foamer was obtained by mixing lauric diethanolamide with builder and a hydrotrope (Mooney et al., *Ger. 1,120,627*). Liquid compositions with improved stability toward phase separation may be obtained by using various water dispersible polymers (Monsanto Chemical Co., *Brit. 872,530*). Stabilized alkali-soluble acrylic emulsions for liquid compositions and their effect on phase stability were described (Fordyce et al., *Soap Chem. Specialties 38*, No. 3, 57). A nonionic surfactant with vitreous polyphosphates was claimed for bath use (Colas et al., *Fr. 1,267,947*). A liquid detergent was described containing—in addition to a specially treated polyphosphate—monoethanolamine, triethanolammonium alkylbenzenesulfonate, and a nonionic surfactant, (Schneider et al., *Ger. 1,066,692*). An aqueous ammonium hyroxide-containing liquid was described which included both anionic and nonionic surfactants (Stahler and Stahler, *U.S. 3,001,947*). A liquid abrasive composition was disclosed which contained an anionic surfactant, fatty acid, and alkanolamide (Jones and Stephens, *Brit. 882,569*). A liquid detergent containing active chlorine and small amounts of alkaline silicate and tripolyphosphate with the ammonium salt of a surfactant was claimed for high foam and synergistic action (Lorenzo, *Span. 255,974*). Improved temperature stability of an alkyl sulfate liquid detergent containing mineral oil was achieved by adding amounts of ammonium sulfate and dimethylcyclohexanol (Steward, *Ger. 1,098,661*).

METAL CLEANING COMPOSITIONS. Engine carbon and sludge are removed by a solvent composition containing penetrants and lead deposit removers (Sweetman, *U.S. 3,005,778*). A metal cleaning and corrosion inhibiting composition contained substituted alkyl- or arylthiourea, a nonionic surfactant of low emulsifying power in a solvent (Union Chimique Belge Soc. Anon., *Belg. 604,747*). Sulfonated surfactant solutions were used for removing spontaneously combustible oil deposits from compressor lines (Vainshtein and Pashaev, *Trudy Vsesoyuz. Nauch.-Issledovatel. Inst. po Tekh. Bezopasn. Ministerstva Neft. Azerbaidzahn 1959, No. 11*, 68). A phosphoric acid ester in hydrocarbon solution was used to clean pipes and protect them from oxidation (Elliott, *U.S. 3,010,853*). The various types of cleaners used in the aero-space field were reviewed and use technics described (Weast and Coryell, *Soap Chem. Specialties 37*, No. 10, 52). Fingerprint removal processes and materials and the use of radiotracers for the purpose were discussed (Kashima and Nose, *Kogyo Kagaku Zasshi 64*, 2149). A polish for metals was a mixture of butylpyrocatechol-soap-isopropanol-kerosene *and tripoli powder* (Becher, *Belg. 615,021*). Abrasive compositions useful at various pH values containing various acids, other ingredients, and sodium dodecylbenzenesulfonate were described (Kirschenbauer, *U.S. 3,042,622*).

PASTES. Inhibitors of paste-hardening during storage were found to be unsulfonated substances such as grease solvents (Getmanskii and Vagina, *Tr. Nauchn.-Issld. Inst. Sintetich. Zhirozamenitelei i Meyushchikh Sredstv 1961*, No. 2, 98). A heavy-duty detergent composition can be prepared in paste form *containing anionic and/or nonionic surfactants*, polyphosphates and a water-soluble cellulose derivative (Ehrenhalt et al., *Israeli 14,595*). A stable high-foam paste contains builders, carboxymethylcellulose and lauryl sulfate (Cohen, *U.S. 3,039,971*). A gelled product was an alcoholic-water solution of sodium stearate, silicones and a nonionic surfactant (Societa Zampoli & Brogi, *Ital. 596,676*). Soft soaps were discussed, and such products with 30–45% ionic or nonionic surfactants were said to be available (Weiss, *3rd. Congr.* 4*, 226). Homogeneous dispersions of alkaline earth and aluminum silicates and fatty acids useful as detergents were prepared using hydrophobic organic liquids (Hustix, *Ger. 1,126,874*).

POWDERED PRODUCTS. A powdered syndet was produced by neutralizing a mixture of alkylbenzenesulfonic acid, palm oil fatty acid, and monoethanolamide with dry soda ash (Kawakami, *Japan 15,368('61)*. Powdered alkylarenesulfonates are stabilized against caking by adding a small amount of sodium methyl sulfate (Mills, *U.S. 3,030,312*). A dry, powdered product was obtained by mixing until dry, borax and potash soft soap (Dunn, *U.S. 3,020,237*). Various dry builder salts are intimately mixed wtih a monocarboxylic acid or monoester to give powdered products (Curtin, *Ger. 1,133,061*). Dry builders are mixed with anionic or nonionic surfactants, a hypochlorite salt, and p-substituted benzenesulfonamide (Hurt and Mitchell, *U.S. 3,054,753*).

Dust-free compositions were maintained by adding a salt whose vapor pressure was at least 11 mm at 20C. An example was sodium sulfate decahydrate added to quartz powder, alkyl sulfate, and alkylbenzenesulfonate (Fries and Sinner, *Ger. 1,119,435*). A uniform dust-free composition containing polyphosphates, a per salt or chlorine bearing material, and an anionic surfactant was obtained in a three-step process (Pfrengle and Pietruck, *Ger. 1,125,580*). Dust-free detergents were obtained by spraying a nonionic surfactant on an alkali metal phosphate, then finally dusting on a voluminous adsorptive material prepared by spray-drying a specific composition comprising a detergent mix (Pfrengle et al., *Ger. 1,107,870*). A nodular, non-dusting composition was described where caustic soda and tetrasodium pyrophosphate were steam-hydrated in a mixer and an alkyldimethylbenzylammonium chloride and hydrated silica added with further mixing (Allen, *U.S. 3,007,877*). A process was described for spray drying an alkylarenesulfonate, sodium sulfate, and a higher-alkylbenzenesulfonate (Monick, *U.S. 3,055,835*).

SHAMPOO. Quaternary ammonium salts of a urea reaction product was combined with an alkylene oxide condensate with aliphatic mercaptans (Cibe Ltd., *Brit. 878,519*). A quaternary compound such as [2-hydroxy-3-(dodecyloxy)propyl] triethylammonium chloride is used in shampoos (Laboratoires Scientifiques de Neuilly, *Fr. 1,265,818*). Cationic compounds are made by treating the condensation product of a fatty acid with a polyamine containing hydroxyl groups with a carbamic acid ester (Ciba Ltd., *Ger. 1,092,460*). Liquid anhydrous compounds are prepared by neutralizing acid sulfates such as alkyl(polyethoxy)sulfates with an amine (N.V. Chemische Fabriek ''Andrelon'', *Brit. 891,631*). A product initially neutral contains substances *permitting* subsequent control of acidity. This comprises a fatty oil, soap, alkylarenesulfonate, and the acidity resulting from inclusion of organic acids (Kirschenbauer, *U.S. 3,042,621*).

Cream shampoos were obtained using sodium secondary alkyl sulfates as the active ingredient (Getmanskii, *Tr. Nauchn.-Issled. Inst. Sintetich. Zhirozamenitelai i Moyushchilch Sredstv 1961, No. 2*, 92). Shampoos containing organic sulfur were prepared from ethylene oxide-mercaptan condensates (Tissot, *Fr. 1,256,633*). A combined shampoo and hair tinting shampoo contained a nitro amine dye and as sufactant a mixture of dialkyl sulfosuccinate and a fatty acid alkanolamide (Frohnsdorff, *Brit. 889,327*). An analytical study of titratable acidity, alkalinity, and other factors were studied for their possible effect on the adsorption by hair of arsenic, present in shampoo (Griffon et al., *Ann. Pharm. Franc. 19*, 407).

STABILIZATION. Soap autoxidation and prevention were reviewed (Paquot, *Parfum. Cosmet. Savons 4*, 502). Cause for decomposition of dyes and corrosion of metals by polyoxyethylene nonionic surfactants was found to be due to autoxidation of the ether, which did not occur with amides or amine types (Nemoto et al., *Kogyo Kagaku Zasshi 62*, 483). Perfumes in toilet soaps were stabilized by adding fatty acid alkanolamides (Rosenthal, *Ger. 1,124,962*). Color spotting, darkening, and rancidity of soaps and/or syndets may be prevented by addition of 0.05–0.5% of a mixture, for example, of sorbitol and sodium diaminetetraacetate (Martins et al., *Brit. 875,720*). Storage stability of a washing composition whose active ingredient was the sulfate of sperm whale-oil fatty alcohols was adversely affected by the presence of alkyl disulfates and methods for their removal were suggested (Varlamov, *Masloboino-Zhirovaya Prom. 27*, No. 9, 15). Detergents containing alkanolamine salts of tetrapropylenebenzenesulfonate were stabilized against discoloration by adding 0.5–1.0% of 1,2-propylene glycol sulfite (Rosenthal, *Ger. 1,124,962*).

SURFACTANT MIXTURES. Synergistic action by using combinations of surfactants for a variety of detergent compositions is becoming a recognized means for surfactant utilization. A composition of pentadecylbenzenesulfonate was combined with the dodecyl compound (Wilson, *U.S. 3,017,363*).

Mixtures of secondary alkyl sulfates and alkylarenesulfonates were produced from petroleum fractions (Ministry of Romania Petroleum and Chemical Industry, *Brit. 884,656*). Improvement resulted by mixing secondary alkyl sulfates with alkylbenzene sulfonates for liquid compositions (Bataafse Petroleum Maatschappij N.V. *Brit. 818,369*). Mixtures of alkyl sulfates and alkylarenesulfonates were claimed (Stewart, *Ger. 1,097,607*). Propylene polymer fractions were condensed with benzene to give alkylbenzene compositions (McLean, *Brit. 884,755*). Alkylarenesulfonates were improved by adding mahogany sulfonates (Anderson and McDonald, *Ger. 1,091,108*). A low molecular weight kerosene sulfonate is improved by adding a high molecular weight sulfonate (Myshkin, *Khim. Prom. 1961,* 535). A surfactant of the sulfate or sulfonate type is improved by adding a sulfobetaine (Guendel et al., *Ger. 1,135,606*). Claimed was a composition comprising a mixture of an anionic or nonionic surfactant with a water insoluble boric acid ester of a fatty alkylolamide (Dohr et al., *U.S. 3,024,197*). A multicomponent composition was described comprising 1–3 parts of an anionic or nonionic syndet, 1–3 parts of the condensation product of a higher fatty alcohol or alkyl phenol with up to 3 ethylene oxide units, 1–3 parts of a polyalkoxy dihydric alcohol and a polyphosphate. (Michaels, *U.S. 3,034,989*). An antistatic composition for textiles contains nonionic, anionic, and cationic surfactants (Brunt and Williams, *Brit. 873,214*).

TEXTILE COMPOSITIONS. Reviewed were alkylbenzenesulfonates, alkyl sulfates and mercerizing wetting agents for textile usage (Ermolaeva and Lobanova, *Nauchn.-Issled. Tr. Tsentr. Nauchn.-Issled. Inst. Khlopchatobumazhn. Prom.* Moscow 1958, 144). A recipe for a liquid detergent for washing wool, silk, and synthetic textiles was based on secondary alkyl sulfates (Getmanskii and Panchenke, *Tr. Nauchn.-Issled. Inst. Sintetich. Zhirozamenitelei i Myuschchikh Sredstv* 1961, No. 2, 95). A composition containing a sulfated imidazoline softener was used with anionic or nonionic surfactants (Sherrill and Linfield, *Belg. 613,062*). A fabric softener was comprised of the reaction product of, for example, diethylene triamine distearamide and mono-2-hydroxychloropropyl ether of glycerol (Zech. *Ger. 1,103,923*). A detergent for colored linen becomes more effective by suppressing optical brighteners by adding a mixture of poly(vinyl pyrrolidinone) and a cationic surfactant (Oster, *Ger. 1,114,606*). A washing agent for nylon with antistatic and brightening properties for polyamides and cellulose materials is produced by dissolving a cationic surfactant in water, adding twice its weight of sodium sulfate, optical brightener, drying, and adding 2–1000 times the weight of an anionic surfactant (Brunt and Williams, *Ger. 1,120,625*). A rug and upholstery cleaner is comprised of an alkyl sulfate and N-fatty acid sarcosinate (Bissell Carpet Sweeper Co. *Brit. 882,635*).

MISCELLANEOUS COMPOSITIONS. Colored soaps were reviewed. (Dambe, *Koroido To Kaimen-Kasseizai 2,* 130). A hard surface detergent was comprised of a mixture of ammonium ortho- or polyphosphates, sesquicarbonates, tetraborates, and a small amount of alkylbenzenesulfonate (Hemingway, *Ger. 1,130,546*). Surfaces of textiles, wool, and leather have improved luster when impregnated with solutions of anionic and cationic surfactants (Du Perray, *Fr. 1,295,416*). A solid grease-absorbing composition to degrease hair is packed for use in a pressure spray container (Huber, *Brit. 889,156*). An aerosol detergent product is prepared from a normal propellant and the alkyl or alkylaryl polyoxyethylene ammonium sulfate (Charles and Pomot, *U.S. 3,055,834; Fr. 1,204,531*). An anhydrous surfactant such as an alkyl or alkylaryl polyethoxy ammonium sulfate is claimed as an aerosol composition (Societe Monsavon-l'Oreal, *Ger. 1,118,385*). An aerosol oven cleaner was patented comprising anhydrous sodium or potassium hydroxide in an alcohol solvent mixture along with a wax or a cellosolve stabilizer. Amphoteric or ionic surfactants are added (Pulman and Norden, *U.S. 3,031,408*), while the amphoteric surfactant and wax were omitted in another coverage (*U.S. 3,031,409*). Radioactive decontamination of the skin is claimed for mixtures of soaps and complex-forming amino acids (Florent, *Fr. 1,273,724*). Artificial sponges of polyurethane with large cellular structure are mixed and cured to contain "Joy" (Parker and Coultrap, *U.S.3,002,937*). Copolymers from acrylamide and N-alkylacrylamides soluble in water and alcohols are used for detergent tablet coatings and capsules (Henkel & Cie. *Brit. 901,709*). An anionic surfactant was combined with 3,5-diaminopyrazine-2,6-dicarboxylic acid diamide (Daglish et al., *U.S. 3,043,780*). A solid cleaning composition comprising a self-supporting film completely soluble in water is comprised of carboxymethylcellulose, sodium lauryl sulfate and glycerol. Details of production are given (Anderson, *Brit. 899,100*). Recommended for practical purposes in improvement in detergency with surfactants was a few percent of sodium carboxymethylcellulose (Ishizaki, *Nagasaki Daigaku Gakugeigakubu Shizen Kagaku Kenyyu Hokoku 7,* 49).

ANALYSIS

Various methods involving titration were discussed (Cullum, *Mfg. Chemist 33.* 352). Reviewed was the present state of detergent analytical chemistry (Bachmann, *Ind. Quim.* (Buenos Aires), *21,* 105). The use of ion-exchange resins in cleaning composition analysis was reviewed (Kopecky, *Chem. Listy 56,* 1033), as was the subject of another review (Voogt, *3rd. Congr.* 3,* 78). Infrared data were used on over 400 detergents for their systematic classification (Hummel, *3rd. Congr.* 3,* 104). Paper chromatographic methods were described for differentiating between anionic-cationic and amphoteric-nonionic mixtures (Tajiri, *Kogyo Kagaku Zasshi 64,* 1024). Ion-exchange resins were used in a percolation technic for separation of anionic and cationic mixtures (Arpino and De Rosa, *Riv. Italiana Sostanze Grasse 37,* 521).

A colorimetric technic was used in a differentiation test for anionic, nonionic, and cationic surfactants (Greenburg, *Chemist-Analyst 51,* 11). The separation of nonionics in mixtures with cationic and ampholytic surfactants was accomplished by ion-exchange and estimation of the nonionic by ultraviolet absorption spectroscopy. In another project, nonionic surfactants were determined directly in such mixtures by direct ultraviolet spectroscopy (Izawa and Kimura, *J. Oil Chem. Soc. Japan 11,* No. 4, 174, 180). Sodium alkanesulfonates, alkyl sulfates, and alkylarenesulfonates are determined by using an excess of zinc uranylacetate in heptane (Smirnov et al., *U.S.S.R. 145,792*). A simple and rapid analytical method for true sulfonates particularly dodecyl sulfonates in the presence of akyl sulfates, soap, or sulfonated oils was described based upon conversion of sulfonate to sulfonic acid which dissolves in butanol and is titrated with sodium hydroxide using selected indicators (Espector, *3rd. Congr.* 3,* 68). A rapid volumetric method for analysis of mixtures of carboxylated and sulfated anionic surfactants is based on a standard cationic titrant and colorimetric indicator (Achnieteli and Savino, *Proc. Sci. Sect. Toilet Goods Assoc. 37,* 4). Germicides alone, and in mixtures contained in soaps and detergents, are separated from the detergents and the germicides estimated spectrophotometrically (Jungermann and Beck, *JAOCS 38,* 513). Reviewed was apparatus and applications of gas-liquid chromatography for the analysis of a variety of surfactant components (Vasilescu, *J. Prakt. Chem. 15,* 192).

ANIONIC SURFACTANTS. Useful as a method for quality control of raw materials is the gas chromatographic determination of alkylbenzenes (Jungermann and Beck, *Soap Chem. Specialties 38, No. 5,* 72). Used as a collaborative study of alkylbenzenesulfonates in sewage, sewage effluent, and surface waters were the methods of Longwell and Maniece, Webster and Halliday, and Sallee. Significant differences were found between the methods used for each of the waters and improvements in the colorimetric methods were reported (Bolton et al., *Analyst 86,* 719). Alkylarenesulfonate in surface water was determined after removal of nitrogen-containing bodies by saponification. The acidified solution was treated with methylene blue and chloroform. The chloroform layer was separated and its absorbance measured. The measurement is disturbed by SCN-, NO_3-, and Cl-ions (Wickbold, *Ger. Mitt. Ver. Grosskeselbesitzer 65,* 106). Gas-liquid chromatography is claimed to identify completely linear detergent alkylates (Blakeway and Thomas, *J. Chromatog. 6,* 74).

Conductivity was used as a means for analysis of solutions of surfactants by p-toluidine hydrochloride (Niewiadomski et al., *3rd. Congr.* 3,* 125). Titration of anionic surfactants using p-toluidine hydrochloride, separation by carbon tetrachloride, and titration with NaOH was used (Podshibyakina, *Tekstil. Prom. 21,* No. 3, 63). Sulfated higher alcohols were determined potentiometrically using alcoholic diphenylguanidine as a titrant (Kurasova and Zolotarava, *Maslob.-Zhir. Prom. 28,* No. 2, 20). An analytical method for control of the sulfonation using the methylene blue-cationic titrant procedure was described (Milwidsky, *Soap, Perfumery Cosmetics 35,* 247). A two-phase titration suitable for sulfonates, sulfates, or carboxylates is described using thymolphthalein to form a blue salt soluble in chloroform (Kupfer, *3rd. Congr.* 3,* 62). Anionic surfactants are analyzed by reducing to the sulfonic acid, separating these in butanol, and titrating the butanol extract with NaOH using methyl orange and phenolphthalein as indicators (de Santillan and Castro, *Ind. Quim.* (Buenos Aires), *21,* No. 3, 176).

Determination of anionic surfactants of the activated sludge plant effluent should be made either immediately after taking the samples or upon a sterilized sample (Gameson et al., *Inst. Sewage Purif. J. Proc.* 1962, Pt. 4, 288).

Evaluation of the toluidine, cetavlon, benzidine, and cetylpyridinium chloride methods for 25 detergent samples showed lack of precision. Cetavlon and benzidine methods gave high results, and the other two low results (Bolzan et al., *Ind. y Quim.* (Buenos Aires), *20,* 535).

A critical review of methods for determining free alkali in soaps was made. A detailed method is given (Fiala, *Prumsyl Potravin 13,* 162). Gas chromatography has been applied to the study of soap stocks. The samples are prepared by refluxing the soap with a methanolic solution of BF_3 and separating the esters so formed by GLC (Beck et al., *JAOCS 39,* 53). Small amounts of unsaponified fats in soap can be determined colorimetrically by extracting with ethanol and by adding a ferric salt solution to the extract (Walcznska, *Chem. Anal.* (Warsaw) *5,* 663). Fatty acids in soaps and surfactants were determined by a separation technic followed by gas chromatography (Sato et al., *Bunseki Kagaku 10,* 196). The fatty acid content of soap was determined conductometrically (Vysochin et al., *U.S.S.R. 145,796*).

The details of an ascending paper chromatography of anionic surfactants were given, the spots on the chromatogram being determined with ultraviolet light after being sprayed with pinacryptol yellow (Yamamoto et al., *Kogyo Kagaku Zasshi 62,* 549). Sulfated fatty alcohols were determined by a paper chromatographic technic (Borecky, *Chem. & Ind.* (London) 1962, 265). A technic was developed for extracting microamounts of anionic surfactant bound to large amounts of protein, with subsequent spectrophotometric determination (Gould, *Anal. Chem. 34,* 567). Small amounts of surfactants are said to be detectable by acidifying with sulfuric acid and adding vanillin to form a colored ring at the interface (Meckel and Krause, *Textil-Praxid 16,* 595). An ion-exchange method was used to recover sodium xylenesulfonate from solution, and another by precipitation with p-toluidine was not successful (Hasegawa, *Kogyo Kagaku Zasshi 64,* 1828).

