

Annual Review of Literature on Fats, Oils, and Soaps. Part 2

Report of the Literature Review Committee *

M. M. PISKUR, Swift and Company, Chicago, Illinois

Deterioration

REVIEWS. A comprehensive review on oxidative spoilage of fats by Täufel & Rothe (*Angew. Chem.* 61, 84) contained information on prooxidants, antioxidants, mechanism of oxidation, and its similarities to intermediate fat metabolism, analytical technic, and organoleptic character. A brief review on same details was made by Althuisius (*Chronica Naturae* 105, 102). Other review communications dealt with rancidification of olive oil (Alcala & Herrera—*Anales real soc. espan. fis. quim.* 44B, 1005, 1019, 1025), antioxidants (Taylor—*Biol. Antioxidants, Trans. 2nd Conf.* 1947, 9; Kiermeier—*Angew. Chem.* 61, 19), and methods, of determining the degree of spoilage of fats (Schmal-fuss—*Milchwissenschaft* 3, 225).

METHODS OF TESTING. Most activity on determining spoilage of fats involved the use of peroxide determinations. Investigation of current procedures for the determinations indicated that precautions should be taken to prevent contact of solutions and reagents with air during analysis. In the iodometric method this was accomplished by bubbling carbon dioxide through the solutions (Mucciolo *et al.*—*Rev. facul-dade vet. med. Univ. Sao Paulo* 3, 272). In the ferric thiocyanate method the effect of atmospheric oxygen was eliminated by use of nitrogen atmosphere over the solution or by use of procedures which permit use of large samples (Chapman & Mackay—*J. Am. Oil Chemists' Soc.* 26, 360).

Watt *et al.* (*Food Tech.* 3, 206) determined the heat stability of fat peroxides to serve as fundamental information in extraction methods for determination of spoilage of fat in natural tissue or food products. In this work the peroxides were higher in fats extracted at room temperatures than when warm solvents were used. Peroxides could be demonstrated in adipose tissue or bacon by a staining technic in which the section was stained with a solution of hemin, pyridine, glacial acetic acid, and leuco-base of 2,6-dichlorophenolindophenol (Glavind *et al.*—*Acta Physiol. Scandinavica* 16, Suppl. 53, 26, 28, 32). The test could also be applied to solutions. Another color reaction of oxidized fatty acid was produced with thiobarbituric acid as the reagent (Wilbur *et al.*—*Arch. Biochim.* 24, 305). The characteristic color was most intense after oxidation catalyzed by ultra-violet light, Linolenates developed most color, linoleates and arachidonates less, and oleic and stearic acid derivatives did not give the color reaction after ultra-violet irradiation.

Lewis *et al.* (*J. Am. Oil Chemists' Soc.* 26, 53; *Anal. Chem.* 21, 762) applied a polarographic procedure for the determination of peroxides in fats. The method was very sensitive at low concentrations of peroxides, the results being linear with chemical methods below peroxide values of 250, and above 250, chemical and polarographic methods did not measure the same compounds. It was suggested that the technic should give an insight to the mechanisms of oxidation for the data indicated that at least three peroxide structures were present. The fundamental data on absorption by fats of ultra-violet light developed by Chevallier *et al.*

(*Arch. sci. Physiol.* 2, 329, 358) should also be of interest in determination of peroxides and elaboration of mechanisms of spoilage reactions.

Procedures for determining stability of fats based on following the development of spoilage reactions on incubation of filter papers saturated with the samples were published (Elvius *et al.*—*Farm. Revy* 48, 17; Dubouloz & Laurent—*Oleagineux* 3, 255). When such a method was compared with a modified Swift Stability Test, the data arranged the oils in the same order of stability, but the relative stabilities approximated for the oils by the two methods were very different (Swain—*Progress Rpt. Pacific Coast Stat., Biol. Sta. & Fisheries Exptl. Sta.* 77, 116). Another method depended on measuring the rate of oxygen absorption of an oil or fat in the Barcroft-Warburg apparatus (Müller—*Mitt. Gebiete Lebensm. Hyg.* 39, 275). In certain oils the stability could be related to the rate of disappearance of vitamin A as determined spectrophotometrically (Kehren—*Oleagineux* 3, 387).

An examination of the catalase test for detection of bacterial contamination of butter was made, because some low catalase butters had undergone serious bacteriological decomposition (Galesloot—*Netherlands Milk and Dairy J.* 3, 113). Several bacteria and yeast were found that could produce serious bacteriological defects without change in the catalase number. A test based on determination of free water-insoluble fatty acid was recommended for adoption as official

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E. W. BLANK
J. B. BROWN
W. H. GOSS
M. M. PISKUR
Chairman

for determination of decomposition of cream and butter (Hillig—*J. Assoc. Offic. Agr. Chemists'* 32, 520).

The Mackey test apparatus for determination of the stability of textile lubricant fats to spontaneous combustion was modified to prevent localized heating (Chambard & Durande-Ayme—*Bull. assoc. franc. chim. inds. cuir* 11, 130).

MECHANISMS OCCURRING DURING SPOILAGE OF FATS. Täufel (*Z. Lebensm.-Untersuch. u. -Forsch* 89, 581) and Täufel & Treimuth (*Ibid.* 121, 511) have developed data to aid the elaboration of the mechanism of the autoxidation of fats. Ketone formation was only connected with the biological action of microorganisms on fats. Literature postulations on formation of epihydrin aldehyde were considered unsound because of lack of experimental data. Suggestions that acrolein was an intermediate in the reaction were disproven by tests which showed that epihydrin aldehyde developed in monoölefinic fats while acrolein did not, and the aldehyde was not obtained from acrolein under conditions which yielded aldehyde from fats. A suggestion that epoxy compounds were intermediates was rejected because pure epoxy oleic and elaidic acids did not yield epihydrin aldehyde under oxidizing conditions. In a search on the source of acrolein formation both free and bound glycerol evolved acrolein, but with tung oil the main source of the acrolein seemed to be from the elaeostearic acid.