NONIONIC SURFACTANTS. Methods for analyzing nonionic surfactants were reviewed (Dell'Acqua, *Fibre Colori 11,* 37). Chromatography was reviewed as a procedure for analysis of these products (Nakagawa, *Koroido To Kaimen-Kasseizai 1,* 141). Polyethylene glycol in nonionic surfactants can be separated by a butanol-water immiscible solvent system (Nagase and Sakaguchi, *Kogyo Kagaku Zasshi 64,* 635). Polyethylene glycol (PEG) in ethoxylated higher alcohols can be determined chromatographilcally (Pollerberg and Heinerth, *3rd. Congr.* 3,* 89). Another chromatographic procedure was used for PEG in nonionic surfactants (Hattori et al., *Kogyo Kagaku Zasshi 64,* 1195). PEG was quantitatively determined by liquid distribution between ethylacetate and aqueous sodium chloride solution (Weibull, *3rd. Congr.* 3,* 121). Nonionic surfactants were separated from insecticide emulsions and identified by infrared spectrophotometry (Chiba, *Kagaku Keisatsu Kenkyusho Hokoku 14,* 380). PEG and their esters form a colored precipitate with flavonol and sodium tetraphenyl borate for identification purposes and can be determined by a paper chromatographic procedure (Neu, *Seifen-Oele-Fette-Wachse 87,* 657). Impurities in nonionic surfactants can be determined by gas chromatography (Nakagawa et al., *Anal. Chem. 33,* 1524).

Nuclear magnetic resonance measurements can be a valuable complement to infrared and ultraviolet analysis of nonionic surfactants (Walz and Kirschnek, *3rd. Congr.* 3,* 92). A microdetermination of nonionic surfactants is made by extraction of complexes of PEG formed with ammonium cobaltothiocyanate, with subsequent determination of cobalt colorimetrically (Morgan, *Analyst 87,* 233). When PEG-type compounds are heated in an acidic methanolic solution of 2,4-dinitrophenylhydrazine and then treated with KOH, a characteristic purple color is formed and measured (Gatewood and Graham, *Anal. Chem. 33,* 1393). Two colorimetric methods were used in the determination of a number of nonionic surfactants (Benisek and Dokovic, *Chem. Prumysl 12,* 1831). Polymer distribution of 1 through 4 ethylene oxide units in surfactant poly(oxyethylene) alkyl ethers is determined by esterification with p-phenylazobenzoyl chloride followed by chromatographic separation (Puthoff and Benedict, *Anal. Chem. 33,* 1884). The water number, and its relationship to structure, was described as a means for characterizing poly(oxyethylene) derivatives of fatty alcohols (Tagawa et al., *Yukagaku 11,* 14). Density and turbidity values were described as satisfactory methods for production control of ethoxylated fatty alcohols (Teupel, *3rd. Congr.* 3,* 177). Cloud points obtained by titrating an alcoholic solution of surfactant were used to characterize nonionic surfactants (Kaertkemeyer and Caty, *3rd. Congr.* 3,* 161). Liquid partition chromatography was used to analyze sorbitol fatty acid esters (Kariyone, *3rd. Congr.* 1,* 56). Ethylene oxide adducts may be determined by reflux with hydriodic acid and the reacted mixture titrated with sodium thiosulfate (Thamm, *Parfuem. Kosmetik. 43,* No. 8, 285). Column chromatography was used to separate a mixture of sucrose and its palmitates (Mima et al., *Kogyo Kagaku Zasshi 65,* 833).

MISCELLANEOUS. Quaternary compounds are determined volumetrically using Marme's reagent (Uno and Miyajima, *Chem. & Pharm. Bull.* (Tokyo) *9,* 326). To an aqueous solution of an anionic alkylarenesulfonate was added bromophenol blue and 0.1 N HCl until indicator turned yellow, followed by $NaHCO_3$, water and chloroform. This mixture was titrated with cationic methyl pyridinium chloride to colorless end point of the aqueous layer (Cullum, *3rd. Congr.* 3,* 42). Iron in sulfonated fatty alcohols was determined by reduction with ascorbic acid, and a phenanthroline in water-ethanol-butanol mixture to dissolve the iron precipitate (Dick and Drugarin, *Acad. Rep. Populare Romaine, Baza-Cercitaria Stinnt Timisoara, Studii Cercetaria, Stinnte Chim. 8,* 219). Flame spectrophotometry was used to determine sodium chloride in neutral detergents (Oda and Sawabe, *Bunseki Kagaku 9,* 418). A method for simultaeous titrimetric determination of boron and silicon in detergents was described (Etienne, *3rd. Congr.* 3,* 99). Phosphate in the presence of silicates was determined by the molybdenum blue method (Shen and Dyroff, *Anal. Chem. 34,* 1367). A preferential precipitation of potassium upon precipitate formation between anionic and cationic surfactants was suggested as potentially applicable to the K-Na selectivity in biological systems (van Oso, *Compt. Rend. 253,* 2946). Polarography of nickel soaps can be used to determine the nickel concentration (Malik and Haque, *Nature 194,* 863). Characterization of detergent compositions and raw materials to identify single components was claimed, using the Brabender Plastograph (Tschakert, *Fette, Seifen, Anstrichmittel 63,* 1132).

PHYSICAL CHARACTERISTICS

ADSORPTION. Chromatography of sodium alkylbenzenesulfonates was carried out on activated charcoal using methanol-benzene-water mixture. In this system the surfactant is adsorbed by both van der Waal's and ionic forces with strong dependence on molecular weight and structure (Mysels et al., *JAOCS 39,* 66). Heated immersion of graphitized carbon black in surfactant solutions was measured (Skewis and Zettlemoyer, *3rd. Congr.* 2,* 401). The adsorption of sulfur dioxide in a surfactant solution was measured in a liquid and a gas at given Reynolds numbers. The minimum of the curve of surfactant concentration versus rate of adsorption depended on the Reynolds number in the liquid (Elenkov and Ikonopisov, *Gadishnik Khim.-Tecknol. Inst. 7,* No. 1-2, 137).

Radioactive adsorption measurements showed that sodium dodecyl sulfate adsorbed on graphitized carbon takes up to one calcium ion for two dodecyl sulfate ions, the chains extending directly from the surface in close-packed array. Traces of calcium ion greatly increased deposition of the dodecyl sulfate coated carbon on cotton by bridging the surfactant on the carbon to that on the cotton (Zettlemoyer et al., *JAOCS 39,* 280). Calculations from the Gibbs adsorption law and from the Stern theory of the double layer show that there is no evidence for chemical action between the calcium ions and the adsorbed surfactant in the interface (van Voorst Vader, *3rd. Congr.*2,* 276). Radiotagged polyphosphates and calcium ion were traced in washing systems. Without detergent and soil, calcium adsorption on cotton from hard water resulted from ion exchange. Polyphosphate retention depends on the presence of multivalent metal ions and the precipitation of readily deposited calcium polyphosphates. Deposition on clay was influenced by unsequestered multivalent ions in the wash solution or attached to the cotton (Rutkowski and Martin, *Textile Research J. 31,* 892).

Penetration of wool by sodium dodecyl sulfate takes place in one, two, or three stages. In the first, equilibrium increases with concentration, pH, and added electrolyte and decreases with temperature. The second stage equilibrium was related to molecular adsorption, and the third stage was apparent only under neutral conditions (Griffith, *3rd. Congr.* 4,* 28). Cotton adsorbed sodium dodecylbenzenesulfonate between 0.04–0.4 mg/gm cotton fiber, and the presence of multivalent cations was shown as the governing factor in adsorption (Schwarz et al., *3rd. Congr.* 4,* 37). The same surfactant after 10 washings and rinsings approached asymptotically that found in sodium chloride treated cotton. The maximum is due to ions such as calcium held on the cotton (Schwarz et al., *Fette, Seifen, Anstrichmittel 64,* 57). Radiotagged dodecylbenzenesulfonate, with and without tetrasodium pyrophosphate, was traced for adsorption on fine fibers, the builders increasing the surfactant adsorption on cellulosic base fibers, but was decreased on nylon and orlon (McDonnell, *3rd. Congr.* 3,* 251). Radiotagged alkylbenzenesulfonate adsorption on sand, and on slime-covered sand showed a marked increase in surfactant adsorption on the slime-covered sand (Ewing et al., *Robert A. Taft Sanitary Eng. Center, Tech. Rept.* W61–5, 166). The adsorption of several alkylpyridinium bromides on silver iodide suspensions was determined. Zeta potentials were measured. Degree of surfactant adsorption increased with increasing hydrocarbon chain length. A rapid increase in adsorption occurred at higher concentrations suggesting the binding of micelles (Jaycock et al., *3rd. Congr.* 2,* 283). A planar surface of octadecylamine was prepared and the rate of adsorption and the equilibrium adsorption isotherm for sodium dodecyl sulfate were measured by the radiotracer technic. Adsorption was concluded as due to interaction between the positive amine surface and the negative ions of the surfactant (Shimozawa et al., *Mem. Fac. Sci. Kyushu Univ. Ser.* C4, 153). Wetting capacity toward dust, paraffin, and fabric was used to characterize the rate of adsorption of several surfactants (Konstantinova et al., *Zhur. Priklad. Khim 34,* 2658). Electrophoretic mobilities of polystyrene latex-soap systems were measured and results were explained on a modification of the Stern localized adsorption model taking into account the effect of ionic strength on the rate of soap anion adsorption by the latex particle (Sieglaff and Mazur, *J. Colloid Sci. 17,* 66).

Adsorbed layers of sodium alkyl sulfates at the interface mercury/solution gave evidence through the anodic curve of a new type of two-dimensional phase change, reversibly from the monolayer to a double layer (Eda and Tamamushi, *3rd. Congr.* 2,* 291). Specific adsorption of tetraalkylammonium iodides at the mercury/water interface was investigated and the structure of the electrical double layer was deduced (Devanathan and Fernando, *Trans. Faraday Soc. 58,* 368). With the help of oscillographic polargraphy the adsorption on mercury-drop electrode from solutions containing binary additions of surface active substances of molecular and ionic type were investigated. The data confirmed the significance of the more negative desorption peak as a parameter of determination of the efficiency of surface active substances (Dobren'kov and Bankovskii, *Izv. Vysshikh Uchebn. Zavedenii, Khim. i Khim. Tekhnol. 5,* No. 1, 75). Nonionic alkylphenyl polyglycol ethers have the hydrophobic phenol groups oriented at the air/water interface, whereas the hydrophilic oxyethylene chains are oriented down in the aqueous phase (Heusch, *3rd. Congr.* 2,* 85). The cmc and surface tension of sodium dodecyl sulfate are in inverse logarithmic proportion to the concentration of sodium salts, and to the valence of the cation, being almost independent of the valence of the anion (Matsuura et al., *Bull. Chem. Soc. Japan 35,* 552). Surface adsorption was measured by the adsorption at a gas/liquid interface for purified radiotagged sodium lauryl sulfate. It was assumed from the accumulated data that a bubble covering was by a layer of micelles whose thickness corresponded to a serrated bimolecular structure (Lauwers et al., *3rd. Congr.* 3,* 195). Adsorption rate constants were determined for a homologous series of alkyl sulfates in the presence and absence of added electrolyte. With increasing sodium chloride concentration, the rate and equilibrium adsorption increased (Matsuura et al., *Bull. Chem. Soc. Japan 34,* 1512). Adsorption phenomena for air/liquid, liquid/liquid, and solid/liquid interfaces were surveyed, including kinetics of adsorption and desorption, irreversible adsorption, and other data and methods (Alexander, *3rd. Congr.* 2,* 60). Boundary surface activity, adsorption, and association were discussed as to interrelation (Neumann and Baschant, *Z. Physik. Chem.* (Leipzig), *219,* 85). The interrelationship between adsorption and surface tension was studied on liquid/liquid boundaries of unassociated liquids (Wolf and Neumann, *Z. Physik. Chem.* (Leipzig) *219,* 60).

DETERGENCY. The physical-chemical mechanism of detergency was reviewed (Gomez, *Grasas y Aceites 13,* 28). Preferential wetting, solubilization, complex formation, penetration and mechanical action play parts in the mechanism. Substrate nature, soiling matter, and the aqueous phase determine the extent each mechanism contributes to the process. Other factors are discussed (Stevenson, *J. Soc. Cosmetic Chemists 12,* 353). Pictured was a mechanism in which the detergency of a polar dirt was a spontaneous process of penetration of soap and water into the dirt followed by peptization. Cryoscopic and diffusion processes and not surface forces are the controlling phenomena (Lawrence, *Chem. & Ind.* (London) *1961,* 1764). Detergency reaches a near maximum at concentrations in excess of cmc. In hard surface detergency the mechanism of oily soil removal was first a displacement caused by preferential wetting, followed by solubilization, emulsification, and precipitation of the acid soil. A linear relation occurred between the HLB value and the reciprocal of the ethylene oxide mole ratio for the PEG ethers of nonylphenol (Mankowich, *JAOCS 38,* 589). In hard surface detergency the 90% soil removal point and the point of twice such concentration, it was found that detergency was a linear function of micellar solubilization (Mankowich, *JAOCS 39,* 206). Studied were the detergency and surfactant properties of various normal and branched chain fatty acid sodium salts. Salts of branched chain compounds had higher cmc values and lower detergency (Nevohn et al., *Maslob.-Zhir. Prom. 28,* No. 7, 15). Various changes in several fabric characteristics as a function of multiple washing with several detergents were found (Ester et al., *Am. Dyestuff Reptr. 51,* No. 6, 25). The relationship between structure of phenyldodecane isomers as the sulfonates and their performance was determined. The nearer the center of the alkyl chain to the phenyl group the better the foaming and wetting properties (Tjepkema et al., *World Petrol Conf. Proc. 5th N.Y. 1959, 4,* 237). Mixtures of alkylbenzenesulfonates with alkyl sulfates and with nonionic products were evaluated. The ABS and alkyl sulfate mixtures were better detergents in all proportions than either component alone. The same was true for mixtures of ABS and nonionic surfactant with certain exceptions. These three surfactants mixed individually with soap showed lowest detergency with 70% soap and 30% surfactant (Nevolin et al., *Trudy Vsesoyuz. Nauch.-Issled. Inst. Zhirov* 1960, No. 20, 293).

EMULSIFICATION. A study of cholesterol and its esters showed that the water-in-oil emulsifying power of a compound is determined largely by the mechanical properties of the interfacial film it forms. The ideal emulsifying agent should provide a viscoelastic, semi-ordered interfacial phase (Truter, *J. Soc. Cosmetic Chemists 13,* 173). Addition of lower fatty acid salts to sodium dibutylnaphthalene sulfonate act similarly to electrolyte addition, while longer chain fatty acids act similarly to an increase in the emulsifier (Ivanchov and Yurzhenko, *Kolloidn. Zh. 23,* 706). The relation between solubility and emulsification of alginic acid propylene glycol esters was studied. The emulsification properties were much greater in the lower molecular weight than the higher esters (Nishide, *Kogyo Kagaku Zasshi 62,* 419). The HLB values for several types of nonionic surfactants were determined by emulsification technic. The HLB values for several oil mixtures were studied in the same way. Additivity of the required HLB values which were very large, in oil mixtures was shown to hold for saturated fatty acids, normal fatty alcohols, and some acylated esters (Ohba, *Bull. Chem. Soc. Japan 35,* 1016). A lecture discussing the properties and application of water—various solvents—dodecylbenzenesulfonic acid systems was delivered (Davidsohn, *3rd. Congr.* 4,* 165). Demulsifying effects of various surfactant substances and the structural-mechanical properties of their adsorption layers were discussed (Filatova et al., *Dokl. Akad. Nauk SSSR 140,* 874). Emulsion breaking and interfacial tension properties of water-oil systems were determined for four oils in which polyglycol ethers proved most effective as demulsifiers (Petrov, *Primenenie Poverkhn.-Aktion. Veschestv v Neft. Prom. Ufimsk. Neft. Nauchn.-Issled. Inst. Tr. Porgovo Vses. Soveshch* 1961, 112). The demulsifying properties of salts of naphthenic acid were studied (Ashimov and Rafiev, *Ibid.,* 1961, 194). Desalting of crude petroleum was improved by using nonionic surfactants (Bernadyuk, *Ibid.,* 1961, 126). Demulsification of Devonian petroleum by added surfactants and electrolytes was studied (Mavlyutova, *Ibid.,* 1961, 137). Polyoxypropylene glycol condensed with equal parts of ethylene and propylene oxides proved an excellent demulsifier for cleaning ships' oil tanks which have been ballasted with sea water (Shackleton and Moore, *Brit. 884,181*). A study of retention of higher alkane liquids in soap/hydrocarbon systems provided desorption isotherms and indicated some organic structures giving strong retention values (Groszek and Bell, *NLGI Spokesman 23,* No. 12, 467). Studies of the wax/emulsifier systems in nonionic self-glaze emulsions were made by breaking the emulsion and separating the emulsifier, those fractions of low HLB value together with residual nonalkylated polyglycols probably entering the wax phase during the emulsifying process (Kindscher, *Fette, Seifen, Anstrichmittel 64,* 449). Demulsifiers for oil-in-water emulsions were prepared by oxyalkylation of dipropylene glycol with ethylene oxide and this was used alone or by further condensing with phthalic anhydride (Monson and Jenkins, *U.S. 3,009,883*). Similarly polyalkylene polyamines can be the starting materials (Monson and Jenkins, *U.S. 3,009,884*).

INTERACTIONS. The pH values of sodium palmitate solutions were lowered by anionic, nonionic, and cationic surfactants (Bespyatov and Livshits, *Maslob.-Zhir. Prom. 28,* No. 5, 20). The exchange extraction of soaps dissolved in an organic phase of fatty acid or fatty acid solution in inert solvent with an aqueous solution of metallic salts was studied. The less alkaline metal passes into the organic liquid phase (Gindin et al., *Zhur. Neorg. Khim. 6,* 2797). Blood clotting with sodium soaps and the importance of hydrocarbon chain length was investigated. Specific actions are noted (Korsan-Bengsten and Ygge, Scand. *J. Clin. Lab. Invest. 13,* 594). Good levelling of cationic dyes on a polyacrylonitrile fiber was obtained with anionic surfactants. Spectra examination showed formation of an addition product which lowers the dye cation concentration on the fiber (Beckmann, *3rd. Congr.* 4,* 20). Nonionic surfactants interacting most intensely with certain acid and direct dyes produced increasing dyeing rates of the acid dyes for wool (Nemoto, *Kogyo Kagaku Zasshi 62,* 542). Interactions of nonionic, polymeric surfactants, and some high molecular weight alkylene oxide polymers with C.I. Acid Blue 120 dye were studied spectroscopically. Determined were molar ratios of reaction, degrees of dissociation, reaction affinities, heats of reaction, and entropies of reaction (Nemoto and Imai, *Kogyo Kagaku Zasshi 62,* 1286). Though aqueous solutions of a nonionic surfactant and a water soluble zirconium salt are stable, drying the mixture results in inactivation of the surfactant (Isoda and Nishida, *Japan. 9761('60).*

Mixtures of cationic polysoaps with nonionic surfactants were evaluated for viscosity changes, surface tension, wetting and solubilizing power (Narasaki, *Kogyo Kagaku Zasshi 64,* 1955). Metallic salts of tetradecyl sulfate were prepared and their solubilities determined (Horne et al., *3rd. Congr.* 1,* 203). Polymers like poly(vinyl formal) can be solubilized by surfactants such as sodium laurate and sodium dodecyl sulfate. Gelatin and amylose mixtures are also prepared and characterized (Isemura et al., *3rd. Congr.* 2,* 628). The properties of the interaction of sodium dodecyl sulfate with nonionic polymers such as poly(vinylpyrrolidone) or poly(vinyl alcohol) were determined (Ogata and Takahashi, *Hoshi Yakka Daigaku Kiyo 10,* 20). The mode of interaction of various types of surfactants with gelatin was studied. The surfactants combine with gelatin according to a derived isotherm. The sign of the charge on the surfactant and the length of chain affect the binding characteristics (Isemura et al., *Bull. Chem. Soc. Japan 35,* 240). Interaction of surfactants with gelatin was investigated by the flow birefringence method. The binding of surfactants was due to energetic stabilization simultaneously with the formation of a large hydration lattice between the nonpolar side of the surfactant and the protein side chain (Isemura and Imanishi, *Ann. Pept. Sci. Works, Fac. Sci. Osaka Univ. 8,* 93). Formation of precipitates or soluble complexes between solutions of zinc chloride and surfactants was investigated by pH and conductivity titrations, and precipitates were characterized by chemical analysis and x-ray diffraction (Vold and Singh, *JAOCS 39,* 424).

MICELLES. Nephelometry, dye titration and surface tension gave concordant values for minimum and maximum concentrations for micelle formation with aqueous solutions of esters of sulfosuccinic acid, dodecylammonium acetate, and a polyethylene oxide tertiary-butylphenol product (Taubman and Nikitina, *Doklady Akad. Nauk. SSSR 135,* 1179). Two new methods for determination of critical micelle concentration (cmc) were given: transference numbers through diffusion potentials along a concentration gradient, and determination of counterion activity coefficients by membrane potentials, useful both for anionic and cationic surfactants (Botre et al., *3rd. Congr.* 1,* 302). Nonionic surfactant cmc values were determined using a Benzopurpurin 4 B titration method and results were in agreement with surface tension and iodine methods for two nonionic surfactants tested (Becher, *J. Phys. Chem. 66,* 374). Micellar weights of anionic-nonionic mixtures were determined by light-scattering measurements. It was postulated, and supported by data, that interaction occurs with formation of a single species of mixed micelle system (Mankowich, *U.S. Dept. Com. Office Tech. Serv. PB Report* 129,486). Micellar charge of dilute aqueous detergent solutions were measured by conductivity. The linearity of the equivalent conductance of the ionic solution breaks at the cmc and this was attributed to the presence of high-valency micellar ion increasing the ionic strength of the solution (Kimizuka and Satake, *Bull. Chem. Soc. Japan 35,* 251). Various micellar properties of pure alkyl polyglycol ethers were determined (Lange, *Fette, Seifen, Anstrichmittel 64,* 457). Flow birefringence studies of extraction angles lead to the conclusion that these surfactant micelles act as rigid bodies (*Kuroiwa, Kogyo Kagaku Zasshi 63,* 1968). The temperature dependence of the anhydrous weight-average micellar molecular weight of a polyoxyethylated nonylphenol was determined by analytical ultracentrifugal determinations (Dwiggins and Bolen, *J. Phys. Chem. 65,* 1787). Variation in the effect of temperature upon the micellar molecular weight of an anionic-nonionic surfactant mixture was determined by measurement of scatter of the green mercury line at 5461 A (Kuriyama, *Kolloid-Z. 180, 55*). The effect of addition of sodium or calcium chlorides to a nonionic binary mixture was determined by measurement of light scattering. At fixed temperature, micellar weight increases with salt addition, and increases with rising temperature at a fixed salt concentration. Increase in micellar molecular weight with rising temperature is a dominating factor in cloud formation and phase separation (Kuriyawa, *Ibid., 181,* 144).

The micellar weight, cmc and second virial coefficient of mixtures of methoxy poly(oxyethylene)dodecyl ether, and sodium dodecyl sulfate were determined from light scattering, and cloud points. Values for the nonionic surfactant increased with temperature rise while those of the anionic decreased (Kuriyama et al., *Ibid., 183,* No. 1, 68). The cmc values of several homologous sodium alkyl sulfates were determined over a range of 10–70C and an equation of the Clausius-Clapeyron type was used to calculate change in heat content associated with micelle association. In every case heat content was positive to 20C and then decreased becoming negative at 30–40C. Increased chain length decreased heat content. Shift of the sulfate group to mid-chain increased heat content (Flockhart, *J. Colloid Sci., 16,* 484). Increased pressure of from 1–2000 atmospheres of sodium dodecyl sulfate solutions caused increased cmc up to 1000 atmospheres, decreasing slightly thereafter (Hamann, *J. Phys. Chem. 66,* 1359). Previous work had indicated a change in pH of metal dodecyl sulfates at the cmc, but this work indicates that this happened only when alkali was added to the solution (Matshura et al., *Bull. Chem. Soc. Japan 35,* No. 6, 1050). Specific conductance and pH of soap solutions were measured and compared favorably with those of other investigators (Eagland and Franks, *Nature 191,* 1003). The effect of electrolyte addition upon the micellar characteristics of several nonionic surfactants showed lowering of cmc analogously with ionic surfactants but little increase in micelle size except with the least soluble compounds (Becher, *J. Colloid Sci., 17,* 325). Cylindrical micelles of high molecular weight were formed of sodium(tripentylmethyl)benzenesulfonate in n heptane using dissemination and diffusion technics (Reerink, *3rd. Congr.* 1,* 255). The cmc of sucrose monoesters of saturated fatty acids were measured by surface and interfacial tension measurements, a direct proportionality between cmc values and carbon number of fatty acids was discovered (Wachs and Hayano, *Kolloid-Z. 181,* 139). The cmc values for some polyoxyethylene glycol monohexyl ethers in binary and ternary systems were determined by surface tension measurements. No changes in cmc were observed in systems containing any one of several organic solvents, suggesting shifts in cmc in the presence of solubilized materials occurring only when ionic effects are important (Mulley and Metcalf, *J. Colloid Sci. 17,* 523). The nonionic-cationic micellar properties of dimethyldodecyl amine oxide were determined by light-scattering methods. Differences in cmc, micellar molecular weights and interaction coefficients in water or in sodium chloride solution were recorded and discussed (Herrmann, *J. Phys. Chem. 66,* 295). The cmc values for naphthenate soaps and their solubilizing properties were determined (Demchenko, *Ukrain. Khim. Zhur. 27,* 322). The cmc values for homologs of alkylbenzenesulfonates, alkyl sulfates, soaps of naphthenic acids and soaps of saturated fatty acids were determined and discussed. Low molecular weight surfactants having a cmc greater than 7g/liter are poor detergents, but better wetters or flotation reagents. High molecular weight homologs of cmc less than 0.3 g/liter are sparingly water soluble and not effective as detergents. Other conclusions are drawn (Demchenko, *Kopovidi Akad. Nauk. Ukr. RSR 1961,* 928).

Micellar solute species in a coacervate system is one of relatively low charge density. The addition of eletrolyte to such systems produces a tremendous growth of the micellar species prior to two-phase formation (Cohen and Vassiliades, *JAOCS 39,* 246). Detergent solutions were measured conductometrically to investigate a reported slow micelle dissociation but none slower than 2.5 min was observed (Phillips and Porter, *3rd. Congr.* 1,* 239). A critical study of the micellar structure of several nonionic surfactants showed much lower cmc values than ionics with comparable hydrophobic groups. The cmc decreases with increasingly long hydrophobic chain length. Aggregation numbers increase with increase in hydrophobic group length. For aggregate molecular weight regions of 45,000–100,000, spheres appear to be the most probable shape, and for larger micelles disks and rods (Schick et al., *J. Phys. Chem. 66,* 1326). It was theorized that different micelles or aggregates present in solutions actively participate in the formation of mesomorphous phase (Ekwall et al., *3rd. Congr.* 1,* 189). Cloud points of nonionic surfactants were used to study the effects of various inorganic chlorides and organic solutes on the postulated mantle of water molecules held by hydrogen bonding. Data developed are to be used to separate the processes of micelle penetration, solubilization and hydrotropism, and their effects on nonionic surfactants (Bolle, *3rd. Congr.* 1,* 294). An x-ray diffraction study of the mesomorphic phases of some alkaline soaps showed all to have a structure corresponding to aggregation of the polar groups into ribbons, but differing from one another in arrangements in space due to differing crystal habit. Lamellar structures just below the melting point are related to melting of the polar groups (Gallot and Skoulios, *Acta Cryst. 15,* 826). In a correlation between critical micelle concentration, fatty soil removal, and solubilization, it was shown that soil removal began at or near cmc, that maximum detergency occurred at concentrations considerably in excess of cmc. Direct correlation between cmc, solubilization, and soil removal was demonstrated (Ginn and Harris, *JAOCS 38,* 605).

PROPERTIES AND STRUCTURE. The distribution of commercial soap components in an aqueous medium as a function of temperature was determined (Dervichian and Prevot, *Rev. Franc. Corps Gras 9,* 96). The proton-magnetic-resonance absorption of anhydrous potassium soaps was determined. Pronounced line width changes correspond to known phase transitions (Grant and Dunell, *Can. J. Chem. 39,* 359). Of 15 branched chain aliphatic soaps the branched acids of the same molecular weight those with longer side chains possess greater flotation activity and their efficiency further increased by adding more high molecular weight acids of normal structure (Aleinikov et al., *Neftekhimiya 1,* 418). Presence of sodium tridecanedicarboxylates in mixtures with the mono acid soap enhanced the detergent properties (Asinger and Bochnia, *J. Prakt. Chem. 13, 1*).

The structural characteristics of several sodium soaps were determined by x-ray diffraction data (Ogino, *Kogyo Kagaku Zasshi 64,* 1021). Various mesomorphic phases of magnesium stearate were measured by x-ray diffraction (Spegt and Skoulios, *Compt. Rend. 254,* 4316). Distributions of the degree of polymerization of condensates of ethylene oxide with lauryl alcohol and nonylphenol were determined by precise fractional distribution. Comparison of these data with the results of Poisson's and Weibull's formulas indicated the latter as the more satisfactory (Nagase and Sakaguchi, *Kogyo Kagaku Zasshi 63,* 588). The structure of ethylene oxide condensates either anhydrous or in water was discussed. X-ray diffraction data, melting point, viscosity curves, and rapid freeze technics were used. Ethylene oxide condensation chains consist of equidistant, antiparallel dipoles arranged in zigzag pattern, and contraction occurs upon rearrangement in meander structure when in aqueous solution. Other data given (Roesch, *3rd. Congr.* 1,* 163). Van der Waal's force between alkyl groups of adjacent surfactant nonionic alkylphenol molecules had a dominant effect upon surface activity when the alkyl group was long (Ishii et al., *Kogyo Kagaku Zasshi 64,* 1794). Various physico-chemical properties of pure dodecyl polyglycol ethers were determined (Lange, *3rd Congr.* 1,* 279). The HLB, physiological, and surface properties of sucrose nonionic esters were given and compared with other nonionics (Passedollet, *Parfums Cosmet. Savons 4,* 350). The properties of aqueous solutions of sodium p-(n-alkyl) benzenesulfonates were examined as a function of chain length, concentration, and temperature. Determined were emulsifying, dispersing, and washing powers. The washing effect improved initially and remained constant above the cmc. The temperature influence on washing was almost equal for all C_8 to C_{18} chains of the alkyl products (Koelbel et al., *3rd. Congr.* 4,* 173). The surface properties of 2,5-di-n alkylbenzenesulfonates were described. Both water solubility and cmc decreased with increasing side-chain length. Other data given (Koelbel et al., *3rd. Congr.* 1,* 27). The structure and surface active properties of sulfate salts of ricinoleo and its acetoxy glycerides were determined (Subrahmanyam and Achaya, *J. Chem. Eng. Data 6,* 548). Surface tension, cmc, wetting, and foaming powers, and detergency for a series of various sulfates and sulfonates were given (Koelbel et al., *3rd. Congr.* 1,* 1). The surface activity of monoglyceride monosulfates was described (Jedlinski, *3rd. Congr.* 1,* 51). An optimum concentration was found for each of several surfactants and a detergent composition for the suppression of ripples to attain a smooth water surface (Tailby and Portalski, *Trans. Inst. Chem. Engrs.* (London) *39,* No. 5,328). Theoretical and practical work with some experiments in damping waves and ripples were reviewed (Davies, *Chem. & Eng.* (London) 1962, 906). The interfacial resistance due to thin films of straight-chain higher alcohols and their effect on water evaporation were measured. Cetyl alcohol was most effective, next stearyl alcohol, and least lauryl and myristyl alcohols (Udani and Gordon, *A.I.Ch.E. Journal 5,* 510). Diffusion of hydrogen sulfide and carbon dioxide across the air/water interface in the presence of surface films was measured (Hawke and Alexander, Retardation Evaporation Monolayers, Papers, Symp. New York, N.Y. 1962, 67). A method was used where a thermistor determined the surface temperatures of water layers and th effect of insoluble monolayers upon evaporation (Jarvis et al., *Ibid.,* 1962, 41). Spreading and pressure evolution rates of hexa- and octadecanol and their mixtures were studied. Small quantities of nonvolatile paraffin oil improved spreading properties (Miller and Bavly-Luz, *Ibid.,* 1962, 161). While addition of ordinary strong electrolytes raises the vapor pressure of aqueous ethanol, pure surfactants have the opposite effect. At higher concentrations the interaction between micelles and alcohol molecules is sufficiently strong to overcome the surfactant-water interaction (Flockhart, *J. Colloid Sci. 16,* 443).

SOLUBILIZATION. Lyophilic colloids such as lecithin added to potassium laurate doubled the amount of methylbenzene solubilized, but none of these colloids solubilized methylbenzene alone (Demchenko and Dumanskii, *Doklady. Akad. Nauk. SSSR 140,* 398). Reviewed was solubilization by nonionic surfactants (Kitahara, *Koroido To Kaimen-Kasseizai 1,* 30). Increase in micellar weight of nonionic surfactants depended on the number of surfactant molecules in the micelle, which increased with added solubilizate, thus solubilization is accompanied by reconstruction of micelles (Nakagawa, *3rd. Congr.* 2,* 636). Addition of potassium sulfate and to a lesser degree, of potassium chloride and nitrate increased the solubilization of methyl benzene and decreased the cmc of potassium laurate solutions (Demchenko, *Ukr. Khim. Zh. 28,* 46). The solubilization of toluene and heptane in sodium laurate solutions by sodium phosphate and polyphosphates was indicated as causing two-phase separation and improvement in colloidal properties (Demchenko, *Maslob.-Zhur. Prom. 28,* No. 2, 23). Greater solubilizing capacities of polymeric phosphates is explained by association in the micelles, lessening the repulsion of the hydrophilic groups, increasing the micelle dimensions, and increasing the oleophilicity of the system (Demchenko and Dumanskii, *Doklady Akad. Nauk. SSSR 139,* 919). Small quantities of highly fluorinated insoluble alcohols can be solubilized in conventional surfactants and thus lower surface tension. A mutual solvent may be used to increase the solvent power of the continuous phase (Bernett and Zisman, *J. Phys. Chem. 65,* 448).

SUSPENSIONS. Sediment activity and thixotropy were reviewed (Hofmann, *3rd. Congr.* 2,* 658). A chromic oxide powder was used in suspension studies of three types of surfactants (both built and unbuilt) at two temperature levels. The powder was suspended well by both anionic and nonionic surfactants and temperature coefficients were small: negative for an anionic and a nonionic product and positive for sodium oleate. Suspension isotherms were typical and explained by initial adsorption of surfactant anions on the particles until maximum zeta potentials were reached, then always dropped in suspendability due to increasing sodium ion effect (Mankowich, *U.S. Dept. Com. Office Tech. Serv. PB Rept.* 150,089). Dispersability of several powders in solid paraffin was studied. Addition of surfactants appeared to be specific in action (Mugishima et al., *Kogyo Kagaku Zasshi 63,* 90).

VISCOSITY. The fundamental role of high viscosity at the interface was demonstrated by the fact that the most stable oil-in-water emulsions, depending upon a combination of surfactant and electrolyte for their stability, gave the highest viscosities when measured at the interface of continuous phases of the corresponding compositions (Rebinder and Taubman, *3rd. Congr.* 1,* 209). Viscosity and density of potassium oleate solutions were measured. Two transitions at 40C and 60C exist as was evidenced by thermal analysis; that at 40C is endothermal (Fukutomi, *Oyo Butsuri 30,* 142). The viscosity of sodium ricinoleate solutions containing p-cresol, sodium chloride, or both was measured at 20–80C. Unlike sodium oleate or stearate the ricinoleate does not form micelles (Angelescu and Popescu, *Rev. Chim. Acad. Rep. Populaire Roumaine 6,* 73). The intrinsic viscosity of aluminum stearate was measured immediately after dissolution in benzene which represents well the degree of polymerization (Nakagaki and Nishino, *Yakugaku Zasshi 82,* 821). The properties of aqueous solutions of mixtures of cationic polysoaps were measured. The low viscosity decreased as the monosoap content increased, and other properties were determined (Narasaki and Ito, *Kogyo Kagaku Zasshi 64,* 1790). The viscoelasticities of solutions of polyethylene glycol dodecyl ether and poly(oxyethylene)nonylphenol of varying degrees of polymerization were measured with an oscillational rheometer. Both viscosity and rigidity were maximum at about 60% (Kuroiwa, *Kogyo Kagaku Zasshi 63,* 1384). With the same nonionics viscoelasticity decreased remarkably at less than 20C. Rigidity of an 80% solution decreased with increasing temperature but was constant at less than 40–50C (Kurowai, *Ibid., 63,* 2065). Surface mobility of surfactant solutions was directly proportional to the opposite hydrostatic pressure (Chifu and Cadariu, *Studia Univ. Babes-Bolyai Ser. 1,* No. 2, 19). Viscosity maxima for ethoxylated dodecanol and cocoanut fat amides were determined. Gel formation was common, some products gelling in a narrow range, others over a broader range (Schoenfeldt, *3rd. Congr.* 1,* 173). The viscosities of oil/water emulsions stabilized by nonionic fatty ester products were found to give maxima related to the HLB valence (Ohba, *Bull. Chem. Soc. Japan 35,* 1171). The temperature at which nonionic surfactants form nonspherical micelles in water and consequently non-Newtonian flow, depends on the sample nature. Unsaturated alkyl groups are favorable to non-Newtonian flow (Kurowai, *Kogyo Kagaku Zasshi 62,* 1588).

Biological Degradation. A comprehensive review on the production, uses, economics, determination, removal from water, biological and toxicological properties, and occurrence in sewage, ground water, and drinking water of detergents appeared (Heinz and Fischer, *Fette, Seifen, Anstrichmittel 64,* 270). Reviewed was a symposium on improved methods for treating waste waters (Marshall, *Chem. Eng. 69,* No. 16, 107). Detergents in water supplies were discussed (Olivier, *Water & Sewage Works 108,* 271). Presence of alkylbenzenesulfonate cannot be tasted or smelled until it is many times the level ever found in surface, ground, well, or any kind of drinking water (Justice, *Soap Chem. Specialties 38,* No. 3, 51). Methods for overcoming the foaming nuisance in sewage-treatment processes were described (Truesdale, *Chem. Prods. 24,* 499). The disturbing effect of anionic detergents on the biological purification of waste waters and autopurification of river waters was discussed. Adsorbability on activated carbon, charcoal, pond mud, and humus is indicated (Chambon, *Eau 48,* No. 11, 329). A general inhibition of anaerobic bacterial digestion of organic sludge components, particularly of fatty acids is exhibited by the presence of detergents (Malz and Jendreyko, *3rd. Congr.* 3,* 315). The amount of alkylarenesulfonate in the Isar River and canal in the Munich area was determnied to be 4–10 times as large on Monday as on other days of the week, increasing with a dry season (Gebhardt, *3rd. Congr.* 3,* 287). Reviewed were waste disposal methods and detergent production in different countries including Poland (Kucharski, *Chemik* (Gliwice)*15,* 94).

A flocculation-flotation system for removal of both emulsified oils and greases and some detergents is described, aeration being used to float detergent scum which is held in a pond to condense, and the sludge hauled away for land disposal (Anon, *Water Waste Treat. J. 9,* No. 1, 19). An olefinic side chain in alkylbenzenesulfonate detergents caused less foam and less residual anionic product in sewage plant effluents than straight or branched chain aliphatic detergents (Tarring, *3rd. Congr.* 3,* 324). Resistance to biodegradation of ABS is caused by branching of the carbon chain. For nonionics resistance increases with the number of ethylene oxide units and the degree of branching (Huyser, *3rd. Congr.* 3,* 295). Biologically soft detergents are prepared by treating straight chain monoolefins with monocyclic aryl compounds (Jones, *Belg. 612,036*).

Ozonation of sewage influents and effluents was effective for foam control and ABS destruction (Buescher and Ryckman, *Purdue Univ. Eng. Bull. Ext. Series No. 109,* 251). Vigorous aeration of settled sewage causes foam in which detergent concentrates and is removed (Burgess and Wood, *Inst. Sewage Purif. J. Proc.* 1962, 158). Branched chain alkylbenzenesulfonates of known constitution were tested for ease of biodegradation and 94% of the soft detergent can be biologically removed as compared with 67% of the hard type (Eden and Truesdale, *Water & Sewage Works 108,* 275). Since tetrapropylene benzene sulfonate resists biological degradation it is proposed that aeration be increased and the foam be skimmed off for evaporation and combustion (Husmann, *Textil-Rundschau 17,* 88). Aeration and foam removal is suggested for a village of 3000 population (Klotter, *3rd. Congr.* 3,* 302). Foam flotation is not a substitute for sedimentation but intensified mechanical treatment and increased sludge separation. Foam can be reduced by spraying with fresh sludge (Klotter and Kuhn, *Stadedthygiene 9,* 235). No correlation between ABS foaming and flavor concentration (8–20 ppm), or amount of carbon to correct foam was found. Activated carbon 1–2 ppm of syndet was suggested to correct both foam and flavor (Sigworth, *Taste Odor Control J. 27,* 9). Wastes from store laundries can be treated with alum and activated carbon to produce a clear effluent containing less than 1–2 ppm syndet as ABS (Eckenfelder and Barnhart, *Water & Sewage Works 108,* 347). Removal of ABS by precipitation with hydroxides of bi- and trivalent metals appeared favorable and was recommended for effluent treatment from laundries and institutions when ABS content is high (Spohn, *3rd. Congr.* 3,* 319). Reduced rate of sedimentation resulted when detergent was added to sewage prepared with hard water and pH adjusted to 6.7 with sulfuric acid (Swanwick and White, *Water Waste Treatment J. 8,* No. 9, 440). Ferrous sulfate and aluminum sulfate were tested as coagulants for anionic surfactants, greatest coagulation occurring when the operation took place in acid media (Pitter and Chudoba, *Vodni Hospodarstvi 12,* 164).

Miscellaneous Properties. Gelation of 1% soap solutions occurs in 100 hours at 1–6C below the titer point of the fatty acids derived from the soaps (Ganguli et al., *J. Sci. Ind. Research* (India) *20D,* 389). The properties and uses of anionic and nonionic surfactants were reviewed (Moilliet, *Office Dig. Federation Soc. Paint Technol. 33,* 1103). Characteristics of Brij surfactants were discussed (King, *Drug Cosmetic Ind. 90,* 24). The latest developments in detergents and surfactants with regard to Japanese production, and other problems were discussed (Raphael, *Mfg. Chemist 33,* 114). Polyethylene glycols, Spans and Tweens were discussed (Ungar, *Horokeach Haivri 8,* 529).