Iselin (*Mitt. Gebiete Lebensm. Hyg.* 39, 105, 310) recorded the changes occurring in meat fats on two to three years' storage. The dienolic acid oxidized rapidly, the peroxide forming in 12,13-position and later on, closing the double bond nearer to the carboxyl group; vaccenic acid was more labile than oleic. Data on different characteristics, appearance of aldehydes, polymerization, etc., were also recorded. Swift *et al.* (*J. Am. Oil Chem. Soc.* 26, 297) following identification of 2,4-decadienal and 2-octenal in the oxidation products of cottonseed oil suggested that these develop from the decomposition of isomeric hydroperoxides of linoleic acid. Ellis (*Biochem. J.* 44, XXXVI) in similar work found the initial products were 8- and 11-keto-acids. He suggested that autoxidative mechanisms might present some biochemical significance.

Other communications containing information of interest with regard to the mechanism of spoilage of fats were a record of the relationships between peroxide value and oxygen-uptake of palm oil (Loury & Mellier—*Oleagineux* 4, 665) and a record of the spectral changes of carotene-oil mixtures (Holman—*Arch. Biochem.* 21, 51).

The fact that carcinogenic agents had antioxidant properties and were demethylated during the oxidation suggested that these characteristics might have some implication on tumor formation in the living organism (Rusch—*Biol. Antioxidants. Trans. 2nd Conf.* 1947, 106).

Bombardment of oleic acid with radio active particles caused formation of stearic acid, heptadecene, and polymerized acids (Burton—*J. Am. Chem. Soc.* 71, 4117).

STABILITY OF PRODUCTS. The factors which influence the stability of butters were studied by Barnicoat (*New Zealand J. Sci. Technol.* 29A, 185, 193, 199). Effects of various lights during processing, presence of iron and copper, and use of preserva-

tives were recorded. Low air content of the finished butter was no advantage; citrates added to cream containing iron and copper improve the keeping quality of butter therefrom; and where the bacterial activity maintained a high reduction potential, oxidation was prevented. Storage tests on "Alfa-process" and sour-cream butter showed that at 30 or more days the former deteriorated less (Kellermann—*Die Milchwissenschaft* 4, 40). In works on the effect of metals on storage of butter, copper was very detrimental, while not all agreed as to whether iron salts had some influence (Kruisheer *et al.*—*Netherlands Milk and Dairy J.* 3, 25; Mulder *et al.*—*Ibid.* 37; Staeger—*Molkerei-Ztg.* 1, 5).

A significant correlation was found between the tocopherol content of milk fat and its ability to resist flavor deterioration (Krukovsky *et al.*—*J. Dairy Sci.* 32, 196). It was found that the stability of dairy products could be improved by dairy cattle feeds of high tocopherol level (*Ibid.* 700) or by the addition of tocopherol to the products (Godel—*Molochnaya Prom.* 10, No. 7, 28). However, the best preservation of dairy fat with chemicals was obtained with the use of nordihydroguaiaretic acid (Krukovsky *et al.*—*J. Dairy Sci.* 32, 679, 695; Stull *et al.*—*Ibid.* 31, 1024; 32, 301). Cell suspensions of special yeast strains were found suitable for inhibiting molding and souring of butter (Bogdanov & Maksimova—*Molochnaya Prom.* 10, No. 3, 21; Blok—*Ibid.* No. 4, 19). A method of renovating rancid butter comprised emulsifying in water with skim milk powder and churning (North—*U. S.* 2,485,308).

Storage tests on the Indian butterfat product, ghee, demonstrated that spoilage was chiefly due to oxidative rancidity (Achaya—*Biochem. J.* 44, 561; Ramaswamy & Banerjee—*Ann. Biochem. & Exptl. Med., India* 8, 123). The authors recorded the length of the induction period, amount of oxygen absorbed, the physical characteristics, and the decomposition products isolated. Trivedy (*Indian J. Dairy Sci.* 1, 93) pointed out that much of the retail ghee had decreased in vitamin A content because of oxidation.

In tests on the effect of feeds on the storability of eviscerated poultry, the rancidification of the carcass fats was used as a criterion (Hite *et al.*—*Poultry Sci.* 28, 244, 249). Carcasses of birds fed two per cent hydrogenated fat, 10 per cent alfalfa leaf meal, or one per cent linseed could be stored nine months, but only those that received rations containing supplements of ethanolamine or choline were acceptable after 12 months' storage. The supplements of ethanolamine or choline reduced the amount of linolenate and arachidonate found in the body fat depots.

Physical and chemical methods for control of rancidity in stored frozen fish were recorded by Tarr (*J. Fisheries Res. Board Can.* 7, 237). Storage in atmosphere of carbon dioxide or nitrogen was effective, but the carbon dioxide-stored fish took on other undesirable flavors. Glazing with water containing ethyl gallate or ascorbic acid was effective, while avenex concentrates gave but little protection. The use of ascorbic acid for preservation of fish was also cited in a review on the uses of the acid (Du Bois—*Food Tech.* 3, 119). An investigation on preservation of shark liver oil demonstrated that hydroquinone was the best antioxidant followed by butyl gallate and that citric acid was a synergist for these. Lecithin

was ineffective, and α -tocopherol and di-*tert*-butylhydroquinone were disappointing in the tests.

A part of the investigations on preventing deterioration dealt with the stability of vitamin A in oils. In one series of tests four per cent lecithin with two per cent tocopherol proved most effective (Kern *et al.*—*Ind. Eng. Chem.* 41, 2849). Other advantages of these preservatives were that lecithin enhanced the absorption of vitamin A in humans and tocopherols exerted a sparing action on vitamin A. According to Miles *et al.* (*Proc. Soc. Exptl. Biol. Med.* 70, 162) the sparing of vitamin A occurred with α -tocopherol but not with α -tocopherol acetate. Another report showed that vitamin A alcohol and its palmitic and acetic esters slightly accelerated oxidation of glycerides, but that this effect could be inhibited with very small amounts of tocopherol and estrone (Chevallier *et al.*—*Compt. rend. soc. biol.* 142, 1010). The work of Dassow & Stansby (*J. Am. Oil Chemists' Soc.* 26, 475) demonstrated that nordihydroguaiaretic acid with citric or orthophosphoric acid as the synergist gave good protection of vitamin A in halibut liver oil. These authors recorded the relations between vitamin A destruction and the concentration of antioxidant with and without synergist, and also the relation between vitamin A loss and peroxide formation in the oil.