A lecture was given concerning generation of foam by alkyl sulfates in flotation (Schubert et al., *Freiberger Forschungsh. A231,* 58). Surfactant bubble size is constant at low gas flow rates, increases with increasing gas flow at high flow rates, and bubble surfaces change constantly under the influence of time, surface tension, and other physicochemical properties (Mancy et al., *Conf. Biol. Waste Treat. Manhattan Coll., New York, N.Y.*). Loss of organic substance during spray drying of alkyl sulfates may amount to as much as 20% of the fat alcohol used. Alkyl sulfate was heated in a stream of hot dry air and the volatile organic substance was recovered, and analyzed by gas chromatography. Olefins and lower hydrocarbons were formed but the origin of undecene could not be explained (Schaurich, *3rd. Congr.* 1,* 199). The toxicity to fish in a laboratory experiment was determined using a nonionic surfactant based on octylphenol, and sodium dodecylarenesulfonate. Short term exposures to 15–500 ppm were toxic. Longer term exposure at 3 ppm levels were also toxic (Wurtz-Arlet, *3rd. Congr.* 3,* 329). A number of surfactants when introduced into the eye mucosa were capable of producing an anesthesia or an increase in the pain threshold. Agents exhibiting these phenomena were alkylaryl compounds condensed with polyethylene oxide, as well as similar fatty acid amine condensates (Martin et al., *Drug Cosmetic Ind. 91,* No. 1, 30). Binary and ternary phase diagrams with additional data were given for Brij and Tween surfactants (Mulley, *J. Pharm. & Pharmacol. 13,* Suppl 205T). Formation of hydrophilic adsorption layers by surfactants on metal surfaces was shown to improve electrodeposited films (Gristan and Shun, *Dopovidi Akad. Nauk. Ukr. R.S.R. 1961,* No. 1, 64). Several alkenylsuccinic acid derivatives were prepared and their wetting properties were determined in connection with the preparation of photographic emulsions (Smirnov et al., *Sbornik Statei, Nauch. Issledovatel. Inst. Org. Popuprod i Krasitelei 1961,* No. 2, 168). The surface activity of several polysoaps was determined. Conclusions concerning their physicochemical properties were drawn (Jorgensen and Strauss, *J. Phys. Chem. 65,* 1873). Sorbitan and glyceryl monostearate properties in benzene solutions were studied. Equilibrium ultracentrifuge measurements support the results of vapor-pressure lowering determinations and not those of light scattering (Sirianni et al., *Can. J. Chem. 40,* 957). Differences were found in the effect of various surfactants upon the decomposition of cellulose (Klust and Mann, *Vom Wasser 27,* 99). Alpha-monoglycerides in nonaqueous solvents were studied for molecular weight, dipole moments, surface tension, light scattering, hydrogen-bonding, sequestering of acids, and dye solubilization (Debye and Coll, *U.S. Dept. Com. Office Tech. Serv. PB Rept.* 146,513). The colloidal properties of pure sodium mono- and dibutylnaphthalene sulfonates were studied (Muresan-Kertesz et al., *Rev. Chim.* (Bucharest) *11,* 71). The Siemens electron microscope was used to obtain single-crystal electron diffraction patterns from selected areas of particles of soaps and fats a few microns across (Camp, *Proc. European Regional Conf. Electron Microscopy*, Delft *1*, 235). The absorptive properties of cobalt oleate and stearate for oxygen, nitrogen, and hydrogen were determined (Funakoshi, *Bull. Chem. Soc. Japan 35*, 1025). The appearance, densities, and x-ray analysis of crystal structures of cobalt soaps were determined (Kambe, *Bull. Chem. Soc. Japan 35*, 78). The luteo- and purpureo-cobalt stearates were shown to be ionic soaps (Kambe, *Ibid.*, 35, 388).

PERFORMANCE AND USE TESTING

The electropotentiometric titration technic supplemented by the stalagmometric method were used as a measurement of detergent skin-irritating capacity. Epicutaneous tests with ABS, dodecyl sulfate, and other surfactants showed surprisingly frequent allergic reactions (Nejedly and Piper, *Arch. Klin. u. Exptl. Dermatol. 213*, 434). The influence of soap on the permeability of the epidermis was determined by clamping separated human abdominal epidermis between plastic cylinders so that the test solution was in contact with one surface and an isotonic solution with the other. Penetration of potassium oleate was measured using the radiotagged molecules (Bettley, *Brit. J. Dermatol. 73*, 448). Test methods for surfactants used principally in Europe were discussed (Schwen, *3rd. Congr.* 3*, 127). Discussed were the principles and methods of radio chemistry applicable to surfactant evaluation (Kuechler, *3rd. Congr.* 3*, 235). Surface tension of liquids was measured by maximum pressure in gaseous bubbles or in a drop (Timofevicheva and Lazarev, *Kolloidn. Zh. 24*, 227). The statistical theory of surface tension was discussed (Chang et al., *Progr. Instrn. Res. Thermodyn. Transport Properties Papers Symp. Thermophys. Properties, 2nd. Princeton, N.J. 1962*, 88). A film tensiometer was designed and used for the continuous measurement of surface tension of liquids and solutions (Peterson, *Kolloid Z. 183*, 141). Problems in wetting were discussed, covering various methods for measurement in capillaries, fabric, and solution (Sand, *Kolloid Z. 183*, 57). Comparison of a "blast" mehod involving increase in surfactant concentration to halve the surface tension of water, with a dipping method to secure sinking in 100 sec, showed lesser variation in results with the former (Schwen and Blum, *Melliand Textilber. 43*, 514).

The wetting power of surfactants was evaluated using a canvas disk method and extrapolation to the concentration required to immerse the disk in 100 sec, but reproducible results were difficult to attain because of disk variation (Goette, *Grasas y Aceites 12*, 255). Using the German standard DIN 53 901 procedure a standard procedure is under development depending upon penetration of a gauze disk (Goette, *Chem. Weekblad 58*, No. 18, 201). A new semimicro disk wetting test (by horizontal or vertical means) was developed and agrees well with macro tests (Yano et al., *J. Oil Chem. Soc. Japan 11*, No. 4,183). A semi-micro continuous method was developed (Yano and Kimura, *Ibid.*, 190).

A method for measuring sudsing and stability of foam used nitrogen introduced through a fritted glass disk into a cylinder. The displacement of liquid level was compensated by gas pressure. Several surfactants were evaluated and the data discussed (Lauwers and Ruyssen, *3rd. Congr.* 3*, 146). Measurement of drainage rates of liquid films by movement of interference fringes showed that the cross section of a fast-draining film is wedge-shaped and its average thickness equals the thickness at its mid-height (Thompson, *J. Appl. Chem.* (London) *12*, 12). Drainage times were determined and the factors of surfactant and foam promoter structure, relation between draining properties and their form, visual aspects, and liquid-retaining capacities (Spitzer, *3rd. Congr.* 2*, 556), were studied. A lecture on detergents and detergency was delivered (Roesch, *Z. Ges. Textil-Ind. 53*, 771). The redeposition of a hydrophobic pigment soil and the influence of detergent concentration were studied for anionic and nonionic surfactants and the soil suspending power passes through a maximum at a given concentration (Vaeck and Brouwer, *Fette, Seifen, Anstrichmittel 63*, 1065).

A statistical approach to detergency evaluation was made and though the chemical compositions were identical significant differences were found, and these differences corresponded to small structural variations determined by gas chromatographic analyses of the alkylbenzenes after desulfonation (Jungermann et al., *JAOCS 39*, 50). A standardized detergency evaluation procedure was developed using the Tergotometer and a commercially available standard soiled fabric (Linfield et al., *JAOCS 39*, 47). The "stretchability" of liquid molecules in a special apparatus was claimed to differentiate between wetting and detergent action, higher detergency occurring with molecules with the greater ratio of length to cross section (Vallee and Guillaumin, *3rd. Congr.* 3*, 151). A recording photoelectric reflectometer was described capable of measurement of change in reflectance during the washing process (Bertsch et al., *Fette, Seifen, Anstrichmittel 63*, 843). Analysis of cotton bed sheets after usage showed the presence of small amounts of calcium, magnesium, other alkaline matter, iron, aluminum, phosphorous and silicon. These data were sufficient to indicate the approximate amounts of inorganic constituents to be incorporated into a synthetic soiling mixture (Wagg and Kwan, *J. Textile Inst. Proc. 53*, T102). Radioactive iron as ferric oxide was used as a soiling ingredient for soiling cotton yarn for a detergency test. Effectiveness of the system was discussed (Sauerwein, *3rd. Cong.* 3*, 265). A satisfactory procedure for class-room demonstration of soap making was described (Richardson, *School Sci. Rev. 43*, 730).

MISCELLANEOUS

The detergent statistics for Japan were discussed (Anon, *Soap Chem. Specialties 37*, No. 11, 55). Surfactants were reviewed (Niyazov and Karryev, *Tr. Inst. Khim. Akad. Nauk Turkm. SSR 1*, 52). Detergent materials and recent developments were described (Pueschel, *Nahrung 6*, No. 4, 388). The present state of detergent chemistry was described (Jaag, *Parfum. Cosmet. Savons 5*, 284; Chima (Switz) *15*, 450). New surfactants were reviewed (Kuwamura, *Koroido to Kaimen-Kasseizai 2*, 406). Fatty acid derivatives of aluminum, titanium, and zirconium were reviewed (Mehrotra, *J. Indian Chem. Soc. 38*, 509). Reviewed were surfactants of the polyhydroxy alcohol-ester type (Konishi and Takada, *Koroido to Kaimen-Kasseizai 1*, 150), and surfactants based on sucrose (Ames, *Trop. Sci. 4*, 64). Classification of surfactants is based on four control groups, each subdivided into three subgroups. Eight rules are proposed for use of the classification which is proposed by the Comite International de la Detergence (Anon, *Melliand Textilber. 42*, 1054). The basis for a decimal classification of surfactants is given (Bolle, *3rd. Congr.* 3*, 3). A linear decimal notation for classification is described (Quaedvlieg, *3rd. Congr.* 3*, 7). Based upon the proposed decimal system, punched cards can be used for documentation of surfactants (Gottschaldt, *3rd. Congr.* 3*, 18). Reviewed were surfactants for the paper industry (Poschmann, *3rd. Congr.* 4*, 121). The use and importance of surfactants in the petroleum industry were reported (Rebinder, Pirmenenie Poverkhn.-Aktivn. Veschestv v Neft. Prom. Ufimsk. Neft. *Nauchn.-Issled. Inst. Tr. Pefrvogo Vses. Soveshch.* 1961, 8). Surfactants in the ceramic industry were reviewed (Sazawa, *Koroido to Kaimen-Kasseizai 3*, 45).

PRODUCTS (EXCEPTING DETERGENTS)

EDIBLE, PHARMACEUTICAL, AND COSMETIC PRODUCTS

In a study of the fatty acids of cow's milk, gas-liquid chromatography was supplmeented by iodine values, UV and IR spectrophotometry and hydrogenation (Magidman, et al., *JAOCS 39*, 137). Further refining this procedure, the milk fatty acids were fractionated by distillation into 11 fractions and these submitted to adsorption chromatography in addition to GLC (Herb, et al., *JAOCS 39*, 142). Twenty-seven minor components were identified. A review of the biochemical properties of milk and the factors leading to lipid deterioration in milk and milk products (Krukovsky, *J. Agr. Food Chem. 9*, 439) noted that light catalyzed reactions and fat splitting by lipase are important factors.

C-18 polyethenoic acids of butter fat were prepared by low temperature crystallization and fractional distillation, chromatographed on a silicic acid column and the fractions analyzed by UV and IR (Sambasivarao and Brown, *JAOCS 39*, 340). A major portion of non-conjugated dienoic acids other than linoleic had widely separated double bonds with cis-trans configuration. Gas-liquid chromatography showed the presence of a C-15 saturated acid, a branched chain C-17 saturated acid and a heptadecenoic acid. Trans-octadec-16-enoic acid was isolated from summer butter in an amount equal to 0.2% by weight of the total acids (Hansen and Cooke, *Biochem. J. 81*, 233). Chlorinated hydrocarbon pesticide residues in butterfat were removed by silicic acid chromatography (Moats, *J. Assoc. Offic. Agr. Chem. 45*, (2), 355). A patent was issued (*Coulter, U.S. 3,017,275*) for improving the spreadability of butter by agitation in direct contact with a refrigerated brine.

A coconut type oil interesterified with the triglycerides of fatty acids having 8,10,16, and 18 carbon atoms was patented as a fat composition for incorporation in margarine (Babayan, *U.S. 3,006,771*). Satisfactory margarine stocks were also prepared from soybean oil, peanut oil, and a 70:30 mixture of soybean oil and cottonseed oil by hydrogenation at 10–50 psi, using Pd on charcoal as catalyst (Zajcew, *JAOCS 39,* 301). Another margarine composition consisted of an oil in hydrated fatty carboxylic acid soap emulsion, the oil phase constituting at least 80% of the margarine and containing at least 20% polyunsautrated fatty acids (Murray, *U.S. 3,026,207*). A process for manufacture of margarine free of occluded air was described (Elwood, et al., *U.S. 3,033,689*). To make margarine antispattering, it was treated with a mixed ester of an aliphatic polyalcohol, a fatty acid residue and an aliphatic polycarboxylic acid under conditions causing interesterification (Houben and Jonker, *U.S. 3,006,772*). Analysis of 26 major brands of margarines, representing over 90% of U.S. produced margarine, showed the polyunsaturated acid content varied from less than 10% to more than 40% (Rice, et al., *J. Am. Dietet. Assoc. 41,* 319). Comparison of Japanese bakery margarine and household margarine showed the bakery margarine discolored considerably faster than the household margarine, but oxidation took place at about the same rate in both kinds and occurred in the interior of samples at about the same rate as at the surface (Nakazawa, et al., *J. Oil Chem. Soc. Japan 11* (4), 195).

Several cocoa butter substitutes were described: Low temperature crystallization of palm oil from acetone to remove a low melting fraction yielded 40% of a fat with a melting point comparable to that of cocoa butter (Tateishi, et al., *J. Oil Chem. Soc. Japan 10*(10), 592). Analysis of this fractionated oil showed linoleic acid and caprylic acid were reduced to zero; oleic acid and capric acid were decreased, and myristic acid and palmitic acid were increased in the cocoa butter-like fraction (Tateishi, et al., *Ibid.*, 662) Using methyl ethyl ketone as solvent, the yield of cocoa butter-like fat was only 15%, but this was increased to 20% when the palm oil was first selectively hydrogenated (Tateishi, et al., *J. Oil Chem. Soc. Japan 10*(12), 717). A cocoa butter substitute replacing 25–50% cocoa butter was prepared from a mixture of palm oil and palm oil fractions and shea oil butter fractions (Best, et al., *U.S. 3,012,891*). Equal quantities of tripalmitin and tristearin were reacted with glycerol in the presence of triacetin and sodium methoxide to obtain a random mixture of 1,3-diglycerides which could be acylated with oleoyl chloride to form a cocoa butter substitute (Dutton and Scholfield, *U.S. 2,012,890*). The cost of producing a cocoa butter-like fat from hydrogenated cottonseed oil and triolein was estimated at 36.9¢ per lb if annual production was at least 8.4 million lb (Decossas, et al., *JAOCS 39,* 146).

Graininess in lard was eliminated by interesterification to reduce 2-palmitoyloleoylstearin to its random proportion (Lutton, et al., *JAOCS 39,* 233). Fluid shortenings were patented consisting of an edible oil base containing 3–4% of a mixture of finely divided saturated mono-, di- and trigylcerides in stable suspension (Schmidt, *U.S. 3,047,402*), or 2–20% of a mixture of 1,2- and 1,3-diglycerides in which the 1-position was esterified with a monohydroxy aliphatic monocarboxylic acid of 2–7 carbon atoms (Radlove, *U.S. 3,029,147*). Controlled crystallization of shortening to bring about an improved equilibrium between inter- and intracrystal composition was achieved by spraying a liquefied solid shortening into a crystallizing atmosphere and adjusting the temperature to 60–70F to cause formation of plastic particles (Wiedermann, *U.S. 3,006,770*).

A mixture of concurrently solidified fatty acid monoesters of glycerol and 1,2-propanediol was patented as an additive to be included in baking mixes prior to baking (Kuhrt and Broxholm, *U.S. 3,034,897*). Preparation of the additive was described in a separate patent (Kuhrt and Broxholm, *U.S. 3,034,898*). To prepare fatty fillings for baked products, a melted solid fat was mixed with a pastry filling containing flour, sugar, and flavoring and the resulting mixture cooled below the solidification point of the fat. The resulting supercooled liquid was then coated onto pastry and further cooled until it set (Pentzlin, *U.S. 3,056,678*). A dry, waxy culinary mix was prepared by mixing sugar, flour and a shortening containing up to 20% by weight of an ester of a monomeric polyhydric alcohol and a saturated fatty acid (Bedenk, *U.S. 3,037,864*). A procedure was described for separating polyoxyethylene monostearate from bakery products and determining it semiquantitatively by paper chromatography (Veitch and Jones, *Cereal Chem. 39*(3), 220). A unified hypothesis for the role of lipids in oxidation of doughs included evidence that peroxides are formed in dough during mixing in air or oxygen (Tsen and Hlynka, *Cereal Chem. 39*(3), 209). Mono- and diglycerides and an aqueous alcohol-soluble prolamine were obtained to form a nutritive food glaze. (Alikonis, *U.S. 3,302,422*).

Fat derivatives for use as foods were reviewed in a 1961 Short Course lecture (Feuge, *JAOCS 39,* 521). A review of the markets for oil seeds noted that, for edible purposes, the vegetable oils produced from seeds are interchangeable—and therefore competitive—with other vegetable oils, marine oils and animal fats (Breslin, *Tropical Science 4,* 87). Palatable oil and feed meal were prepared from mustard seed by enzymatic decomposition of the pungent factor, the thioglucoside sinigrin (Mustakas et al., *JAOCS 39,* 372). A process was patented for removing gossypol from cottonseed oil by treating the oil with an aqueous solution of borax (Seshadri and Chander, *U.S. 3,043,856*).

In a search for a hydrogenation catalyst of high selectivity and low isomerizing characteristics, required for the manufacture of an improved salad oil from soybean oil, platinum catalysts were found to have the lowest isomerizing characteristics but selectivity was also low (Johnston et al., *JAOCS 39,* 273). Evaluations of the keeping qualities of edible oils by accelerated oxidation tests and by organoleptic ratings were found to be in better agreement if the oils were treated with aluminum chloride before adding antioxidants (Paul and Roylance, *JAOCS 39,* 163). Another procedure which was found to be effective for removing components antagonistic to antioxidants comprised passing the oil in petroleum ether through a column of activated alumina (Crossley et al., *JAOCS 39,* 165).

Patents were issued for a salad oil consisting of mixed triglycerides of combined high molecular weight and low molecular weight fatty acids, reported to have outstanding oxidative stability (Baur, *U.S. 3,027,259*); for an all-purpose culinary oil to which was added up to 5% of erucic acid to improve the smoke point and the cloud point (Baur, *U.S. 3,047,401*); and for the use of hydroxy aromatic antioxidants to stabilize carotene added to popping oil for preparation of yellow popped corn (Borenstein, *U.S. 3,039,877*).

A shape-retaining peanut butter spread was prepared by including in the spread up to 60% of an edible oil composition containing 0.5–10% of cotton stearine and 2.5–20% of higher fatty acid monoglycerides (Ferguson, *U.S. 3,044,883*). Up to 40% by weight of fat was included in a table syrup composition consisting of a flavored oil-in-water emulsion stabilized by addition of water-dispersible proteins, gum arabic or an edible algin derivative (Pader, *U.S. 3,057,734*). Fatty acid glycerides in which at least one hydroxyl of the glycerine was esterified with lactic acid were included in a hard butter composition suitable for coatings (Babayan and Comes, *U.S. 3,051,577*). A flavoring composition having the flavor and odor of blue cheese was described as consisting of acetone, 2-pentanone, 2-heptanone, 2-octanone, 2-nonanone, 2-undecanone, ethanol and acetaldehyde dissolved in stabilized cottonseed oil and a second concentrate containing butyric acid, caproic acid, caprylic acid and capric acid in cottonseed oil, the two concentrates to be added to a product in amounts not exceeding 0.9 parts per 100 and 0.45 parts per hundred, respectively, to produce the desired flavor and odor (Bavisotto, *U.S. 3,034,902*).

Mixtures of saturated fatty acids having from 14–18 carbon atoms and having a melting point of at least 45C were patented as chewable coatings for pharmaceutical iron preparations (Stoyle et al., *U.S. 3,035,985*), and for vitamin B preparations (Stoyle et al., *U.S. 3,037,911*). Linseed oil was included, with diethyl ether and camphor gum, in an ophthalmic composition (Anderson, *U.S. 3,035,971*). An aluminum salt of a higher fatty acid having at least 12 carbon atoms was patented as a coating for antibiotic particles to be administered non-orally in a suspension of pharmaceutical oil (Jacobsen, *U.S. 3,016,330*). Water vapor porosity of a fat component in a cosmetic product was incraesed by combining the fat with up to 10% of an alkyl branched aliphatic compound (Weitzel, *U.S. 3,035,987*). Neomycin salts of higher fatty acids were patented (Dale, *U.S. 3,013,007*). One procedure specified dissolving approximately equivalent quantities of neomycin base and a higher fatty acid in a lower alkanol to form a reaction mixture having a final pH between 6.5 and 7.5 (van de Griendt, *U.S. 3,022,286*). Fatty acid salts of lysine, arginine and orinthine were also described (Ginger and Kartinos, *U.S. 3,055,923*).

EMULSIFIERS

A formulation for the oil phase of a stable oil-in-water emulsion included a water-immiscible ester such as diisobutyl adipate or ethyl hexyl diphenyl phosphate, 2,6-di-tert.-butyl-4-methyl phenol and a fatty acid ester of polyoxyethylene sorbitan (Ferrante, *U.S. 3,052,562*). An emulsifying system for a fat emulsion for intravenous injection of dogs contained 15% cottonseed oil, 1.2% polyethylene glycol monoplamitate, 0.3% tartaric acid ester of monoglycerides, and 0.3% polyoxyethylene-polyoxypropylene in isotonic dextrose solution

(Singleton et al., *JAOCS 39, 260*). Procedures were given for the preparation of the polyethyleneglycol monopalmitate and the tartaric acid ester of a monoglyceride. To improve emulsions of water and fat suitable for frying, there was included in them a surface-active agent composed of polyfunctional molecules containing carbon, hydrogen, oxygen, and nitrogen in which the ratio of carbon atoms to carboxyl groups or anhydride groups was at least 40:1 when other hydrophilic groups were absent and at least 55:1 when such groups were present (Becker and Wieske, *U.S. 3,015,566*). A plasticizer for a highly unsaturated edible vegetable oil included sodium lauryl sulfate, coconut oil, and a fatty acid monoester of a polyhydric alcohol (Farbak et al., *U.S. 3,021,221*). The product obtained by reaction between an aliphatic hydroxy polycarboxylic acid, an aliphatic dihydric alcohol, and monoglycerides was patented as an additive to improve creaming properties in shortening (Kidger, *U.S. 3,042,530*). Stabilization against separation of solids in refrigerated dairy products was obtained by adding a fluid emulsifier containing propylene glycol and a mixed partial glyceride of saturated and unsaturated higher fatty acids in which there was 40–70% of monoester and at least 87% of the acyl radicals were unsaturated (Knightly, *U.S. 3,017,276*). Mayonnaise of outstanding low temperature stability included a liquidous fat consisting of mixed triglycerides of low molecular weight and high molecular weight fatty acids. (Baur, *U.S. 3,027,260*). The iodine value of the oil ranged from 55 to more than 80, depending upon the amount of low molecular weight fatty acids in the mixed triglycerides.

ESTERS, ACIDS, ALCOHOLS AND OTHER FAT DERIVATIVES

A study of the formation of monoglycerides by direct esterification showed an excess of glycol promotes formation of the monoglyceride, but maximum yield at equilibrium is only 55–60% (Choudhury, *JAOCS 39*, 345). Alkaline catalyst depressed formation of diglycerides, and increased the initial rate of reaction without affecting the time required to reach equilibrium. Stoichiometric transesterification of glycerides to obtain methyl esters for gas chromatography was accomplished by conducting the transesterification in a sealed vial with methyl alcohol and sodium methylate (deFrancesco and Maglitto, *Revista Ital. Sost. Grasse, 5*, 245). The method failed when free acidity of the glycerides was above 0.5%, but it was found that complete transesterification could be obtained in this case by using methyl alcohol and a dehydrating agent such as concentrated sulfuric acid or zinc plus zinc chloride. A Twitchell-type reagent was used with at least 5 moles excess of methyl alcohol to esterify soapstocks in 10–20 min at 115–125C under pressure sufficient to keep the methyl alcohol in the liquid state (Eaves and Spadaro, *U.S. 3,010,977*). In a method for esterifying soapstocks for gas chromatography, the soapstocks were first treated with 6% sulfuric acid, then heated with an equal volume of methyl alcohol containing 120 g BF_3 per liter (Beck et al., *JAOCS 39*, 53). Cholesterol esters of long chain saturated and unsaturated fatty acids were prepared by ester interchange starting with cholesterol acetate and the methyl esters of the fatty acids and using sodium ethylate as catalyst (Mahadevan and Lundberg, *J. Lipid Research 3*, 106).

Ester acids having an iodine number below 3 and an epoxy oxygen content below 1% were prepared by reacting a long chain fatty acid with hydrogen peroxide in the presence of acetic acid and an esterification catalyst such as sulfuric acid or sulfonic acid at a temperature of 70–85C (Pierarski and Reinecke, *U.S. 3,031,480*). Glycidyl esters were prepared by reacting aqueous alkali metal salts of carboxylic acids, including the long chain fatty acids, with epichlorohydrin in the presence of a quaternary ammonium halide (Maerker and Port, *U.S. 3,053,855*). An epoxidized diacetoglyceride of lard or tallow fatty acids was patented as a plasticizer-stabilizer for polyvinylchloride (Swern and Knight, *U.S. 3,049,504*). Fatty acid esters of polyvinyl alcohol were reviewed (Rheineck, et al., *JAOCS 39*, 450).

Countercurrent distribution between iso-octane and 0.2 M silver nitrate in 90% methanol was used to separate methyl oleate from methyl elaidate, to separate selenium isomerized methyl linoleate into its geometric isomers, and to separate alkali conjugated methyl linoleate into 4 fractions (Dutton, et al., *Chem. & Ind.* (*Lond.*) *1961*, 1874). Glycerol esters were converted to derivatives suitable for gas chromatography by hydrogenolysis with lithium aluminum hydride followed by direct acetylation of the lithium aluminum alcoholates with acetic anhydride (Horrocks and Cornwell, *J. Lipid Research 3*, 165). Hydrogenation of randomly rearranged soybean oil and subsequent determination of the composition of the various fatty acids occupying the various positions of the resulting triglyceride molecules showed that the position of an unsaturated fatty acid radical in the glyceride molecule does not affect its hydrogenation rate (Mattson and Volpenhein, *JAOCS 39*, 307).

Differential thermal analysis was applied to the study of ternary mixtures of 1-monostearin, 1,3-distearin and tristearin (Perron et al., *Rev. Franc. Corps Gras 9*, 341). Fatty acid esters separated by thin layer chromatography were extracted from the silica gel and converted to iron hydroxamic esters for colorimetric determination of the amounts present (Vioque and Holman, *JAOCS 39*, 63). It was shown that IR absorption in the 3.3 and 3.75 μ regions can be used to establish the average chain length of very small amounts of saturated fatty acid esters (Keeney, *JAOCS 39*, 304). Determination of the mutton tallow content of mixed beef and mutton tallow samples was based on the determination of conjugated-*trans* diene acids and mono-*cis* unsaturated acids (Szonyi et al., *JAOCS 39*, 185).

Glycerides having an antioxidant activity similar to that of propyl gallate or butylated hydroxytoluene were prepared by condensing 1,2-0-propylidene glycerol, monopalmitin and glycerol, respectively, with dicarbomethoxycaffeoyl chloride and subsequently removing the protecting carbomethoxy groups (King, *Chem. & Ind.* (*Lond.*) *1962*, 1468). Brominated oils were treated with an epoxidized fatty acid ester and then deodorized to remove objectionable flavor and odor while retaining the desirable color of the oils (Becktel et al., *U.S. 3,008,833*). Fumaric acid was mixed with a partial ester of a long chain fatty acid and a polyoxyethylene derivative of a hexitol anhydride to improve solubility (Raffensperger and Takashima, *U.S. 3,009,810*).

Kinetic studies of the acetolysis of glycerides showed that water is an essential component of the reaction mixture and the temperature coefficient of the reaction velocity is 1.6 per 10C in the range from 77–118C (Meade and Walder, *JAOCS 39*, 1). 70% yields of triacetin and 80% yields of fatty acids were recovered from the acetolysis reactions (Meade and Walder, *JAOCS 39*, 4). Water soluble alkyl-substituted acetic acid was isolated from aqueous hydrolysis solutions by precipitating the insoluble aluminum soaps, separating these by filtration and then freeing the acid by decomposing the Al soaps using cold conc phosphoric acid (Bulloff, *U.S. 3,017,434*).

Linoleate of 95% purity and linolenate of 90% purity were obtained by reacting methyl esters with mercuric acetate in methanol to form the acetoxymercurimethoxy derivatives which were decomposed with gaseous HCl after separation of the adducts (Stearns, et al., *JAOCS 39*, 61). The mercuric acetate adducts of methyl oleate and methyl linoleate could be separated readily from methyl palmitate by chromatography on alumina, but the linolenate adduct could not be separated completely from the linoleate adduct by this method (White and Quackenbush, *JAOCS 39*, 511). Separation of essentially pure linolenate (99%) was accomplished by submitting an ether solution of the mercuric acetate adducts of linseed oil methyl esters to continuous liquid-liquid extraction with 10% methanol in water (White and Quackenbush, *JAOCS 39*, 517). An exhaustive study of the linoleic acid separated by low temperature crystallization of fatty acids from acetone and petroleum ether showed the presence of 5% of dienes with the first double bond at C-8 and the second at C-12 or C-13, and small amounts of non-conjugatable 9,15-*cis,cis*-dienes. Fatty acids from commercial corn oil, cottonseed oil and safflower oil were studied (Sreenivasan, et al., *JAOCS 39*, 255).

Carbonation was used to facilitate the fractionation of tallow fatty acids by precipitation of the soaps of the saturated acids (Munns et al., *JAOCS 39*, 189). A further study of the carbonation procedure for separating fatty acid mixtures into high and low iodine value fractions showed that the CO_2 can be replaced by mineral acids or by the fatty acids themselves. Electrolyte content of the completely saponified solution was found to be critical (Munns et al., *JAOCS 39*, 192). Using the carbonated or acidified solvent procedure to fractionate tall oil fatty acids, it was found that practical filtration rates were obtained only with methanol or acetone as the solvent (Meade, *JAOCS 39*, 235). Erucic acid of 94–98% purity was obtained in 2 low temperature crystallizations of technical grade commercial acid from an acetone-water system (Hagemann et al., *JAOCS 39*, 196).

Influence of the carboxyl group during hydrogenation of epoxy acids was shown by identification of both 6- and 7-hydroxy acids as products of the hydrogenation of *cis*-6,7-epoxyoctadecanoic acid, while hydrogenation of 9,10-epoxyoctadecanoic acid yielded only the 10-hydroxy compound (Fore and Bickford, *J. Org. Chem. 26*, 2104). Phase diagrams for mixtures of 9- and 10-oxo-octadecanoic acids and 9- and 10-hydroxy octadecanoic acids were used to identify these compounds in reaction mixtures and the results were interpreted as invalidating evidence for preferential reactivity at the 9- or 10- positions in C-18 fatty acid molecules (Cochrane and Harwood, *J. Org. Chem. 26,* 1278). Methyl linoleate hydroperoxide prepared by oxidation of the ester in petroleum ether was shown to be a mixture of *cis-trans* and *trans-trans* conjugated dienes (Banks et al., *J. Sci. Food Agr. 12,* 724). Oxidation of synthetic glycerides at 200C in air was shown to lead to long chain carbonyls in the early stages of the oxidation. Prolonged oxidation produced saturated aldehydes ranging in chain length from C-1 to C-16, methyl ketones and other ketones and C-4 to C-16 fatty acids. Dicarboxylic acids were formed from unsaturated fatty acids (Endres et al., *JAOCS 39,* 159). Alpha-keto acids were converted quantitatively to substituted quinoxalines by reaction with o-phenylenediamine in a procedure devised for fluorometric analysis of submicrogram quantities of the alpha-keto acids (Spikner and Towne, *Anal. Chem. 34,* 1468).

Reviews of fatty acid chemistry included: 1962 Short Course review of fat chemistry (Harwood, *JAOCS 39,* 459); 1962 Short Course review of dimer acids (Cowan, *JAOCS 39,* 534); 1962 Short Course review of liquid C-18 saturated monocarboxylic acids (Friedrich and Beal, *JAOCS 39,* 528); some new aspects of fatty acid chemistry (Swern et al., *Chem. & Ind.* (Lond.) *1962,* 1304); and industrial applications of cottonseed and soybean soapstocks (Blasi, *Lipidos 21,* 108).

Anhydrous ammonium soaps were prepared by heating fatty acids with ammonium carbonate or bicarbonate (Reiling, *U.S. 3,053,867*). Aluminum soaps were prepared by a double decomposition reaction between a basic aluminum salt of an inorganic acid and an alkali salt of a fatty acid in aqueous solution (Tanabe, *U.S. 3,056,819*). Metal soaps of the wool wax fatty acids were prepared and their solubility determined (Noble et al., *JAOCS 39,* 31). Evaluation of these latter soaps as plasticizers for PVC showed the soaps of the whole acid fraction of the wool wax were more effective than the soaps of either the isolated hydroxy fractions or the non-hydroxy fraction (Eisner et al., *JAOCS 39,* 181). Polyvalent metal soaps of C-6 to C-18 fatty acids and a synthetic resin binder were suspended in an organic solvent to form a paper coating which would become transparent on application of pressure (Dalton, *U.S. 3,009,890*). A waterproof coating for granules of urea or soluble nitrate included a thin film of metal resinate overlaid with a thin film of a gelled hydrocarbon lubricating oil in which the gelling agent was a metal salt of a fatty acid or a dimer of the fatty acid (Young, *U.S. 3,014,783*).

Formation of alcohols by Bouveault-Blanc reduction of the methyl esters of oleic, erucic, linoleic, and linolenic acids was shown to have little effect on dienoic and trienoic unsaturation. More conjugation was found in the product alcohols when trienoic unsaturation was present in the original esters or when the reaction was conducted in the presence of an excess of the reducing alcohol (Fukushima, *J. Oil Chem. Soc.* (*Japan*) *10,* 635). Similar reduction of methyl-*alpha*-eleostearate led to formation of a mixture of dienoic and trienoic alcohols. Separation and analysis of these two types of alcohol showed the dienoic alcohol was a mixture of 10,12-octadecadiene-1-ol and 11-octadecene-1-ol, and the trienoic alcohol was a mixture of 9,12,15-, 9,12,14-, and 9,11,13-octadecatriene-1-ol (*Ibid.,* 701). An alcohol, RCH_2OH, and an ester, RCH_2OOCCH_2R, were formed by heating an acid RCH_2COOH to 100C in the presence of oxygen and a cobalt salt of a fatty acid (Blair and Melchiore, *U.S. 3,013,038*). The product obtained by distilling glycerine with ammonium phosphate or phosphoric acid was shown to be identical to that obtained by treating cis-2,6-bis(iodomethyl) dioxane with silver oxide (Summerbell and Poklaski, *JAOCS 39,* 306). Aqueous solutions of higher fatty alcohols and of their esters were prepared by including polyoxyethylene sorbitan monostearate as a solubilizing agent (Hirsch, *U.S. 3,052,607*). Solidification points of binary mixtures of C_{12} to C_{18} alcohols were studied over a broad range of concentrations (Paquot et al., *Oleagineux 6,* 555). Fatty alcohols were separated chromatographically as their acetic esters (Fukushima, *J. Oil Chem. Soc. Japan 11,* 128) or as malonic half esters (Capella et al., *Revista Ital. Sost. Grasse 1,* 2).

The 1962 Short Course included reviews of sulfur derivatives of the long chain fatty acids (Sasin, *JAOCS 39,* 488) and of *alpha*-sulfo fatty acids (Stirton, *JAOCS 39,* 490). Sodium alkyl *alpha*-sulfopelargonates were prepared using alcohols of 2-12 carbons and from a study of their properties it was concluded steric hindrance of the *alpha*-sulfo group inhibits hydrolysis of the esters (Stirton et al., *JAOCS 39,* 55). Methods were described for preparation of dialkyl esters of *alpha*-sulfopalmitic and *alpha*-sulfo stearic acids by reaction of the silver salt of the sulfo acid with an alkyl iodide, and for preparation of the corresponding diamides by reacting the acid chlorides with ammonia, ethanolamine or isopropanolamine (Weil et al., *JAOCS 39,* 168). The dialkyl esters were also prepared by adding ethylene or propylene to the *alpha*-sulfo fatty acids in the presence of BF_3 or its etherate (McBride and Miller, *JAOCS 39,* 105). A study of the sodium salts of the alkyl esters of *alpha*-sulfo fatty acids showed that moving the hydrophilic portion from the center of the molecule toward either end decreased the wetting efficiency and cmc, increased surface and interfacial tension and calcium ion stability, and improved the lime soap dispersing properties (Stirtin et al., *JAOCS 39,* 128). A mixture of esters of sulfosuccinic acid was described which included higher fatty acid glycerides, higher fatty acid glycols, higher fatty alcohols, and mixed diesters incorporating aliphatic alcohols (Fair and Ballou, *U.S. 3,043,706*). Comparison of the thioethers formed by addition of thiols to oleic acid in the presence of methanesulfonic acid with the substituted stearic acids obtained by adding phenolic compounds to oleic acid showed that the thioethers do not undergo the rearrangements which are characteristic of the phenol derivatives (Eisner et al., *JAOCS 39,* 290).

Petroselenonitrile was prepared by distilling the ammonolysis products of petroselenic acid of parsley seed oil over P_2O_5 in the presence of an antioxidant to control polymerization. The resulting nitrile was converted to primary petroselenylamine in good yields by reduction with metallic sodium and alcohol in toluene (Placek and Dollear, *JAOCS 39,* 347). Dodecylamine and 6-aminohexanoic acid were prepared from parsley seed oil by ozonolysis and reduction of the oximes of the cleavage products (Holmes et al., *JAOCS 39,* 411). Lauric diethanolamide was prepared from methyl laurate and diethanolamine by a continuous high temperature process in the presence of an alkaline catalyst (Monick, *JAOCS 39,* 213). Mixed amides, nitriles, and amines of hydrogenated cyclized acids derived from C-18 triene-containing fatty acid sources were prepared and characterized. The resulting amides were found to be more compatible with synthetic resins and to have greater solubility in organic solvents than the amides of normal fatty acids. Morpholides, diethanolamides, ethenoxylated amides, and quaternary amines were also prepared from these hydrogenated cyclic acids and their surface active properties evaluated (DeJarlais and Teeter, *JAOCS 39,* 421).

Dark colored raw fatty acid amides were purified by heating them with oxygen yielding agents such as hydrogen peroxide, perborates, percarbonates, performic acid, or peracetic acid (Dieckelmann, *U.S. 3,006,934*). Freezing point diagrams for the ternary reciprocal salt systems involving morpholine and 2,2′-dipyridylamine salts of stearic and palmitic acids were used to predict the crystallization temperature and yield of each of the four possible salts (Mod et al., *JAOCS 39,* 444). Isothiocyanates were prepared by enzymatic hydrolysis of *Lesquerella* seed meals (Daxenbichler et al., *JAOCS 39,* 244). Aliphatic amines were used in a treatment to reduce gossypol in cottonseed meal (King et al., *JAOCS 39,* 58). Nitrogen derivatives of the long chain fatty acids were reviewed in a 1962 Short Course (Reck, *JAOCS 39,* 461). A mixture of soap and fatty acyl aminomethane sulfonate was prepared by reacting a fatty acid amide with sodium or potassium formaldehyde bisulfite in the presence of free fatty acid and alkali metal soap (Alsbury et al., *U.S. 3,047,509*).

Distearoyl-L-*alpha*-glycerylphophoryl-L-threonine, a member cf a recently discovered class of natural phospholipids containing threonine, was synthesized (Baer and Eckstein, *J. Biol. Chem. 237,* 1449). A granular oil-free phosphatide product was obtanied by treating phosphatide with an aqueous solution of reducing saccharide (Davis and Fello, *U.S. 3,012,888*). Salt-free fatty acyl taurates were prepared by reacting a fatty acid with the dry sodium salt of methyl taurine (Burnette and Chiddix, *JAOCS 39,* 477). Hydrated soybean lecithin of high purity was separated from hexane miscella by adding water into the stripper at the end of the stripping process and also by charging water into the settling tank ahead of the finished oil (Yoshitomi et al., *J. Oil Chem. Soc. Japan 11,* 123). Sources, uses, and properties of the vegetable phosphatides were reviewed (Loew, *Revista del Centro Quimicos Industriales 1961,* 34); and the 1962 Short Course included a review of phosphorus derivatives of the fatty acids (Sasin, *JAOCS 39,* 448).

Esters of amylose and the even numbered saturated fatty acids from acetic acid through stearic acid, and oleic and cottonseed oil fatty acids were prepared and evaluated as dip-coating materials (Gros and Feuge, *JAOCS 39,* 19). Sucrose esters were prepared by interesterification with fatty acid lower alkyl esters and glycerides in the presence of a basic catalyst (D'Amato, *U.S. 3,054,789*). Another for preparing sucrose monoesters comprised mixing an equimolecular amount of sucrose and a monovalent aliphatic radical-containing succinic anhydride in a solvent consisting of dialkylacylamides or dialkylsulfoxides and heating at temperatures up to 120C in the presence of a tertiary amine catalyst (Gaertner, *U.S. 3,053,830*). Sucrose monopalmitate was separated from mixtures with di- and tripalmitates by chromatography on a column of silica gel, eluting with 25% ethanol in benzene (Mima and Kitamori, *JAOCS 39,* 546). A procedure was patented for preparation of fatty acid esters of aldose semicarbazones and aldose thioureides (Knoevenagel, *U.S. 3,027,364*).

Ethylene was shown to react quantitatively with *trans, trans*-9,11-octadecadienoic acid to form a C-20 monocarboxylic acid containing one double bond and having an infrared absorption characteristic of the cyclohexene ring (Friedrich et al., *JAOCS 39,* 420). The Diels-Alder adduct of tetracyanoethylene and *trans, trans*-9,11-octadecadienoic acid was shown to form spontaneously when the reactants were dissolved in tetrahydrofuran at room temperature (Miller and Cowan, *JAOCS 39,* 380). Results of the thermal polymerization of four different isomers of methyl linoleate were reported to confirm that isomerization of the double bond is the initial reaction and the resulting conjugated *trans, trans*-compounds then undergo Diels-Alder addition to non-conjugated *trans*-isomers (Nagano and Tanaka, *J. Oil Chem. Soc. Japan 11,* 119). Heating unsaturated fatty acids containing more than four non-conjugated double bonds changed their structure to the conjugated form with formation of increasing amounts of cyclic compounds as the temperature was raised. At 200C the products were linear and had the *cis, cis, trans*-conjugated triene form. At 215C nearly equal amounts of cyclic and linear compounds were formed. At 250C the conjugated diene product had an absorption maximum at 236 mμ and consisted mainly of dimeric compounds having a cyclohexene ring structure (Miyakawa and Nomizu, *J. Oil Chem. Soc. Japan 10,* 724).