Work on stability of potato chips indicated that coconut and peanut oils as frying fats yielded the most stable products (Mitchell—*Food Inds.* 21, No. 8, 55). In using antioxidants in potato chip frying fats, Magoffin & Bentz (*J. Am. Oil Chemists' Soc.* 26, 687) recommended butylated hydroxyanisole with propyl gallate and citric acid as synergists, because the protective properties were carried through to the products.

The stabilities of baking fats were investigated. Baking powders inhibited rancidity in baked product fats in the following ascending order of effectiveness: combinations, tartrate, monocalcium phosphate, and pyrophosphate (Watts *et al.*—*Ibid.*, 481). The antioxidant effect of the pyrophosphate powder was shown to be due primarily to a synergism with tocopherol. An investigation on antioxidants for fats used in baking showed that wheat germ oil, nordihydroguaiaretic acid, soybean lecithin, and kamala dye were effective, the first named being the most efficient (Dhar & Aggarwal—*J. Sci. Ind. Res. India* 8b, No. 1, 1). Reinforcement by synergists, such as citric acid, was necessary for all the antioxidants except soybean lecithin. It was also of interest in baking that lard in aqueous systems could not be stabilized with ascorbic acid or cysteine (Scarborough & Watts—*Food Tech.* 3, 152) and that with citric acid, lecithin, and α -tocopherol the stabilizing action was poor (Lips—*Can. J. Res.* 27F, 373). In the latter report gum guaiac gave the greatest protection in lards containing water. The good stability of elaidinization hardened fat was attributed to their hard physical state and to the presence of the residual elaidinization catalyst, selenium, which seems to have antioxidant properties (Bertram—*J. Am. Oil Chemists' Soc.* 26, 83). Olive oil produced by pressing the olives with two per cent ground leaves was more stable than normally pressed oil (Frenguelli—*Olearia* 1948, 675).

General reports on the effectiveness of norconidrin (Fisher *et al.*—*Mfg. Confectioner* 29, No. 4, 24) and butylated hydroxyanisole (Kraybill—*J. Am. Oil*

Chemists' Soc. 26, 449) as antioxidants for baking fats was published. In some baked cracker tests the antioxidant effect of phenols, acyl phenols, and dihydrobenzoic acid did not carry-over to the baked product; gallic esters and 2,3,4-trihydroxybenzoic acid esters showed small "carry-over"; and alkyl catechols had the most activity in both the fats and baked products (Morris & Reimenschneider—*J. Am. Oil Chemists' Soc.* 26, 638).

Comprehensive pharmacological tests were made on hydroquinone in connection with its proposed use as an antioxidant (Woodard *et al.*—*Federation Proc.* 8, 348). Subsequently, the Food & Drug Administration ruled out hydroquinone as a fat preservative (Anon.—*Food Inds.* 21, 369). Other pharmacological data of interest in connection with antioxidants was that propyl gallate showed toxicity only when 100 times more was consumed than would have been possible if all dietary fat was treated with the antioxidant (Orten *et al.*—*Food Tech.* 2, 308). Butyl gallate had a weak inhibiting effect on some fungi and a still weaker effect on bacteria and higher plants (Mainardi & Pesenti—*Farm Sci. e. tec., Pavia*, 3, 542). The substance inhibited *in vitro* action of peroxidase, reductase, and the fermentation of sugar by brewer's yeast.

A patented method of manufacture of gallic esters (W. V. Chem. "Fabriek" Naarden—*Dutch* 63,319), a method of determination of nordihydroguaiaretic in the creosote bush and its distribution in the plant (Duisberg *et al.*—*Anal. chem.* 21, 1393; Botkin *et al.*—*N. Mex. Coll. Agr. Mech. Arts, Agr. Sta. Bull.* 349, 18 pp.), and a patented method of synthesizing nordihydroguaiaretic acid (Mueller *et al.*—*U. S.* 2,456,443) pertained to production of antioxidants for fats. The *l*-ascorbyl fatty acid esters which were prepared to render the acid fat-soluble for use as an antioxidant retained both antioxidant and antiscorbutic activity (Goswami—*Sci. & Culture* 14, 35). Solubility and specific rotation data on *l*-ascorbyl palmitate and laurate were developed by Swern (*J. Am. Chem. Soc.* 71, 3256). In an investigation on the effect of geometric isomerization on antioxidant properties, maleic acid was more effective than its geometric isomere, fumaric acid (Heimann—*Z. Lebensm.-Untersuch. u. Forsch* 88, 586).

Some of the newly patented antioxidants were natural antioxidants treated with ammonia (Buxton & Dryden—*U. S.* 2,461,807-8), β -mercaptopropionyl esters (Gribbins—*U. S.* 2,462,633), mixture of *p*-amino-benzoic acid and caffeic acid (Norris—*U. S.* 2,462,663), mixture of *l*-ascorbic and gallic acids (Norris—*U. S.* 2,462,664), mixture of tocopherol, alkyl ester of gallic acid and lecithin (Hall & Gershein—*U. S.* 2,464,927), gallic acid esters dissolved in lecithin (Hall—*U. S.* 2,464,928), a high protein oat fraction (Musher—*U. S.* 2,466,260), tetrachloro-*p*-benzoquinone (Mueller—*U. S.* 2,472,119), aconitic acid, aconitic acid anhydride, itaconic acid, and itaconic acid anhydride (Lindsey & Maxwell—*U. S.* 2,486,424), various alkyl esters of citric acid (Vahlteich *et al.*—*U. S.* 2,485,631-40), ammonium gallate (Korner—*U. S.* 2,486,177), glycerophosphate (Soc. anon. prod. cano agr.—*Ital.* 426,138), unsaturated aldehydes such as lactaroviolin and lycopinol (Willstaedt—*Swed.* 124,320) and condensation products of 1,2-diketone with aromatic amines (Willstaedt—*Swed.* 124,321). Rendering animal fats in the presence of citrus fruit pulp increased the stability of the prod-

ucts (Piskur & Higgins—*U. S. 2,461,080*). Rancidity of soap was inhibited by addition of five per cent of an organic compound containing at least one trivalent nitrogen (Lever Bros. & Unilever Ltd.—*Brit. 574,504*). The accelerating effect of prooxidant metals in technical fats was retarded with aldol α -naphthylamine (Ducommun—*Bull. Ann. Soc. Swiss. Chron. 2, 507*). The incorporation of salts of bis (*p*-aminobenzoyl-4,4'-diaminostilbene-2,2'-disulfonic acid in transparent wrapping films afforded protection against the rancidity accelerating effect of light rays on wrapped oily or fatty foodstuffs (British Cellophane Ltd.—*Brit. 573,236*).

Deterioration of fats by enzymes was only a small part of the literature on fat spoilage. Fat oxidases capable of causing oxidation of unsaturated fats and carotenoid pigments were extracted from soybeans and other legumes (Strain—*Acta Phytochim., Japan 15, 9*) and evidence of their presence in bacon was discussed (Reiser—*J. Am. Oil Chemists' Soc. 26, 116*). In poultry fats the oxidase activities from different birds and fat deposits from different parts of the same bird showed large variations (Ellman & McLaren—*Science 110, 591*).

When butterfat was made rancid by the action of microorganisms, moulds produced more free fatty acids than bacteria under identical conditions, but bacteria produced greater quantities of butyric and caproic acids (Richards & El-Sadek—*J. Dairy Res. 16, 46*). Stale flavor components could be successfully removed from milk fats by steam distillation (Whitney *et al.*—*J. Dairy Sci. 32, 383, 701*).

FLAVOR REVERSION SPOILAGE. In a search for the components which cause flavor reversion, low temperature extraction, and double degumming did not alter the heat reversion characteristics of the oil (Taylor—*J. Am. Oil Chemists' Soc. 26, 413*). Material washed from soybean with water and the unsaponifiable did not have the typical heat-reversion flavor. In a preliminary report on a search for the reversion component of linseed oil an isomeric linoleic acid which was formed during hydrogenation was isolated chromatographically (Lemon—*Can. J. Res. 27B, 605*). Dutton (*U. S. Dept. Agr., Bur. Agr. Ind. Chem. AIC 198, 4 pp.*) and Dutton & co-workers (*J. Am. Oil Chemists' Soc. 26, 441*) reviewed the work on the flavor problem. At present the best means of increasing the shelf life of soybean oil was by treatment with citric acid, certain polybasic acids, and certain polyhydric alcohols. It was suggested that these compounds might function as metal removers, thus decreasing the rate of oxidation of the oil.

Physiology and Biochemistry

REVIEWS. The reviews pertaining to this division of the fat literature were found with the following texts: a comprehensive review on metabolism of lipids (Lehninger—*Ann. Rev. Biochem. 18, 191*), importance of fat in human nutrition (Lang & Cremer—*Z. Lebensm.-Untersuch. u. -Forsch. 88, 633*), fatty acid structure and nutritive value (Daubert—*J. Am. Oil Chemists' Soc. 26, 466*), essential fatty acids (Bernhard—*Arch. sci. physiol. 2, 257*), recent work on glycerides in the nutrition field (King—*J. Am. Oil Chemists' Soc. 26, 316*), influence of internal secretion on fat absorption and transport (Verzar—*Arch. sci. physiol. 2, 43*), biological breakdown of fatty acids (Bernhard—*Ibid. 185*), biological oxida-

tion of fatty acids into tricarboxylic cycle and conversion of fatty acids into carbohydrates (Stadie—*Biol. antioxidants, Trans. 2nd Conf. 1947, 51*), tricarbon compounds in metabolism (Täufel—*Angew. chem. 60A, 173*), biological oxidation of fatty acids (Gurin & Crandall—*Cold Spring Harbor Symposia Quant. Biol. 13, 118*), liver fatty acid oxidase system (Lehninger—*Biol. Antioxidants Trans. 2nd Conf. 1947, 65*), biological fat synthesis (Stotz—*J. Am. Oil Chemists' Soc. 26, 341*), lipoproteins (Macheboeuf—*Arch. sci. physiol. 2, 137*; Chargaff—*Ibid. 157*; Frazer—*Ibid. 169*), and the role of lipids in blood coagulation (Chargaff—*Ibid. 269*).

IMPORTANCE OF FAT IN NUTRITION. The desirability of fat in nutrition was shown in various types of tests. Rats force-fed high fat diets survived longer during fasting than similarly treated rats previously maintained on a high carbohydrate diet (Roberts & Samuels—*Am. J. Physiol. 158, 57*). This appeared to be related to the capacity of a fat regimen to induce continued burning of fat for energy during the fasting, and to the sparing of protein. Rats fed *ad libitum* a diet with no added fat and supplemented with ethyl linoleate gained 29% less than rats fed diets containing eight per cent corn oil or lard (Pearson & Panzer—*J. Nutr. 38, 257*). Rats fed margarine fat or sucrose lived significantly longer at an environmental temperature of $+2^{\circ}\text{C}$., following previous exposure to low temperature, than those fed casein, when these foodstuffs were the sole constituents of the diet (Templeton & Ershoff—*Am. J. Physiol. 159, 33*). At 23° no significant difference in survival time was noted. However, in food consumption investigations of soldiers in a subarctic climate, there was no evidence that these troops had an increased appetite for fats over troops in a temperate climate (Swain—*et al.*—*J. Nutr. 38, 63*).

Tests with isocaloric diets containing 2 to 30% fat showed that fat conferred economy of utilization of food energy to a ration (Swift & Black—*J. Am. Oil Chemists' Soc. 26, 171*; Black *et al.*—*J. Nutr. 37, 275, 289*). Increased weight gains, including increased gains of fat and energy, and decreased heat production were associated with the higher fat diet. In another test with diets low in vitamin B₁, the growth of rats in a pre-exercise and post-exercise period was proportional to the fat content of the diet, but during an exercise period (forced running) the rats practically stopped growing with eight per cent fat in the diet and lost weight proportionally with increases of fat in the diet (Beznak *et al.*—*Hung. Acta Physiol. 1, 35*). The latter disturbance of growth was attributed to an inadequate supply of thiamine in the diet. The isocaloric replacement by lard for one quarter or more of the sucrose in a carious lesion-producing diet of cotton-rats reduced the dental caries attack rate (Shaw—*J. Nutr. 38, 275*).

Certain fat compounds could partially replace biotin for chicks, mosquitoes, and some bacteria (Trager—*J. Biol. Chem. 176, 1211*). This also suggested that biotin must be important for the synthesis of lipids. Intestinal synthesis of biotin in chickens was favored more with diets of 20% fat with dextrin than with five per cent fat with dextrin (Couch *et al.*—*J. Nutr. 37, 251*). An investigation on effect of fats on the microorganisms in the cecal contents of rats was also related to synthesis of B vitamins in the intestines,

for these vitamins may be synthesized by the microflora (Nath *et al.*—*J. Bact.* 56, 783). On sucrose-containing diets the coliform organisms were decreased with high levels of fat, while there was little change in the numbers of lactic group organisms.