Formation of urea adducts was patented as a procedure for separation of the non-cyclized fatty acids from cyclic monomers formed during heating of linseed oil or methyl linolenate with an excess of KOH in an ethylene glycol medium for at least 7 hr at 200–250C (Scholfield et al., *U.S. 3,041,360*). Olefinic esters of fumaric acid were found to form urea inclusion compounds more readily than did the corresponding esters of maleic acid (Randell et al., *J. Org. Chem. 26,* 2960). Catalytic dehydration of methyl ricinoleate during vacuum distillation was shown to result in the formation of 68.9 mole % of the non-conjugated C-18 dienoic acid, 15.6 mole % of *cis, trans-*, 8.3 mole % of *cis, cis-*, and 5.9 mole % of *trans, trans*-conjugated octadecadienoates (Body and Shorland, *Chem. & Ind. (London) 1961,* 1665). Improved yields of hydroxyphenyl stearates were obtained by using methanesulfonic acid (or polyphosphoric acid) as the catalyst in the addition of phenol to oleic acid (Ault and Eisner, *JAOCS 39,* 132). Ricinoleic acid was converted by heating its alkali metal salt in the presence of an alkali metal hydroxide and an alloy consisting of one element from the fifth period of the Periodic Table and one metal from the sixth period in proportions such that the alloy melted below the reaction temperature (Stein and Hennig, *U.S. 3,031,482*).

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

Alkyl phophates, quaternary ammonium type cationics, and betaine type amphoterics were found to be effective antistatic agents on polypropylene fibers even after 90 days of storage (Onoda and Saigusa, *Yukagaku 11,* 69). Hard waxes were formed by reacting fatty acids of more than 18 carbon atoms with a di- or trihydroxy alcohol and a compound such as 2,2-(4,4′-dihydroxy-diphenyl)-propane, resorcinol, pyrocatechol, hydroquinone, 4,4′-diamino-dicyclohexylmethane, benzidine, p-aminophenol or piperazine (Kaupp et al., *U.S. 3,053,681*). Fatty acids and alcohols prepared from *Limnanthes douglasii* seed oil glycerides were converted to a liquid wax by esterification. The liquid wax and the solid wax obtained by hydrogenation were found to be comparable to waxes prepared from jojoba seed oil (Miwa and Wolff, *JAOCS 39,* 320). A waxy composition suitable for impregnating cellulosic materials was prepared by esterifying tall oil acids containing rosin acids and unsaponifiables with long chain fatty alcohols (Johansson, *U.S. 3,006,778*).

An oil base drilling fluid included as one component the reaction product obtained by reacting an alkaline earth metal base with the undistilled bottoms fractions from vacuum distillation of split animal or vegetable oils (Fischer, *U.S. 3,017,349*). The alkali metal soaps of undistilled bottoms from this same source were used as an emulsifying agent in a drilling fluid composition (Fischer, *U.S. 3,017,350*). Lubricating properties of drilling fluids were enhanced by incorporating in the mixture calcium ions, a non-ionic surface active agent and long chain fatty acids, sulfurized fatty acids and alkali metal soaps of fatty acids or sulfurized fatty acids (Rosenberg and Schaub, *U.S. 3,048,538*). A liquid gelling agent for gasoline comprised an aluminum salt of a single fatty acid containing from 6–12 carbon atoms suspended in a solvent (Carson et al., *U.S. 3,018,252*).

Mustard seed meal suitable for animal feed was prepared by extracting the sinigrin or decomposition products of sinigrin with water at 80–85C (Goering, *U.S. 3,044,876*). Feed pellets having a fat content greater than 4% were prepared by pelletizing the feed and then treating, while hot, with liquefied fat (Guidarelli, *U.S. 3,014,800*).

A study of the by-products of olive oil extraction showed *Torulopis utilis* yeast can be grown in the waste liquor (Fiestas, *Grasas y Aceites 12,* 57), and the proteins formed by the yeast contain all of the essential amino acids indicating the yeast would make a valuable animal feed (Fernandez and Fiestas, *Grasas y Aceites 12,* 161). Surfactants were used to destroy lipoproteic membranes in olive oil foots permitting additional recovery of oil from the oil-in-oil emulsion (Martinez et al., *Grasas y Aceites 12,* 118). Several anionic and nonionic detergents were found to be effective in suppressing the emulsification of oil droplets by olive oil extraction liquors (*Ibid.,* 166). Stability of these emulsions was shown to depend upon the amounts of protein and ionic lipids at the interfacial surface (Martinez et al., *Grasas y Aceites 12,* 213). Slight increases in oil yield were noted when pastes from unripe olives were treated with N-(coconut fatty acid)-aminobutyric acid. This was the only effective surfactant in a total of 66 different ones tested for their ability to increase the yield of oil extracted from ground olive oil paste (Martinez et al., *Grasas y Aceites 13,* 61).

The influence of potassium soaps of disproportionated rosins on the emulsion polymerization of styrene and butadiene was studied. The yield of polymer was not affected by the amount of unsaponifiable matter or free rosin acid in the soap except when an unusually large amount of free acid was present (Wakabayashi et al., *Yukagaku 11,* 65).

FATTY MATERIALS IN LUBRICATION, METAL-WORKING AND TEXTILE OILING

Lithium soaps of C-16 to C-22 fatty acids and calcium soaps of C-12 to C-14 fatty acids were combined as a thickener in a grease composition (Franklin and Gebhart, *U.S. 3,042,615*). Monostearoyl tetraethylene pentamine silicate was added to alkali metal and alkaline earth metal fatty acid soaps to form a lubricating oil thickener (Hallowell, *U.S. 3,020,233*). Alkali metal soaps of aliphatic branched open chain carboxylic acids were patented as thickeners for greases (Sandy and Werntz, *U.S. 3,041,154*). Another thickener consisted of the lithium soaps of ethyl hexoic acid and of long chain fatty acids mixed with an ester of 12-hydroxystearic acid (Hallowell, *U.S. 3,053,767*).

Other thickeners for lubricating oil included: Indigo plus a mixture of lithium and sodium soaps of C-12 to C-16 fatty acids, lithium and sodium salts of short chain fatty acids, and lithium and sodium pectates (Roach, *U.S. 3,009,879*); a metal salt of a fatty acid and a metal soap of an alkyl mercapto hydroxy fatty acid (Morway and Bartlett, *U.S. 3,036,970*); the metal soap of an epoxy fatty acid (Eckert, *U.S. 3,009,878*); a mixture of bentonite and an oil-soluble, water-insoluble amino amide formed by heating a fatty acid with approximately two equivalents of polyamine (Peterson, *U.S. 3,050,463*).

An additive for a carboxylic acid ester lubricating oil was described as an acid partial ester formed by reacting a C-4 to C-12 alkanedioic acid with a glycol (Matuszak et al., *U.S. 3,029,204*). A lubricating composition was prepared by mixing together sodium benzoate, sulfonated castor oil, and a liquefied triglyceride of a fatty acid, adding these to mineral oil, heating to effect solution, and then adding phosphoric acid (Shamaienger, *U.S. 3,028,335*).

A coating composition for the formation of metal working films consisted of a non-congealing aqueous solution of an amine soap and an alkali metal soap which were formed from fatty acids having a titer below 30C (Fucinari and King, *U.S.*

3,023,163). A stable calcium carbonate suspension for use in metal working contained an organic emulsifying agent, a triglyceride oil or fat, a hydrocarbon oil, water, magnesium stearate, calcium carbonate and a polyacrylamide (Gaynor and Eisenhauer, *U.S. 3,024,193*).

PRODUCTS (EXCEPTING DETERGENTS)

DRYING OILS, PAINTS, RESINS, AND PLASTICIZERS

A brief review of the history of fat chemistry and of the applications of fatty products referred to protective coatings, plasticizers, stabilizers and plasticizers for polyvinyl chloride, and antistatic agents for plastics (Harwood, *JAOCS, 39,* 459).

Specific Oils

The effect of the Argentine flax crop on the Winnipeg flax market was discussed (McHale, *Ibid., 39* (4), supp. 7). Examination of the seeds of 20 varieties of flax revealed a high negative correlation between seed density and oil content. The former quantity was suggested as a criterion for selection of flax seed of high oil content (Zimmerman, *Ibid., 39,* 77). Recovery of pure linoleic or linolenic acid from safflower or linseed oil involved deaeration, hydrolysis in the absence of oxygen, isolation of the fatty acids, and partition between aqueous furfural and hexane or isooctane (Beal, *U.S. 3,052,699*). Pure methyl linolenate was obtained by extracting its mercuric acetate addition compound from ether solution with 10% methanol in water. This method could also be used to isolate more highly unsaturated acids (White and Quackenbush, *JAOCS, 39,* 517). The fatty acid composition of a number of domestic and foreign castor oils was determined by a method involving consecutive column and gas chromatography (Binder et al., *Ibid.,* 513). The amounts of sterols and tocopherols in the unsaponifiable matter of an *Aleurites montana* tung oil were determined (Shone, *J. Sci. Food Agr., 13,* 315). An eicosapentaenoic and a docosahexanenoic acid were isolated from cuttlefish oil by urea complexing, fractional distillation of methyl esters, and chromatography. The same acids and a docosapentaenoic acid were isolated from finback whale oil by similar methods. The positions of the double bonds in these acids were established by oxidative and reductive ozonolysis (Toyama and Takagi, *Fette, Seifen, Anstrichmittel, 64,* 134, 137).

An examination of the seed oils of 37 plant species from 18 families revealed the presence of dimorphecolic acid (9-hydroxy-*trans,trans*-10,12-octadecadienoic acid) in a second species of Dimorphotheca *(D. pluvialis)*, and also in the species *Osteospermum ecklonis.* Conjugated trienoic acids were found in the seeds of *Osteospermum spinescens* and *Calendula officinalis.* Epoxy acids and non-conjugated trienoic acids were found in some species (Earle et al., *JAOCS, 39,* 381). A method for obtaining dimorphecolic acid by solvent extraction of the seeds and saponification of the oil was patented (Smith et al., *U.S. 3,027,388*). Chemical changes occurring in dimorphotheca oil during air drying and heat bodying were followed by infrared and ultraviolet spectrograms. Comparable phenolic resin varnishes of dimorphotheca and tung oil showed only marginal differences in their performance characteristics (Rheineck and Sobol, *Offic. Dig., Federation Socs. Paint Technol., 34,* 862). The acids of the seed oil of *Thalictrum polycarpum* included *trans*-5,*cis*-9,*cis*-12-octadecatrienoic acid (35%), an unknown C_{18}-dienoic acid (18%), oleic acid, and *trans*-5-octadecenoic acid (Bagby et al., *Biochemistry, 1,* 632). The seed oil of Indian ironweed *(V. anthelmintica)* contained a high percentage of epoxyoleic acid (vernolic acid). If the hydrolytic enzyme present in the seed were inactivated, oil containing more than 50% trivernolin could be obtained (Krewson et al., *JAOCS, 39,* 334).

The Drying Process; Driers

Reactions involved in the formation and deterioration of films derived from drying oils and polymeric products were reviewed (Cowan, *Offic. Dig., Federation Socs. Paint Technol., 34,* 561). The drying and aging of films by chemical means were discussed with the objective of elucidating the role of metallic catalysts in both stages of the history of a film (Myers, *Ibid., 34,* 575). An ultrasonic method of following the changes in mechanical properties during the process of drying has been employed in a rheological study of the kinetics of the coalescence of dispersions and of autoxidative polymerization (Myers and Schultz, *Ibid., 34,* 801).

A review on fatty peroxides included a discussion of the initiation of polymerization by free radicals resulting from the decomposition of peroxides (Silbert, *JAOCS, 39,* 480). Oleic, elaidic, erucic, brassidic, linoleic, linolenic, and ricinoleic acids were oxidized in a study of the effect of structure on the oxidation of fatty acids in the presence of various alkyl isochromans as antioxidants (Rieche et al., *Fette, Seifen, Anstrichmittel, 64,* 198). Autoxidation of methyl linolenate at 37C produced a mixture of four hydroperoxyoctadecatrienoates, the structures of which were determined (Frankel et al., *J. Org. Chem., 26,* 4663). The activation energies for the oxidative polymerization, in the presence of metallic driers, of a number of alkyd resins were determined. A theoretical explanation was offered for the fact that the addition of 1,10-phenanthroline to these systems decreased the activation energies (Wheeler et al., *I&EC Product Res. and Dev., 1,* 52). Aluminum soaps of polyunsaturated fatty acids were good driers, promoting rapid and deep drying but permitting the films to retain lasting elasticity and adhesion (Marwedel, *Farbe und Lack, 68,* 212). A group of patents described a method of rapidly drying a coating vehicle or a printing ink consisting of a non-aqueous solution of a conjugated drying oil and a catalyst by irradiating a film of the material with actinic energy. The catalysts were an organic titanate or diisocyanate, a dye, an acyl chloride, and an organic peroxide plus a tertiary amine, respectively (Sites and Agruss, *U.S. 3,041,203; U.S. 3,047,422; U.S. 3,050,413; U.S. 3,051,591*).

Products Formed Via Double Bond Changes

Isomerization. Cuttlefish acids having more than four nonconjugated double bonds were isomerized to monomeric linear acids having a *cis,cis,trans* conjugated triene structure when heated at 200C. At 215C, the product was a mixture of linear and cyclic monomeric conjugated acids. At 250C, the product was polymeric acids with a conjugated diene structure (Miyakawa et al., *J. Oil Chem. Soc. Japan, 10* (5), 303; Miyakawa and Nomizu, *Ibid., 10* (12), 724). The preparation and properties of monomeric cyclic saturated C_{18}-acids formed by alkaline isomerization of linseed oil, tung oil, or linolenic acid were reviewed (Friedrich and Beal, *JAOCS, 39,* 528). The use of these materials, designated as "cyclic acids," in other products will be referred to below.

Dehydration. Vacuum distillation of methyl ricinoleate over potassium hydrogen sulfate produced 69% and 30% of nonconjugated and conjugated octadecadienoates, respectively (Body and Shorland, *Chem. & Ind.* (London), *1961,* 1665). During the dehydration of castor oil the refractive index correlated with the hydroxyl value, iodine value (I.V.), and viscosity, and could be used to determine each of these latter quantities from a calibration chart (Fauve, *Oleagineux, 17,* 703). Refluxing dimorphotheca seed oil under nitrogen in xylene with a dehydrating agent (glacial acetic acid or p-toluene sulfonic acid) produced all trans-8,10,12-octadecatrienoate (Smith et al., *U.S. 3,013,037*).

Polymerization; Diels-Alder Reactions. The chemistry, manufacture and uses of dimerized fatty acids were reviewed (Byrne, *Offic. Dig. Federation Socs. Paint Technol., 34,* 229). The preparation and properties of dimeric acids formed from two molecules of unsaturated fatty acids, and their uses in polyesters, urethane coatings and foams, alkyd resins, epoxy coatings, and polyamide resins was reviewed (Cowan, *JAOCS, 39,* 534). The formation of polymeric acids during the thermal isomerization of unsaturated cuttlefish acids has been referred to above. Investigation of the products of thermal polymerization of four different isomers of methyl linoleate supported the conclusion that the reaction proceeded by the formation of conjugated *trans,trans,* and nonconjugated *trans* isomers which then underwent Diels-Alder addition (Nagano and Tanaka, *J. Oil Chem. Soc. Japan, 11* (3), 119). Another study of the structure of methyl linoleate dimers formed by heating at 300C for varying lengths of time showed the presence of both conjugated and nonconjugated products. The results supported the hypothesis that Diels-Alder addition was not the only dimerization mechanism (Rivett and Sutton, *Chem. & Ind. (London), 1961,* 1452). A Diels-Alder adduct was formed by the addition of tetracyanoethylene to *trans,trans*-9,11-octadecadienoic acid (Miller and Cowan, *JAOCS, 39,* 380).

Oxidation. A review of fatty peroxides covered the epoxidation of double bonds by peroxy acids and ozonization of unsaturated fatty oils to aldehyde oils (Silbert, *Ibid., 39,* 480). A description of research being conducted at the Eastern Regional Research Laboratory included the preparation of peroxy acids and selective epoxidation (Swern et al., *Chem. & Ind. (London), 1962,* 1304). A process for epoxidizing unsaturated fatty esters at 90–150F with hydrogen peroxide, acetic acid, and sulfuric acid was patented (Hansen and Sedgwick, *U.S. 3,051,729*). Treatment of unsaturated fatty acids at 70–85C with hydrogen peroxide in the presence of acetic acid and an esterification catalyst such as sulfuric or a sulfonic acid resulted in epoxidation followed by cleavage of the epoxide ring and acetylation of the hydroxy groups (Piekarski and Reinecke, *U.S. 3,031,480*). Ozonization of unsaturated oils cleaves the fatty chains at the double bond and introduces an aldehyde group at this point. Preparation and properties of the resulting aldehyde-acids, their triglycerides ("aldehyde oils") and their other derivatives were reviewed (Pryde and Cowan, *JAOCS, 39,* 496). The preparation of other products by reactions of the epoxy and aldehyde groups of these oxidized fatty derivatives will be discussed below.

Products from Carboxyl Reactions

Esters with Polyhydric Alcohols. A study of the esterification of drying oil acids with polyvinyl alcohol revealed no substitute for phenol as the reaction solvent and led to no improvement in yield over previous procedures. Evaluations of the products in clear varnishes and house paints were promising (Rheineck et al., *Ibid.*, 450). Alcoholysis of a conjugated octadecatrienoic acid ester of a lower alcohol with an allyl alcohol-vinylaromatic compound copolymer produced the conjugated fatty acid ester of the copolymer (Zonsveld, *U.S. 3,013,999*). Tung oil was gasproofed without danger of gelation by alcoholysis with trimethylolethane at 565 F (Goldblatt et al., *U.S. 2,999,104*).

Alkyd Resins. The growth and present status of alkyd resins was reviewed briefly (Kraft, *JAOCS, 39,* 501). Novel alkyd resins were prepared from 1-(4-carboxyl phenyl)-1,3,3-trimethyl-6-indane-carboxylic acid and 4,4′-(2,2-butylidene) dibenzoic acid and their drying rates and chemical resistance studied (Petropoulos, *Offic. Dig. Federation Socs. Paint Technol., 34,* 992). The cyclic acids produced by the alkali cyclization of unsaturated fatty acids (preparation referred to above) were used as modifying agents in alkyds. The products were an improvement over those modified with linseed, safflower, or soybean acids, the degree of improvement being greater, the higher the purity of the cyclic acids. (Miller et al., *Ibid.*, 534). A gasproofed non-gelling tung alkyd was prepared by alcoholyzing a mixture of tung oil and tall oil fatty acids with trimethylolethane at 565F (Goldblatt et al., *U.S. 3,008,910*). A condensate of trimellitic anhydride with a 1,2-epoxide was esterified with an unsaturated fatty acid (Stephens and Van Strien, *U.S. 3,012,979*). An unsaturated triglyceride was reacted first with a glycidyl polyether of a dihydric phenol, then with an aliphatic polyhydric alcohol and a dicarboxylic acid anhydride (Masters, *U.S. 3,027,340*). A castor acid-tall fatty acid-penetaerythritol-benzoic acid-isophthalic acid alkyd was modified by heating at 275–350F with styrene in the presence of a peroxy catalyst (Bolton, *U.S. 3,054,763*).

Esters with Other Reactive Alcohols. Reaction of epichlorohydrin with an alkali metal salt of a carboxylic acid (including monomeric and polymeric fatty acids) in the presence of a quaternary ammonium halide produced the glycidyl ester of the acid (Maerker and Port, *U.S. 3,053,855*). An epoxy resin composition consisted of a mixture of a glycidyl ester of a polymeric fatty acid and a glycidyl polyether of a polyhydric phenol or alcohol (Newey, *U.S. 3,057,809*). Polymers were prepared by homopolymerization of vinyl esters of cyclic acids and hydrogenated cyclic acids (from alkali isomerization of unsaturated fatty acids), and by copolymerization of these vinyl esters with vinyl chloride (Friedrich and Beal, *JAOCS, 39,* 528).

Polyamides. A review on dimeric fatty acids covered the use of these materials in polyamides (Cowan, *Ibid.*, 534). Polyamides were prepared in the absence of oxygen from oleic acid, sebacic acid, and triethylene tetramine (Dearborn and Isaacs, *U.S. 3,050,527-8*), and from these same materials plus polymeric fatty acids (containing both dimers and trimers) (Dearborn and Isaacs, *U.S. 3,050,529*).

Products from Carbonyl Reactions

The aldehyde oils obtained (see above) by ozonization of unsaturated oils contained two or three free aldehyde groups per molecule (depending upon the number of unsaturated fatty acid residues in the original glyceride). These aldehyde oils underwent condensation reactions with diols and polyols, phenols, ureas, and diamines to produce polymers which were insoluble, infusible, but resilient solids. Specific compounds whose reactions with aldehyde oils were investigated were phenol, resorcinol, a pelargonaldehyde-resorcinol resin, urea, hydrazine, and o-phenylene diamine. Hydrolysis of the aldehyde oils yielded aldehyde acids, which served as intermediates for a new class of polymers. The pentaerythritol acetal of methyl azelaaldehydate, a diester, was treated with glycols to yield poly(ester-acetals) or with diamines to yield poly(amide-acetals). These polymers were cross-linked rapidly in the presence of certain metal oxides or salts (Pryde and Cowan, *JAOCS, 39,* 496).