The unsaponifiable fraction of yellow bone marrow fat had a beneficial effect on the erythrocyte count of animals (Sandler—*Acta Med. Scand.* 133, Suppl. 225, 72). Vitamin E as wheat germ oil had some therapeutic value for children with a variety of neuromuscular disorders and other disturbances of the nervous system (Stone—*Arch. Pediatrics* 66, 189).

The fact that certain fatty acids are essential in a diet also reflects a desirability for fat in nutrition. The new information on "essential fat acids" was that dehydrated castor oil supplies this factor (Mühr—*Experientia* 4, 355), that these fatty acids promoted the absorption and deposition of fats (Viollier—*Helv. Physiol. et Pharmacol. Acta* 6, 258; Gschaedler & Viollier—*Ibid.* 267; Viollier—*Intern. Z. Vitaminforsch.* 20, 31), and that fat-depleted rats could synthesize some essential fatty acids and make a spontaneous recovery from essential fatty acid symptoms (Barki *et al.*—*Proc. Soc. Exptl. Biol. & Med.* 71, 694).

In one investigation on the desirability of fat in the diet of dairy cows a ration containing 5.2% dietary fat did not increase milk production when compared to a dietary of 2.7% fat (Byers *et al.*—*J. Dairy Sci.* 32, 596). Two communications contained results indicating that inclusion of tocopherols in dairy rations did not influence milk or butterfat yield (Phillips *et al.*—*J. Nutr.* 36, 695; Whiting *et al.*—*J. Dairy Sci.* 32, 133). These results did not confirm older reports to the contrary. One of the above mentioned communications (Whiting *et al.*) showed that feeding five ounces of cod-liver oil daily to cows during the winter feeding period decreased milk fat approximately 14% but increased the total milk production six per cent.

Where intravenous feeding is necessary, it is desirable to include fat emulsions for these concentrated sources of energy combat emaciation, supply calories for growth, and help maintain positive nitrogen balance. In a discussion on the need of fat in intravenous feeding, Stare *et al.* (*J. Am. Oil Chemists' Soc.* 26, 145) gave the method of preparing a typical formula, and listed corn, coconut, and butter oils as suitable fats and soybean phosphatide as a good emulsifier. Geyer *et al.* (*J. Lab. & Clin. Med.* 34, 688) recorded that when using soybean phosphatides as stabilizers for the emulsions the phosphatides must be freshly prepared. Phosphatides on standing in air developed materials which possessed vasodpressor activity in man and the cat but not in the rat or rabbit. Further work by this group (Mann *et al.*—*Ibid.* 699; Gorens *et al.*—*Ibid.* 1627) showed that polyglycerol fatty acid esters were also suitable stabilizers for the emulsions, and that fat emulsions become unsatisfactory for use at approximately four weeks after preparation. Clinical observation on the use of the fat emulsions were presented. The preparation of stable olive oil emulsions in which glycerol monostearate was employed as the emulsifying agent was described and observations on its use for intravenous feeding in dogs recorded (Lerner *et al.*—*Proc. Soc. Exptl. Biol. & Med.* 70, 388). When this group (Lerner *et al.*—*Science* 109, 13) used C¹⁴-labeled tripalmitin as the fat about 50% of the injected labeled

fatty acids were recovered in the liver phosphatides at the end of 24 hours. This was further evidence that parenterally administered emulsified fat pursued a normal metabolic path. Method of preparing combined fat and protein emulsions for intravenous feeding of humans and clinical observation on their use were presented by Shafroff *et al.* (*Proc. Soc. Exptl. Biol. & Med.* 70, 343; 71, 102). These emulsions markedly increased oxygen consumption in human subjects. Acetonuria was occasionally produced.

UNDESIRABLE FATS AND FAT CONSTITUENTS IN NUTRITION. Barnes *et al.* (*Arch. sci. physiol.* 2, 313, 326) comprehensively studied the nutrition of rancid fat. The adverse effect of rancid fat in a diet was inhibited when the diet contained five per cent yeast, liver extract, or 0.5% succinylsulfathiazole. Cod-liver oil could prevent the low liver vitamin A value of rancid fat diets, but could not eliminate the decreased growth. Additions of protein, corn oil, wheat-germ oil, or extra folic acid, biotin, inositol, and *p*-aminobenzoic acid did not reverse the rancid fat effects. It was believed that highly rancid fat destroys some unknown factor which was produced in the intestines.

Although the so-called "essential fatty acids" provide a factor necessary to the nutrition of the rat, they cause diseases in large amounts (Hirsch & Jaquot—*Compt. rend.* 228, 1606). These diseases were offset by adding large amounts of tocopherol to the diets. The addition of oils to chick rations low in riboflavin reduced the efficiency of utilization of the ration and the growth of the chicks (Reiser & Pearson—*Federation Proc.* 8, 242).

The carrier oil or the dietary fat could affect the biological response from vitamin A alcohol or esters (Week & Sevigne—*J. Nutr.* 39, 233, 251). Jojoba oil, ethyl laurate, basking shark liver oil, and mineral oil inhibited hydrolysis of the vitamin A esters. Cottonseed and corn oils also might contain small amounts of factors which interfere with vitamin A absorption. Retarded growth and severe lesions of genital organs and adrenals of mature animals after prolonged administration of fish liver oils were attributed in part to some mechanism involving anti-vitamin E (Cormier—*Bull. soc. chim. biol.* 30, 921). The seborrhea caused in the rat by feeding whale oils was related to the nature and amount of unsaponifiable matter in the oil (Somekawa—*Sci. Papers Inst. Phys. Chem. Res., Tokyo*, 42, 67).

The death rate at 72 hours in rabbits which received at least two cubic centimeters of peanut oil intratracheally was 6.6% (Gobbel—*Am. J. Diseases Children* 77, 175). Toxicity symptoms as outpouring of fluid, erythrocytes and polymorphonuclear leukocytes in the alveolar spaces in these tests was believed due to the presence of free fatty acids in the oil. In general the tests emphasize an absence of marked toxicity of the oil in the rabbit lung. Fatty acid soaps injected in rats produced nervous chronaxia which could be eliminated by administration of a mixture of water-soluble vitamins but not by choline, cystine, or vitamin A (Lecoq *et al.*—*Bull. soc. chim. biol.* 30, 296, 306). These observations were linked with relations between vitamin requirements and metabolism of lipids.

The pharmacological properties of branched chain fatty acids and the fats synthesized from coal and oil was the subject of many communications (Weitzel *et al.*—*Nature* 163, 406; Weitzel—*Angew. Chem.* A60,

263; Ungar *et al.*—*Brit. J. Exptl. Path.* 29, 322; Appel *et al.*—*Z. physiol. Chem.* 282, 220; Thomas & Weitzel—*Ibid.* 180; Schaltenbrand & Schorn—*Deut. Z. Nervenheilk.* 159, 408; Dziewiatkowski *et al.*—*J. Biol. Chem.* 178, 169; Krautwald—*Klin. Wochschr.* 24/25, 493). In general these acids were more or less toxic. Toxic symptoms were most severe with the lower molecular weight synthetic acids. Various degrees of toxic manifestations were evident with presence of odd carbon, the size and position of the side chain, the presence of unsaturation, etc.

Two persons allergic to cottonseed gave no confirmed signs of allergy to ingested cottonseed oil (Bernton *et al.*—*J. Am. Med. Assoc.* 140, 869).

RELATIVE NUTRITIVE VALUE OF FATS. The problem of nutritive value of butter *versus* margarine was kept alive with arguments pro and con as to the superiority of butter. Kentie (*Netherlands Milk & Dairy J.* 3, 182; *Mededeel. Lab. Physiol. Chem. Univ. Amsterdam* 10, No. 8, 92 pp.) favored butter superiority with data that supported his belief that summer butter contained some fatty acid which was a factor in the support of rapid growth of animals. This could not be confirmed by Deuel *et al.* (*J. Nutr.* 38, 361) who tested several fatty acids which have been suggested as being involved in the growth promotion. Lassen & Bacon (*Ibid.* 39, 83) who compared summer butterfat with margarine fat, cottonseed oil, and olive oil for growth of rats also did not confirm Kentie's observations. No significant differences were found between the growth of rats at low environmental temperature on a 20% butter fat ration as compared to similar groups on rations containing 20% margarine fat or various vegetable oils (Ershoff—*Proc. Soc. Exptl. Biol. & Med.* 70, 287). Defatted dry milk reinforced with lard and cod-liver oil was comparable to dried whole milk for meeting growth needs (Schuck & Hanson—*J. Home Econ.* 41, 319). A difference in the calcium assimilation of rats on butter containing rations as compared to a ration containing hydrogenated vegetable oil observed by Dutta (*Ann. Biochem. & Exptl. Med., India*, 8, 137) was attributed to the lack of vitamins A and D in the latter diet.

Other work on butter fat *versus* other fats and oils was connected with manufacture of filled milks to feed dairy calves (Jacobson *et al.*—*J. Dairy Sci.* 32, 429; Jarvis & Waugh—*Ibid.* 665; Barker & Jacobson—*Ibid.* 709; Murley *et al.*—*Ibid.* 609). In general, liquid oils were unsuitable, and hydrogenated oils produced growth and well-being equal or nearly that which occurred when butter oil was used. A patented filled milk dairy calf feed contained glycerides of palmitic, stearic, and higher unsaturated fatty acids in proportions found in milk as the substitute for the milk fat (August & Kleine—*U. S.* 2,472,663).

In an investigation on the relative nutritive value of some tropical oils, papaya seed oil as 10% of the diet of rats gave poor growth in comparison to butter and other oils (Asenjo *et al.*—*Puerto Rico J. Pub. Health, Trop. Med.* 23, 454). Dietary fats which change the unsaturation value of the fish depot fats reduced the heat tolerance of the fish (Hoar & Dorchester—*Can. J. Res.* 27D, 85).

ABSORPTION AND DIGESTION OF FATS. Frazer (*Arch. sci. physiol.* 2, 15, 39) supported the hypothesis on absorption of fats which teaches that absorption of the fats depends on emulsification and dispersion to particles of diameters less than 0.5 μ . With this mech-

anism he postulated explanations for abnormal fat digestions in those diseases which affect the lipase, acidity, and bile salts content of the digestive tract. Molander (*Yale J. Biol. Med.* 21, 201) observed that emulsions of vitamin A in corn or mineral oil with particle size of 0.5 μ were highly absorbed, and that carotene was carried to the liver by corn oil, supported the above mentioned hypothesis. Vitamin A, dispersed in water with the aid of sorbitan laurate, was absorbed three times as fast as when dissolved in corn oil (Popper & Volk—*Proc. Soc. Exptl. Biol. & Med.* 68, 562). The importance of continuous presence of bile for fat digestion was indicated in experiments with bile fistula dogs, who digest fats more completely on one meal a day when given their own bile every hour than when bile was returned every four or eight hours (Searle & Annegers—*Ibid.* 71, 277).

The possibility of relations between absorption of fat and blood fat were investigated. Absorption of olive oil did not cause an appreciable rise in the total lipids of the blood, nor were the rises proportional to the oil absorbed (Glenn *et al.*—*Surg. Gynecol. Obstet.* 89, 200; Gigli *et al.*—*Rass. fisiopatol. clin. e terap.* 20, 39, 48, 53, 88).

Very hard fats, as completely hydrogenated vegetable oils, in addition to being poorly absorbed, increased the fecal excretion of calcium in rats (Calvery & Kennedy—*J. Nutr.* 38, 165). The body was in part compensated for this loss of dietary calcium by a decrease in the urinary excretion of calcium. Similar work was done by Cheng *et al.* (*Ibid.* 37, 237). They found that the presence of calcium and magnesium in the diet markedly decreased the absorption of the higher-melting triglycerides; absorption of these fats was greatest when calcium and magnesium were eliminated from the diet.

Polymerized sardine oil was less digestible than unpolymerized oil (Lassen *et al.*—*Arch Biochem.* 23, 1). It was the polymerized portion of the oil that was not absorbed to any large extent.

The digestibility of rapeseed oil has been found to be 99% in normal men while that of cottonseed oil was 96.7% (Deuel *et al.*—*J. Nutr.* 38, 369). These results demonstrate that a species difference exists in respect to digestibility between man and rat.