Products from Epoxide Reactions

Epoxidized oils reacted with aliphatic alcohols in the presence of a boron trifluoride or stannic chloride catalyst (Findley et al., *U.S. 3,035,069*). Protective coatings were formed by the reaction of epoxidized fatty acids or esters with polycarboxylic acids (Budde, *U.S. 3,050,480*); also from the reaction of polyepoxides with aliphatic unsaturated monocarboxylic acids. The polyepoxides were epoxidized fatty esters or epoxidized polyesters of tetrahydrophthalic acid (Greenlee and Pearce, *U.S. 3,023,178*). Condensation products were formed from the reaction of an epoxidized oil with a polyamine and then with a polycarboxylic acid (including polymeric fatty acids) (Fulmer, *U.S. 3,035,000*). A material which still contained oxirane oxygen was obtained from a reaction of an epoxidized oil or epoxidized fatty ester with an oxychloride such as phosphorus oxychloride, sulfuryl chloride, chromyl chloride, thionyl chloride, or phosgene (Chatfield and Weeks, *U.S. 3,042,687*). A high molecular weight linear thermoplastic polymer of bisphenol-A and epichlorohydrin was developed. This polymer, in contradistinction to conventional epoxies, is inherently a film former (Marshall and Somerville, *Offic. Dig. Federation Socs. Paint Technol., 34,* 286).

Products from Hydroxyl Reactions

Ethers. Vinyl ethers of fatty alcohols serve as monomers in the preparation of polymers. The copolymers of conjugated linseed, conjugated soybean, and nonconjugated linseed vinyl ethers with various cyclic monomers (dihydroabietyl, cyclohexyl, 5-norbornene-2-methyl, di- and tetrahydrodicyclopentadienyl vinyl ethers, and cyclo- and methylcyclopentadiene) gave superior baked films (Dufek et al., *JAOCS, 39,* 238). Polymers which gave good baked films were also formed by heating the preformed polymer of conjugated linseed vinyl ether with monomeric styrene (Schneider et al., *Ibid.*, 241).

Urethanes. A survey of urethane coatings was made and the parameters used to formulate urethane vehicles were discussed (Patton, *Offic. Dig. Federation Socs. Paint Technol., 34,* 342). A review on the use of fatty acid derivatives in polyurethanes classified these materials on the basis of their use as protective coatings, elastomers, or foams, and described the methods of preparing each (Goldblatt, *JAOCS, 39,* 502). Modified drying oils were formed by the reaction of a hydrocarbon diisocyanate with a diol mixture containing a diol ester of an unsaturated fatty acid with a trihydric (or higher) alcohol and etiher a low molecular weight diol (Waythomas, *U.S. 3,022,327*) or a polyglycol (Schroeder and Waythomas, *U.S. 3,022,326*). A polymeric product was formed from the reaction of a polyisocyanate with a polyester prepared from (a) a triglyceride, a monoglyceride, or a fatty acid, (b) a polyhydric alcohol, and (c) 4,4′-(2,2-butyldiene)dibenzoic acid (Petropoulos, *U.S. 2,998,399*). Dimeric fatty acids, hydroxyethylated by treatment with ethylene oxide, were used as diols in the preparation of flexible urethane foams. Hydrogenated aldehyde oil, prepared by the ozonolysis of soybean oil followed by the reductive decomposition of the ozonide, was used as the polyol component in the formation of a rigid foam. The triglyceride of 9-hydroxynonanoic acid should be the predominant structure in this hydrogenated aldehyde oil (Thierfelder et al., *JAOCS, 39,* 215). Economical rigid foams were prepared from crude diphenylmethane diisocyanate and resin blends containing up to 75% of crude tall oil (Gemeinhardt et al., *I & EC Produce Res. and Dev., 1,* 92). Rigid foams were also formed from prepolymers of toluene diisocyanate with polyether polyols (or mixtures of these polyols with castor oil) by reaction with a polyol component containing castor oil, triethanolamine, and/or triisopropanolamine (Lyon et al., *JAOCS, 39,* 69). A high modulus foam was formed from a prepolymer of an aromatic diisocyanate, castor oil, a polyol monoricinoleate, and trimethylolpropane. The prepolymer was foamed and cross-linked by the addition of water and an amine catalyst (Elkin, *U.S. 3,037,947*).

Drying Oil-Resin Combinations

The following products consisting of mixtures or reaction products of drying oil derivatives with other resins were patented: a mixture of a vinyl chloride polymer and an epoxidized glyceride, which formed a protective coating when spread on a surface and heated (Rhodes and Findley, *U.S. 3,047,415*); the product formed by mixing a liquid polymer of a conjugated diolefin with a material such as a drying oil, semi-drying oil, dicyclopentadiene, and methyldicyclopentadiene, and blowing the mixture in a hydrocarbon solvent with air or oxygen at 20–150C (Mozell and Gleason, *U.S. 3,026,279*); a film-forming mixture of (a) a polymer of styrene, a monocarboxylic monomer such as methacrylic or acrylic acid, and an ester of the monocarboxylic monomer, (b) a drying fatty acid ester of a resinous epoxyhydroxypolyether, and (c) a urea- or melamine-formaldehyde-alkanol condensate (LaBarre, *U.S. 3,014,881*).

Water-Dispersible Resins

Water-dispersible coating materials frequently consist of ammonium or amine salts of resins which contain free carboxyl groups. One such material was an ammonium salt of dimeric unsaturated fatty acids or of a styrene-unsaturated fatty acid reaction product, mixed with a water-dispersible polymethyl ether of polymethylol melamine (Layman, *U.S. 3,025,251*). Another was an ammonium or amine salt of the reaction product of a drying oil, maleic anhydride, acrylic or methacrylic acid, and an unsaturated carboxyl-free monomer (Lombardi et al., *U.S. 3,030,321*). Aliphatic amine salts of alkyd resins having acid values of 25–70 were water soluble. One such alkyd contained, in addition to the usual fatty acid, polycarboxylic acid, and polyhydric alcohol, a glycidyl polyether of a dihydric phenol (Boucher and Bussell, *U.S. 3,027,341*). Another amine salt of an acidic alkyd was pigmented and mixed with an acrylic resin latex (Boucher, *U.S. 3,023,177*).

Other water-dispersible coating compositions are emulsions rather than solutions of the resinous components. Raw tung oil was emulsified with a synthetic component and pigmented to give an oil-in-water emulsion paint (Murray et al., *U.S. 3,033,808*). Another emulsion paint consisted of highly-bodied linseed oil, hydroxyethylcellulose, pigment, a naphthenate drier, and (as an emulsifying agent) the potassium salt of dimeric acids (Schwab and Teeter, *U.S. 3,047,413*). Linseed oil emulsion paints having high percentages of nonvolatiles and containing large amounts of zinc oxide have been formulated and found to be stable for over one year (Kubie et al., *Offic. Dig. Federation Socs. Paint Technol.*, *34,* 693).

Miscellaneous Compositions

A cold setting core oil consisted of a mixture of a conjugated oil and a blown nonconjugated drying oil (Tobler, *U.S. 3,023,112*). A prtective composition for metals consisted of an oily material such as sulfonated oil, petroleum, lard oil, linseed oil, tung oil, or soybean oil, a solid absorbent such as diatomaceous earth, an alkali chromate or dichromate as a corrosion inhibitor, and water (Larrieu, *U.S. 3,035,926*). A wood primer consisted of linseed oil-modified glyceryl phthalate, an organic solvent, and a pigment (LaBerge, *U.S. 3,008,847*). When disproportionated rosin had an ultraviolet extinction coefficient higher than 2.8 at 241 mμ, the use of the potassium soap of the rosin in emulsion polymerization of styrene and butadiene had a tendency to decrease the yield. Yield was not affected by the amount of unsaponifiable matter or free resin acid in the soap except when the acid value was over 15 (Wakabayashi et al., *Yukagaku*, *11,* 65).

Analytical Methods

The subcommittee on drying oils of the Fat Analysis Committee of the American Oil Chemists' Society reported on the present status of methods for color, viscosity of transparent liquids by bubble time method, saponification value, and drying time (*JAOCS*, *39,* 431). The advantages and limitations of three methods (gas chromatography, isomerization by soya lipoxidase, and alkaline isomerization) for the determination of polyunsaturates in oils were discussed (Guillaumin and Drouhin, *Rev. Franc. Corps Gras*, *9,* 415). Molecular weights of polymerized linseed oils having viscosities from 8–111 poises were calculated from measurements of sedimentation viscosity in the ultracentrifuge (Lück et al., *Fette, Seifen, Anstrichmittel*, *64,* 239). Small amounts of polymeric materials in oils were concentrated with urea, and dimeric acids were then detected by paper chromatography (Rost, *Ibid.*, 427). The examination of polymers by pyrolysis, followed by gas chromatographic analysis of the degradation products, was an effective procedure (Dandoy, *Ind. Chim. Belge.*, *27,* 355). A procedure for identification of di- and monocarboxylic acids in alkyds and polyester resins involved conversion of the acids to their methyl esters, followed by programmed temperature gas chromatography (Esposito and Swann, *Anal. Chem.*, *34,* 1048). A photofluorometric test procedure for the determination of the dry times of paint films was described (Beispiel and Bricker, *Offic. Dig. Federation Socs. Paint Technol.*, *34,* 59).

Pigments

At certain concentrations, aqueous mixtures of zinc oxide and titanium dioxide showed increases in viscosity, pH, and sedimentation volume. This behavior may be responsible for some of the thickening in emulsion paints containing these pigments (Princen and DeVena, *JAOCS*, *39,* 269). In connection with the instability of bodied linseed oil emulsions containing zinc oxide, interactions between zinc oxide or zinc salts and a variety of surfactants were studied by chemical analysis, x-ray defraction, pH, and conductivity titrations (Vold and Singh, *Ibid.*, 424). The beneficial influence of zinc oxide pigment on the weathering characteristics of the white latex films was discussed (Werthan, *Offic. Dig. Federation Socs. Paint Technol.*, *34,* 1099). The factors influencing the white hiding power of titanium dioxide pigments were discussed (Stieg, *Ibid.*, *34,* 1065). An empirical equation was derived which predicts scattering coefficients for white pigments in acrylic films from a knowledge of pigment concentration, particle size and particle size distribution, relative index of refraction and wavelength of incident light (Mitton, et al., *Ibid.*, *34,* 73). New grade of titanium dioxide pigment and their field of application (Taylor, *Ibid.*, *34,* 95) and optical propenes of newer titanium dioxides were discussed (Gomick, *Ibid.*, *34,* 90). A reflectometry method is given for measuring the tinting strength of white pigments based on Kubelka-Munk theory (Mitton and Jacobsen, *Ibid.*, *34,* 704). The spectral reflectivity of titanium dioxide and aluminum were measured. At all wavelengths higher than 400 mμ, titanium dioxide was shown to be a better reflector than aluminum (Herrington, *Ibid.*, *34,* 1061). Recent trends in organic pigments used in paints were discussed (Matsin, *Ibid.*, *34,* 104).

Plasticizers and Stabilizers

The following types of materials were reported as plasticizers (for vinyl polymers, unless otherwise stated). Vinyl esters of hydrogenated cyclic acids, and nitriles and morpholides of cyclic acids and hydrogenated cyclic acids were evaluated for this use. They were as good as a di-2-ethylhexyl phthalate control in many properties, but the nitriles and morpholides had poor light and heat stability (Friedrich and Beal, *JAOCS*, *39,* 528). An epoxidized diacetoglyceride of lard or tallow acids served as a plasticizer-stabilizer (Swern and Knight, *U.S. 3,049,504*). Another plasticizer consisted of a mixture of fatty esters, some containing two vicinal acyloxy groups, others containing an epoxy group (Rowland and White, *U.S. 3,051,672*). Various acetal-esters of azelaaldehydic acid (formed by ozonization of unsaturated oils) were being evaluated as plasticizers (Pryde and Cowan, *JAOCS*, *39,* 496). Dialkyl 9(10)-phosphonostearates and dialkyl ω-phosphonoundecanoates were good primary plasticizers, having low migration loss and imparting low temperature flexibility (Sasin, *Ibid.*, 448). The toxicity of an epoxy resin paint to chicks was found to be due to a chlorinated biphenyl plasticizer in the paint (McCune et al., *Poultry Sci.*, *41,* 295). *Vernonia anthelmintica* seed oil, trivernolin (the main constituent of the oil), and barium and cadmium vernolate (epoxyoleate) were good stabilizers for plasticized polyvinyl chloride (Riser et al., *JAOCS*, *39,* 266). Barium, cadmium, and lead soaps of wool wax acids imparted light stability to plasticized polyvinyl chloride; barium, magnesium, lead, and nickel soaps of these acids imparted heat stability (Eisner et al., *Ibid.*, 181). Vinyl chloride-vinyl acetate copolymer resins and polyvinyl chloride resins in surface coatings were stabilized against heat through the addition of epoxy materials. The influence of molecular configuration on the effect of epoxy heat stabilizers for vinyl resins is discussed (McGuigan, *Offic. Dig. Federation Socs. Paint Technol.*, *34,* 939).

PRODUCTION PROCESSES

EXTRACTION

Some theoretical aspects of solvent extraction were reported. Hisa et al. found that in the solvent extraction of oil from soybean cake of a given thickness with ethyl acetate, the relationship between extraction time (t), and the residual oil content (Z) could be given by the expression Z = kt 0.439, where k is a constant (P. J. Hisa et al., *Hua Kung Hsuch Pao*, No. 2, 157–162 (1959). In another kinetic study, the relationship between the log rate of extraction and the log of oiliness of the substance was shown to be linear only at a relatively low oiliness. (V. V. Beloborodov, *Tr. Vses. Nauchn Issled. Inst. Zhirov*, No. 21, 127–137 (1961).

The influence of certain additional factors on extraction of oil was investigated. Many anionic and nonionic detergents were found to be effective in suppressing the ability of the liquor to emulsify the oil during extraction of olive oil. (J. M. Martinez, et al., *Grasas y Aceites*, *12,* 166–173 (1961). The effect of ultrasonics on oil extraction from sunflower cake by naphtha was observed to be too small for commercial utilization (E. I. Ivanova, *Tr. Vses. Nauchn-Issled. Inst. Zhirov, No. 19,* 260–268 (1959). In the direct extraction of fresh avocado tissues with a mixture of 95% ethanol and Skelly Solve B (3:2 v/v), ethanol facilitated the extraction by dehydrating the tissues. (G. M. Montano, et al., *Food Tech.*, *16,* 96–101 (1962).

The effect of operating variables on extraction efficiency, loss of solvent and quality of the oil was discussed. Takeshita et al., found that extraction of oil from rice bran with hot solvent gave better extraction efficiency than cold solvent. Further, the plot of extraction rate vs. moisture content had a maximum at 3% moisture (Yasuhiko Takeshita et al., *Yukagaku, 11,* 5–10 (1962). The relationship between operating variables and the loss of naphtha in a commercial extraction unit was discussed (Bernhard Freier et al., *Lucrarile Inst. Cercetari Aliment, 5,* 77–98 (1961). Beloborodov obtained an increase in the rate of distillation and an improvement in the quality of the oil by distilling the miscella of sunflower oil in naphtha, in a stream of carbon-dioxide. (V. V. Beloborodov, *Tr. Vses Nauchn-Issled, Inst. Zhirov,* No. 19, 159–158 (1959).

Takeshita observed that fatty acids were extracted more readily than glycerides and esters during solvent extraction of oil from rice bran stored for 1–2 years. (Y. Takeshita, *Yukagaku, 11,* 105–109 (1962). A comparison of production of rice bran oil by expression and by solvent extraction indicated that in the expression method the wax esters would be separated first followed by free fatty acids while the reverse would occur in the extraction process (Y. Takeshita et al., *J. Oil Chem. Soc. (Japan), 11* (3), 101–104 (1962).

The effect of the chemical nature and composition of the solvent on extraction efficiency and quality of the extracted oil was extensively studied. Extraction of soybean flakes with ethanol of four different concentrations, 90, 95, 98, and 100%, yielded oils of same quality. But lipids were extracted at a more rapid rate than nonlipids with the increase in concentration of the solvent. (Lionel K. Arnold et al., *JAOCS, 39,* 379–380 (1962). The same authors found that hexane was intermediate between absolute ethanol and 95% ethanol in its solvent power for extraction of peanut grits. More nonlipids were extracted by 95% ethanol than by absolute ethanol; none was extracted by hexane. Alcohol-extracted oils were slightly higher in color and free fatty acids than hexane-extracted oil (*Ibid.,* 296–297 (1962).

Arnold et al., observed no significant difference in the quality of the oils extracted from soybean with four different solvents. However, the rate of extraction increased in the following order of these four solvents, isopentane, pentane, isohexane and hexane. (Lionel K. Arnold et al., *JAOCS, 39,* 328 (1962). Extraction of soybean with butane gave a light colored oil with a low acid value and lecithin content. The refined and deodorized oil had good stability showing little flavor reversion (K. Yasuda et al., *J. Oil Chem. Soc. (Japan), 10* (9), 527–531 (1961). The color intensities of oils extracted with three different solvents were shown to be in the following order: trichloroethylene: benzene: petroleum ether at a ratio of 4:2:1 (Yasuhiko Takeshita et al., *Yukagaku, 11,* 5–10 (1962).

A two step extraction of cottonseed, first with 50–80% acetone followed by extraction with pure dry acetone or ligroine, resulted in a gossypol-free lemon yellow oil with low acid value (A. L. Markman et al., *Maslob-Zhir Prom., 28,* 14–17 (1962). King et al., patented a process for extraction of cottonseed meats with a homogeneous, constant boiling mixture of 53 parts of acetone, 44 parts of hexane, and 3 parts of water. The miscella was then mixed with water or hexane and acetone was removed by azeotropic distillation. Then the remaining two layers comprised of a layer of hexane solution of cottonseed oil and pigments and the other layer was an aqueous dispersion of gums. The oil was recovered from hexane layer after removal of the pigments by alkaline washing (W. H. King et al., *U.S. 3,025,314*).

New equipment included an apparatus for continuous prepressing and solvent extraction of palm kernel meats (I. E. Bezuglov et al., *Mazlob-Zhir. Prom., 28,* No. 7, 34–37 (1962) and a unit, consisting of a cylindrical vessel with a steam heated jacket and a centrally located, upwardly directed, tangential atomizer, which was found to be well suited for preliminary desolventization of the miscella of sunflower oil in naphtha. (V. V. Beloborodov et al., *Maslob-Zhir. Prom., 28,* No. 7, 8–10 (1962).

Two processes, patented for recovering animal fat, included a method of rendering by direct contact with steam under pressure. The products of rendering were separated by standing in a non-agitated condition. Then the fat was dried by heating at 230F under a vacuum of 24 inches of mercury (D. J. Krumm et al., *U.S. 3,025,315*), and a continuous process for recovering fat from inedible, slaughter house material consisting of heating the pulverized material at 220–270F and separating the solid and liquid phases by centrifugation (P. Speer et al., *U.S. 3,046,286*).

Modern technology of fats and fatty products was reviewed. (H. P. Kaufmann et al., *Fette, Seifen, Anstrichmittel, 64,* 280–286 (1962).

REFINING

New equipment included a unit for continuous degumming of vegetable oils by intimately mixing the oil and water in a venturi meter-type of hydrator, followed by centrifugal separation of the oil, water and hydrated phospholipids Jakubowski et al., *Rev. Franc. Corps Gras. 9,* 83–87 (1962); a centrifuge for refining of fatty oils (B. H. Thurman, *U.S. 3,027,389*) and an apparatus for continuous neutralization of fats and oils with alkali (N. A. Kominskii et al., *Maslob-Zhir Prom., 28,* No. 7, 12–14 (1962). The suitability of a combination of the Podbielniak Duozon-Hydrazon centrifugal vegetable oil refining machines for continuous refining and simultaneous washing of crude coconut oil was demonstrated (J. E. Morren et al., *JAOCS, 39,* 278–279 (1962).

Several new processes and newer application of old processes were investigated. Thurman patented a process for centrifugal purification of fatty oils (B. H. Thurman, *U.S. 3,027,390*). The centrifugal process was found to be superior to heating and settling process for the separation of moisture and impurities (Y. Takeshita et al., *J. Oil Chem. Soc. (Japan) 11* (3), 165–168 (1962). Neutralization of a fat or oil without emulsification by soap was achieved by treating the oil or fat with alkali in an amount less than that required to neutralize the free fatty acids, sufficient water to dissolve the formed soap and a substance which yielded in water a phosphate or an ammonium ion (M. Mitani, *U.S. 3,008,972*). Bruecke patented a three stage process for refining vegetable and animal oils and fats, by first treatment with HCl and Na_2SO_4, next with Na_2CO_3, followed by concentrated salt solution, (Otto Bruecke, *Ger. 1,099,676*). Refining of castor oil was carried out by treating the oil with 40% aqueous Na_2CO_3 at 80–100C, for ½ hr, followed by water washing of the soapstock at 60C (*Council of Sci. Ind. Res. (India), 69, 383*).

A comparative study of three different, Ranchers Miscella, Modified Soda Ash and Sanbra, refining processes for refining of cottonseed oil showed that Ranchers Miscella refining process yielded more oil of lighter color per ton of seed than either of the other two methods. (G. C. Cavanagh, *JAOCS, 39,* 1, 44–96 (1962). An investigation of the suitability of methyl alcohol for selective extraction of free fatty acids from crude cottonseed oil under high pressure and high temperature showed that the process would not be commercially feasible in as much as excessive amount of solvent would be needed (W. B. Harris, *JAOCS, 39,* 314–315 (1962).