INTERMEDIATE METABOLISM OF FATS. The communications on intermediate metabolism concerned many diversified aspects of the field, such as transport, liver lipids, oxidation, deposition, mobilization, etc.

Some insight regarding the deposition of dietary fat in humans was obtained by administering neutral fat labeled with I-131 (Stanley & Thannhauser—*J. Lab. & Clin. Med.* 34, 1634). Proportions of the radioactive iodine collected by the thyroid, excreted in the urine, and dissolved in the serum indicated that degradation of 50 to 73% of the orally administered iodinated fat took place within 24 hours. Similar work with rats on the fate of injected palmitic acid labeled with C¹⁴ at its sixth carbon showed that 35 to 39% of the C¹⁴ was expired as carbon dioxide in 24 hours, considerable amounts of the fatty acid were stored in adipose tissue throughout the body, and that as much as 78% of the radioactive acid recovered from the liver and small intestine had been incorporated into phospholipids (Lerner *et al.*—*Proc. Soc. Exptl. Biol. & Med.* 70, 384). These results demonstrated that the intermediate metabolism was

similar for injected and orally administered fat. Popjak & Beechmans (*Biochem. J.* 44, XXXVI, XXXVII) used labeled hydrogen to demonstrate that fatty acids and cholesterol were synthesized in the rabbit foetus and not derived from the mother by placental transmission. Arterial tissue incubated *in vitro* with labeled acetate and phosphate synthesized phospholipids (Briggs *et al.*—*J. Biol. Chem.* 179, 103, 113).

The communication on fat metabolism in the liver concerned principally agents which modify the deposition of fat in the liver. A study of lipotropic factors derived from the hog pancreas, Dragstedt's lipocaine and Chaikoff's anti-fatty-liver factor exerted lipotropic activity in the liver of dogs with ligated pancreatic ducts, whereas, inositol, choline, methionine, trypsin, chymotrypsin, and carboxypeptidase failed to account for the lipotropic action of either of these (Canepa *et al.*—*Am. J. Physiol.* 156, 387). Another investigation on lipotropic substances showed that lipocaine was very highly active; betaine and triethylcholine were approximately as active as choline; dimethyl sulfide and S-methylisothiouraea were intermediate; and cystine, betaine and S-methylcysteine had slight activity (Raymond & Treadwell—*Proc. Soc. Exptl. Biol. Med.* 70, 43; Treadwell—*J. Biol. Chem.* 176, 1141). A growth-stimulating effect of choline in the tests was attributed to a methionine-sparing action. The sulfur analog of choline was active lipotropically, but toxic at high levels (Maw & du Vigneaud—*J. Biol. Chem.* 176, 1029). Threonine had a little lipotropic activity (Singal *et al.*—*Federation Proc.* 8, 251). Wick & Laurence (*Arch. Biochem.* 20, 113) believed that the lipotropic action of lipocaine on fatty livers of rats receiving diets low in protein and high in fat was due to the choline it contained. The guinea pig was not as sensitive to choline deficiency as other species hitherto recorded (Handler—*Proc. Soc. Exptl. Biol. & Med.* 70, 70). This observation was correlated with the lack of hepatic choline oxidase activity in the animal as compared to that of other species. In chickens ethanalamine had the same activity as choline (Kummerow *et al.*—*Poultry Sci.* 28, 475).

Several diets low in protein and high in fat were fed to rats to observe the effect of the high fat diet on the liver (Hall & Drill—*Proc. Soc. Exptl. Biol. & Med.* 70, 202). The fatty infiltration of the liver was accompanied by a diffuse progressive hepatic fibrosis, but necrosis and scarring which results from a deficiency of sulfur-containing amino acids did not occur.

Endocrine substances influenced deposition of fat in livers. Several estrogenic hormones showed lipotropic activity, whereas progesterone, desoxycorticosterone acetate and testosterone were inactive in this respect (György & Rose—*Arch. Biochem.* 22, 108; *Proc. Soc. Exptl. Biol. & Med.* 71, 552). Adrenocorticotrophic hormone stimulated production of fatty livers (Li *et al.*—*Ibid.* 70, 753; Hartman *et al.*—*Endocrinology* 41, 213). In acute choline deficiency fat also infiltrates in the adrenal cortex. (Olson & Deane—*J. Nutr.* 39, 31). Thyroidectomy in dogs induced development of fatty livers, whereas hypophysectomy did not (Entenman *et al.*—*Endocrinology* 42, 210, 215).

The lipotropic substances were also investigated for their effects on liver phospholipides. Administration of diethanolamine, triethanolamine, ethylethanol-

amine, diethylethanolamine, triethylcholine, and ethylamine stimulated lipid phosphorylation in liver of rats (Cormatzer & Artom.—*J. Biol. Chem.* 178, 775; Artom. *et al.*—*Ibid.* 180, 495). Observations on phosphatide contents of liver and blood of rabbits on various diets indicated that fats were utilized through a phosphorylation step which occurred in the liver (Barbas & Panashchenko—*Fiziol. Zhur. S.S.S.R.* 34, 739; Panashchenko—*Ibid.* 747). Work with labeled phosphorus compounds and hormones showed that those compounds which increase metabolism accelerate phospholipid turnover in the liver; and compounds that slowed metabolism decreased the phospholipid turnover (Flock *et al.*—*J. Am. Physiol.* 155, 402).