Phase relationships and the effect of process variables in the miscella refining of cottonseed oil were investigated. A study of the phase relationship showed that in caustic refining of hexane miscellas of cottonseed oil, the caustic readily dispersed into the oil-hexane phase. Refining losses were found to be lower for miscellas containing less than 70% oil. Concentrations as low as 40% were refined successfully. The refining loss was inversely related to the viscosity of the solution (M. A. Zeitoun et al., *JAOCS, 39,* 286–289 (1962). For refining of cottonseed oil in miscella the optimal concentration of alkali was about 88–200 mg/litre. Pre-extraction of gossypol from the miscella with anthranilic acid had no effect on alkali refining (A. L. Markman et al., *Maslob-Zhir. Prom., 27,* No. 12, 12–16 (1961). The effect of natural and activated clays, chalk, and citric acid, water washing and centrifugal separation, on the removal of soaps from miscella in the process of miscella refining of cottonseed oil was discussed. (A. L. Markman et al., *Maslob-Zhir. Prom., 28,* No. 3, 18–20 (1962).

Refining losses and bleached color were highest with crude cottonseed oils of high fatty acid content. Increasing the concentration of lye during refining increased refining losses but decreased bleached color. (R. de Castro, *Grasas y Aceites 12,* 259–266 (1961). The presence of phosphatides and phosphatide-bound gossypol increased neutral oil loss during alkali refining, whereas free gossypol reduced oil losses (S. N. Burnsheva et al., *Tr. Vses. Nauchn-Issled. Inst. Zhirov.,* No. 21, 164–177 (1961).

The effect of pre-treatment of cottonseed oil on alkali refining and on the quality of the oil was extensively studied. Borshchev obtained a patent for refining cottonseed oil by a preliminary treatment with calculated amount of anthranilic acid, based upon the gossypol content, followed by alkali refining without separation of gossypol-anthranilates. (S. T. Borshchev et al., *U.S.S.R. 141, 570*). In another patent cottonseed oil was alkali refined after a preliminary treatment with a water solution of Na_2SiO_3 (Ram Das Saxena, *Indian 70, 342*). Initial treatment with p- or o-amino-benzoic acid yielded a light colored cottonseed oil after refining with alkali. The crude oils treated with p- or o-amino-benzoic acid retained their initial refining capacity for 5.5 months. (A. B. Petushina, *Tr. Vses. Nauchn-Issled Inst. Zhirov. No. 20,* 40–58 (1960). An improvement in color and an increase in yield were obtained by addition of 2% (based on the weight of oil) of 30% H_2O_2 solution to the alkali used in refining cottonseed oil. The color of the soapstock was also improved

(P. L. Narayana Rao et al., *Indian oilseeds J. 6,* 130–134 (1962).

New methods for separation of gossypol from cottonseed oil included treatment with an aqueous solution of borax at a concentration of 0.5 to 1.5 parts by weight per 100 parts of oil, followed by water washing of the borate complex (T. R. Sheshadri et al., *U.S. 3,043,856*). Another method involved the mixing of prepressed or solvent extracted oil with 0.53% of anthranilic acid for every percent of gossypol for 1–2 hr at 65–70C and the mixture was then cooled to 25–30C. The anthranilate of gossypol was removed by filtration (V. V. Rzhekhin et al., *Masloboino Zhirovaya Prom., 27,* No. 8, 26–29 (1961).

Anion exchange resins were found to be of practical use in deacidification of fats and oils. (H. Inoue et al., *J. Oil Chem. Soc. (Japan), 11* (3), 109–112 (1962). The regeneration of ion exchange resins used to deacidify olive oils, by alcoholic NaOH was studied. The deacidifying properties of some of the resins studied were still unchanged after 55 regenerations. (M. De Felice et al., *Revista Ital. Sost. Grasse, 3,* 125–129 (1962).

BLEACHING

Two patents for removal of colors from oil which were difficult to bleach included treatment of the oil at 225C with activated alumina comminuted to pass a 400 mesh screen and containing at least 10% by weight of moisture (W. A. Pons, Jr. et al., *U.S. 3,036,102*) and in another method the oil or fat was heated very quickly, preferably, while mixed with bleaching adsorbent, followed by countercurrent contact with steam in a packed column. (Renato Raffaeta, *U.S. 2,980,717*).

New methods of decolorization of oil in solution with different adsorbents were investigated. Rice bran oil dissolved in organic solvents was bleached with ion exchange resins. Use of both anion and cation exchange resins in series was reported to be very effective. (H. Inoue et al., *J. Oil Chem. Soc. (Japan) 11* (3), 162–165 (1962). The normal earth-bleaching process was replaced by a new technique of treating the neutral oil in light petroleum solution with activated alumina. The products were found to have improved color and longer keeping quality as compared with conventionally bleached oils (A. Crossley et al., *JAOCS, 39,* 165–168 (1962).

The suitability of several clays as bleaching earths was investigated. The ability of 5 different clays, activated with 10% H_2SO_4 for 3 hr, to bleach cottonseed oil was described (A. C. Markman et al., *Tr. Vses. Nauchn-Issled Inst. Zhirov.,* No. 21, 178–192 (1961). The bleaching activity of 7 bentonites was studied (Nasir-Ud-Din et al., *Pakistan J. Sci. Ind. Res., 4,* No. 2, 66–69 (1961).

In the bleaching of oils with various earths under different conditions, Langmuir equation suited better than other mathematical relationships to interpret the theoretical maximum color removal to be expected in the bleaching operation. In general, bleaching at atmospheric pressure was considerably more effective than bleaching under vacuum. (P. Fontana et al., *Revista Ital. Sost. Grasse 4,* 180–187 (1962).

Two processes were reported for reclamation of spent bleaching adsorbent. A water slurry of the spent clay was treated with lipase from castorseed, followed by extraction with an aqueous solution of Na_2CO_3. The recovered clay had bleaching action equal to that of the original. (Bombay Chemicals (P) Ltd., *Indian 69,921*). Spent alumina from bleaching of cottonseed oil was washed with pet. ether, dried and reactivated by simple incineration at 400–700C and remoistening to at least 10% moisture content. The cycle of bleaching and regeneration may be repeated indefinitely with only nominal mechanical losses of alumina. (J. C. Kuck et al., *JAOCS, 39,* 84–86 (1962).

DEODORIZATION, WINTERIZATION AND FRACTIONATION

Two new equipments for deodorization of fats and oils were patented. A continuous deodorization unit consisted of four horizontal, cylindrical chambers using direct or indirect steam for heating the oil and a central cylindrical duct for receiving vapors from each of the heating chambers. (*Deggendorfer Werft Und Eisenbau* G. m. b. H., *Ger. 1,117,804*). Adler used a series of vessels with separate steam inlets capable of producing a finely divided spray of steam for deodorizing oils. The vessels were connected to a common vapor outlet pipe (Rolf Adler et al., *Ger. (East), 19, 229*).

A progressive deterioration in the quality of soybean oil was observed with the increase in the temperature of deodorization from 240–295C (K. Yasuda et al., *J. Oil Chem. Soc. (Japan) 11* (1), 2–5 (1962).

Various methods were reported for fractionation of fatty acids as well as triglycerides. A blend of winterized oils was obtained by subjecting to winterization a mixture of at least two oils in the ratio of 30:70. (C. M. Gooding, *U.S. 3,048,491*). Modified lard was fractionated by heating to a temperature at which all crystalline nuclei disappeared and then cooled slowly until the appearance of first crystals. This temperature was maintained for 12–72 hr. The temperature was lowered slowly (0.05–0.5C/hr) to a point at which the mixture was still mobile. The mixture was held at this temperature for 2–6 hr and the solid was separated by filtration (Juan de la Cierva, *Span. 259,918*).

The formation of acid soaps was used successfully for the separation of a mixture of fatty acids. The acid soaps of saturated acids were crystallized from water leaving their unsaturated counterparts in solution. Optimum crystallization conditions involved a soap concentration of 2–5%, pH adjustment to between 7.0 and 8.0, an initial crystallization temperature not below 25C and a crystallization period of at least 4 hr, during which time cooling gradually to a final temperature of 5–10C. (W. O. Munns et al., *JAOCS, 39,* 189–192 (1962), *Ibid.,* 192–195). Tall oil fatty acids were fractionated into 80–90% oleic acid and 60–80% linoleic acid by precipitation of oleic acid as acid soap from polar solvents. Though the choice of solvent was not critical, attractive filtration rates were obtained only with methanol and acetone. (E. M. Meade, *JAOCS, 39,* 235–237 (1962).

HARDENING

Several studies on kinetics of hydrogenation of different systems were reported. In the investigation of consecutive catalytic reduction of linolenate to linoleate to oleate, with the aid of radioisotopic tracers and gas chromatography, evidence for a triene to monoene shunt was obtained. When the reduction was carried out with hydrazine no evidence for such a shunt was noticeable. Three different methods for determination of the reaction rates from these kinetic measurements were discussed (C. R. Schofield et al., *JAOCS, 39,* 90–95 (1962). Specific rate constants for hydrogenation of linoleic and oleic radicals were calculated from the data obtained from hydrogenation of sunflower oil and from the determination of the surface of the Ni-catalyst used for hydrogenation. (S. Yu. Elovich et al., *Zh. Fiz. Khim., 36,* 1255–1260 (1962). Dutton derived equations from kinetic theory for determining the ratio of hydrogenation rates of linoleate and linolenate acyl groups. These equations were based upon the analysis for linolenate after absorption of 0.5 mole of hydrogen by an equal mixture of linolenate and linoleate. These equations were applied to evaluate the selectivity of hydrogenation catalysts (H. J. Dutton, *JAOCS, 39,* 95–97 (1962). In the hydrogenation of cottonseed oil and methyl oleate, in which mass transfer resistances were eliminated by sufficient agitation, the rates of hydrogenation for both geometrical isomers and positional isomers were essentially identical. A method for evaluating the relative reaction rate constants for each step was outlined. Selectivity was quantitatively evaluated from these rate constants. (L. F. Albright et al., *JAOCS, 39,* 1, 14–19 (1962).

The influence of certain factors like free fatty acids on hydrogenation was investigated. In the hydrogenation of cottonseed oil with 0.2% of Cu-Ni (1:1) catalyst at 240C, the rate of hydrogenation fell significantly with the increase in free fatty acid concentration above 2.5%. Similar observation was made in respect of selectivity which, although, increased by addition of free fatty acids up to 1% (R. M. Mirzakarimov, *Uzbeksk Khim. Zh.,* No. 3, 52–58 (1961), "KW-type" and Adkin catalysts were least sensitive to the presence of free fatty acids during hydrogenation; but lowering of acid number to less than 0.4 was indispensable if the catalyst was to be recycled. A simple method was also proposed to regain and accelerate the activity of the catalyst by treating the spent catalyst with alkaline solution under aeration. (Kazutomo Maebashi, *Yukagaku, 11,* 60–64 (1962). The presence of 1% trichloromethylene in rice bran oil aided in regulating hydrogenation velocity in high pressure hydrogenation. The presence of water up to 2% did not affect the results. (Y. Takeshita et al., *J. Oil Chem. Soc. (Japan), 11* (3), 168–174 (1962). A study of the hydrogenation of randomly rearranged soybean oil demonstrated that the position occupied by an unsaturated fatty acid in a triglyceride did not influence its rate of hydrogenation (F. H. Maltson et al., *JAOCS, 39,* 307–308 (1962).

New methods of preparation and use of different catalysts were investigated. A patent for the preparation of highly selective palladium catalyst for hydrogenation at low temperatures avoiding undesirable side reactions was issued (Engelhard Industries Inc., *Ger. 1,123,423*). Satisfactory margarine stocks were made with a palladium on carbon catalyst. The catalyst was shown to make a satisfactory product even when, on continued reuse, the ratio of oil to metal reached 400,000–1. (Mykola Zajcew, *JAOCS, 39,* 301–304 (1962). The catalyst regenerated at 350C had greater activity than at 250C and 450C when refined sunflower in ethanol solution was hydrogenated at 40, 50, and 60C with Ni-Zno catalyst containing different quantities of Ni. (N. M. Popova et al., *Tr. Inst-Khim Nauk. Kaz. S.S.R.*, *7*, 26–32 (1961).

Processes for hydrogenation in vapor phase and in solution were developed. Hydrogenation of unsaturated higher fatty acids in the vapor phase was carried out with a mixture of water vapor and hydrogen in the molecular proportion from 2:100–200:100 (L. H. Ruiter et al., *U.S. 3,015,667*). In the solvent extraction of glycerides from vegetable or animal origin, the glycerides were hydrogenated by passing the hexane solution over Raney Ni alloyed with aluminum or copper or silica gel catalyst. The degree of hydrogenation was controlled by refractometry (Hans Paul Kaufmann, *Brit. 870,393*). A product with I. V. of 2 was obtained by hydrogenation of an ethanol-castor oil mixture with 2% Raney Ni catalyst for 3 hr at a temperature of 75–100C and 250–300 lb/sq inch pressure. 12-hydroxystearic acid was separated in 76% yield from the hydrogenated product. (K. M. Manerkar et al., *J. Sci. Ind. Res. (India) 20 D*, 465–466 (1961). In a study of the effect of operating variables on the selective hydrogenation of castor oil, the best results were obtained with 2% catalyst (Raney Ni or Ni formate) at a temperature of 150C and 150 lb/inch^2 pressure for 5 hr. Hydrogenation in ethanol reduced the time but required higher pressure. (H. Sripathi Rao et al., *J. Sci. Ind. Res. (India) 21O*, 89–92 (1962).

Processes for removal of catalyst poisons from oils and reclamation of spent Ni catalyst were described. Catalyst poisons from vegetable and animal fats were removed before hardening by treatment with an aqueous solution of 0.03% $CuCl_2$ and 0.3% water glass at 85–90C (Erich Hlavsa, *Czech. 97,727*). An economical method of reclaiming spent Ni catalyst consisted of saponification of fatty material with NaOH, hydrolysis of the soaps and conversion of Ni to sulfate with conc. H_2SO_4 followed by precipitation of Ni as carbonate and conversion of carbonate to formate (Paul Fr. Seibel, *Seifen-Oele-Fette-Wachse 88*, 391–392 (1962).

A survey of commercially available catalysts for hydrogenation was made in a search for high selectivity and low isomerizing characteristics. Nickel, Palladium and Platinum catalysts were examined. Although platinum catalysts produced the lowest isomerization, their selectivities were low. (A. E. Johnston et al., *JAOCS, 39*, 273–276 (1962). Practical aspects of hydrogenation were discussed (R. R. Allen, *JAOCS, 39*, 457–458 (1962).

INTERESTERIFICATION

Random interesterification eliminated graininess in lard by reducing the content of 2-palmitoyloleoyl-stearin to its random proportion and by producing a mixture of disaturated glycerides which was substantially lower melting than original 2-palmitoyloleoyl-stearin. (E. S. Lutton et al., *JAOCS, 39*, 233–235 (1962). Good yields (up to 72%) of monoglycerides were obtained by catalytic interesterification of natural fats in pyridive solution at 100–110C for 15 min with $NaHCO_3$ or Sn $(OH)_2$ as catalyst. (K. Taeufel et al., *Fette, Seifen, Anstrichmittel, 64*, 513–517 (1962).

A study of the kinetics of acetolysis of glycerides showed that the reaction was essentially unimolecular. Using a threefold weight of 98% acetic acid, 1.0% catalyst and a reaction time of 12–24 hr at 100–118C, acetolysis of around 90% were obtained. Perchloric acid was found to be the most powerful catalyst and water was an essential reactant. (E. M. Meade et al., *JAOCS, 39*, 1–3 (1962); *Ibid.*, 4–6).

A review on modification of natural fats by interesterification was published (D. Domingo Martin, *Grasas y Aceites, 12*, 126–133 (1961).

PARTIAL ESTERS AND FAT SPLITTING

The effect of operating variables in the manufacture of monoesters was studied. In the direct esterification of fatty acids with glycerol at 180C with 0.1% NaOH catalyst, the maximum amount of monoglyceride formed was in the range of 55–60% of the fatty product at equilibrium. The alkaline catalyst increased the initial rate of reaction without lowering the time required to reach equilibrium. Catalyst also helped in depressing the diglyceride formation. (R. Basu Roy Choudhury, *JAOCS, 39*, 345–347 (1962). Use of high pressure in the preparation of monoglycerides from castor oil increased the concentration of monoglycerides at equilibrium, while increased temperature enhanced the rate of formation of the monoglycerides. (R. K. Kochhar et al., *Indian Oilseeds J., 6*, 144–152 (1962). In the manufacture of partial esters from glycerol and triglycerides by transesterification, the reaction could be followed by solubility of the products in 95% ethanol. (A. P. Smirov, *Tr. Vses Nauchn-Issled. Inst. Zhirov.*, No. 21, 258–267 (1961).

Methyl esters of fatty acids were prepared from soapstocks by heating a homogeneous mixture of vegetable oil soapstock in methanol for 10–20 min at a temperature of 115–125C and at a pressure sufficient to maintain the methanol in the liquid state and in the presence of 1–5% Twitchell Catalyst. The unreacted methanol and water were flash evaporated. (P. H. Eaves et al., *U.S. 3,010,997*).

A longer induction period and greater use of Twitchell reagent in splitting rancid oils were attributed to the effects of negative catalysts present in the oil. (Shunroku Kanno et al., *Kogyo Kagaku Zasshi, 64*, 311–315 (1961). Rate of hydrolysis of sunflower oil at 225C in the presence of different catalysts was studied (M. P. Bespyatov et al., *Maslob-Zhir. Prom., 28*, 14–17 (1962).

VEGETABLE AND ANIMAL FATS AND OILS

The modern technology of fats and fatty products and high pressure method of oil manufacture (H. P. Kaufmann, et al., *Fette, Seifen, Anstrichmittel 63*, 862–869 (1961) and production and utilization of sunflower seed oil in India (N. Bhojaraj Naidu, *Indian Oilseeds J., 4*, 62–69 (1960); *Ibid.*, 70–84) were reviewed.

Process control problems such as the control of a discontinuous process, the automation of a semi-continuous process, and the operation of a continuous process in the fat industry were discussed (F. Piwinger, *Fette, Seifen, Anstrichmittel 63*, 1144–1149 (1961).

Heating of sesame seed at 100C for 15 min before expressing the oil improved the stability of the oil (Bruno J. C. Cristini et al., *oleagineaux 17*, 463–467 (1962). Experiments on olive oil showed that the formation of brown color on HNO_3 addition was not specifically due to the presence of esterified fatty acids in the oil but an index of alteration of unidentified color forming compounds accompanying natural oils. (V. G. B. Martinenghi, *olearia 15*, 308–311 (1961). The dehydration of castor oil yielded a maximum of 30% 9, 11- and 70% 9, 12-linoleic acid. The different stages of dehydration was followed by the determination of refractive index. (M. Fauve, *oleagineux 17*, 703–705 (1962).

The fatty acid composition of the oil of *C. Crista* was nearly the same whether fractionation was accomplished by (a) stepwise addition of urea or (b) urea-adduct elution method (G. B. Behera et al., *Indian J. of Applied Chem., 23*, 221–225 (1960).

BY-PRODUCTS

New or improved processes for the production of protein rich material from oilseeds were reported. A free flowing, light colored soybean flour with 72–77% protein was obtained by washing the defatted soybean meal with aqueous alcohols followed by flash-desolventization. (G. C. Mustakas et al., *JAOCS, 39*, 222–226 (1962). Benchscale studies were carried out for the removal of mustard glucosides by enzymatic conversion to allyl isothiocyanate which could be removed during cooking step before filtration-extraction of the meal. (G. C. Mustakas et al., *Ibid.*, 372–377). The cost for production of protein rich, defatted peanuts were studied (L. J. Molaison et al., *Ibid.*, 473–476).

Methods for obtaining high grade soybean lecithin were described. Lecithin containing 1–2% residual oil was obtained in 95% yield by countercurrent extraction of crude soybean lecithin with a mixture of hydrocarbon-acetone-water solvent (H. Pardun, *Fette, Seifen, Anstrichmittel 64*, 536–541 (1961). High purity lecithin was separated from the concentrated hexane miscella of soybean oil by addition of water. A powdered lecithin of 92–96% purity was prepared by treating the hydrated lecithin with a mixture of hydrogen peroxide and ammonium hydroxide. (K. Yoshitomi et al., *J. Oil Chem. Soc. (Japan), 11* (3), 123–127 (1962). Considerable discoloration of lecithin occurred in the miscella concentrating process followed by further browning in the stripping process. (K. Yoshitomi et al., *J. Oil Chem. Soc. (Japan), 10* (9), 540–542 (1961).

The use of ion exchange resins in the process of glycerol recovery was investigated. Fats and colloidal impurities were removed by boiling and centrifugation before the treatment of aqueous glycerol with ion exchange. (Ditta Fratelli Gianazza, *Ital. 579,936*). Ion exchange method was found to be more economical than distillation for the isolation of glycerol from sweet waters obtained from splitting of oils and fats (Emil Krejcar et al., *Chem. Prumysl 11*, 660–663 (1961).

A process for the manufacture of wax-like esters of fatty acids of more than 18 carbon atoms and a major alcohol component having 2–3 alcoholic hydroxyl groups was patented (J. Kaupp et al., *U.S. 3,053,681*). Benzene was found to be the best solvent for extraction of crude peat wax. (A. J. Howard et al., *JAOCS, 39*, 250–255 (1962).

Industrial applications of cottonseed and soybean soapstocks were reviewed (M. Balsi, *Lipidos 21*, 108–109 (1961).