Various aspects of biological oxidation and synthesis of lipids were studied. Rat liver homogenates oxidize fatty acids and phospholipids with production of acetoacetic acid from the former but not from the latter (O'Connell & Stotz—*Proc. Soc. Exptl. Biol. & Med.* 70, 675). Octanoic acid, when labeled with C¹⁴ at the seventh carbon and C¹³ in the carboxylic carbon and administered to rats with glucose, was traced to show metabolism through two-carbon fragments arising from β -oxidation. The isotopes entered respiratory carbon dioxide at identical rates but relatively more carboxyl carbon than C¹⁴ appeared in the liver glycogen (Lorber *et al.*—*J. Biol. Chem.* 181, 475). Mice on fat-free diets containing C¹⁴ labeled glucose eliminated 60% of the C¹⁴ as carbon dioxide in 24-48 hours, and the remainder was found principally in the liver and small intestines (Masoro *et al.*—*Ibid.* 179, 1117). This was interpreted to favor the liver as the primary site of synthesis of fatty acids from glucose. In the same series of tests, the finding of appreciable amounts of C¹⁴ in the palmitic acid isolated from depots in rats deprived of both liver and gastrointestinal tract demonstrated that conversion of carbohydrates to fatty acids proceeds in tissues other than the liver and intestines. The analysis of the fat and cholesterol of rats fed a lipid-free diet containing deuterioacetate indicated that 20 and 45%, respectively, of the carbon atoms of these lipids were derived from acetate (Ponticorvo *et al.*—*Ibid.* 179, 839). Another investigator on biological fatty acid synthesis suggested that bone marrow might be capable of effecting conversion of glycine to acetate (Altman—*Ibid.* 177, 985). Acetate was also suggested as the precursor of ruminant milk fat, particularly the short-chain fatty acids (Folley & French—*Nature* 163, 174).

Interesting information on metabolism was derived from *in vitro* studies of oxidation of fatty acids by the enzyme system of tissues. The oxidation of fatty acids by liver slices was not inhibited by carbohydrates, but, in many cases was accelerated (Weinhouse *et al.*—*J. Biol. Chem.* 181, 489). In these tests when acetate and butyrate were used as substrate, ketogenesis was inhibited by the addition of pyruvate. Some tissues from scorbutic guinea pigs oxidized fatty acids less readily than the same tissue from normal animals (Abramson—*Ibid.* 178, 179). *In vitro* ascorbic acid corrected this subnormal oxidation. Another investigation with liver enzymes showed that the oxidation of fatty acids and other compounds was accompanied by esterification of inorganic phosphates, in which two or three atoms of phosphorus were taken up for each oxygen absorbed (Cross *et al.*—

Ibid. 177, 655). Inorganic phosphorus was necessary for the oxidation. The inhibition of oxidation by liver enzyme with surface active agents was correlated with the change in the surface activity irrespective of the nature of the detergent (Hockenhull—*Nature* 162, 850). The discussion on oxidation of fatty acids by rabbit kidney enzymes was presented by Knox (*Biol. Antioxidants Trans. 2nd Conf.* 1947, 70).

Laboratory studies on the effect of anoxia on lipid metabolism showed that acetone bodies in the blood increase slightly, whereas total fat and phospholipids decrease (Sillani—*Rev. med. aeronaut.* 11, 15). Similar work where anoxia was obtained in high elevations confirmed increase of blood ketones, but found the total fat in the blood plasma unchanged (Vladimirov *et al.*—*Fiziol. Zhur. S.S.S.R.* 34, 381). Acclimatization, usually attained after several days, allowed normal fat metabolism.

Considerable differences were noticed in metabolism of saturated and unsaturated fatty acids (Campbell *et al.*—*Biochem. J.* 45, 105). Large amounts of dietary palmitic acid as compared to oleic acid imposed release of large amounts of depot fat, increased liver fat, and decreased liver phospholipids. These results indicate that there occurs more mobilization of depot fat on diets of saturated fats as compared to unsaturated fats. A difference in metabolism of saturated and unsaturated acids might also be indicated in the results of Jones *et al.* (*J. Biol. Chem.* 181, 755) who found that at 1.7 to 1.9% free α -monopalmitin existed in hog pancreas. Other tissues contained very small amounts of this compound. An investigation of unsaturated fatty acid metabolism indicated that rats retain a considerable amount of their polyunsaturated depot fat on fat-free diets; on fat-deficient diets supplemented with corn oil, considerable amounts of tetra unsaturated acids were deposited; and with cod-liver oil more highly unsaturated acids appeared in the tissues (Rieckehoff *et al.*—*Arch. Biochem.* 20, 331). The data showed that considerable amounts of unsaturated acids could be synthesized by the rat.

Mobilization of fat was induced by several means and the process followed by lipid analyses in the blood, liver, and muscle. Thus exercise increased blood fat and sometimes blood phospholipids, whereas in the muscle and liver the fat decreased (Cahn & Houget—*Arch. sci. physiol.* 2, 105). In this series of tests hyperthermia induced by 2,4-dinitrophenol usually led to a drop in the fatty acids and a rise in the phospholipids of the blood, and a rise in both in the liver. In a clinical and biochemical study of the Eskimos, an exclusive diet of pemmican (75% of the calories from beef fat) increased the levels of all lipids in the serum, and a subsequent fast caused further increases in fatty acids, phospholipids and cholesterol in the serum (Sinclair *et al.*—*Federation Proc.* 8, 251). Fasting by Eskimos on customary diets caused increases in serum fat but not increases in phospholipids and cholesterol. The fat increases produced by fasting were not as marked in cirrhotic as in normal animals (Kretchmer & Barnum—*Proc. Exptl. Biol. & Med.* 70, 153). Injuries such as hepatectomy, laparotomy, scalding, injection of salt, or a vesicant increased the lipid constituents of whole plasma of animals (Chanutin & Gjessing—*J. Biol. Chem.* 178, 1). Nervous and hormonal factors affect mobilization of fats (Clement—*Arch. sci. physiol.* 2,

95). All fractions of blood increased on removal of both kidneys in dogs (Díaz & Castro-Mendoza—*Bull. Inst. Med. Res. U. Madrid* 1, 1). Under this condition daily injections of pig kidney extracts prevented the rise in blood lipids. It was postulated that the kidney contains an active agent which inhibits lipid mobilization.

FAT IN DISEASE. Eleven children with nephrotic syndrome and lipemia were treated with one or more lipotropic substances (Wang *et al.*—*Federation Proc.* 8, 264). Inositol, choline, and a combination of these lowered lipids in one or two patients out of five; methionine lowered lipids in four out of five patients; and none of the lipotropic substances produced a change in the protein fraction of the blood. Experimental results on dogs indicated that choline could be used in managing clinical cases of fat embolism (Monson & Dennis—*Proc. Soc. Exptl. Biol. & Med.* 70, 330). Amyl nitrite was also suggested for control of fat embolism (Fahr—*Arch. path. Anat. Physiol.* 314, 499).

High fat diets reduced the diabetic symptoms of alloxan diabetic rats (Abelin—*Helv. Physiol. Pharmacol. Acta* 7, 79; Bornstein & Nelson—*Med. J. Australia* 1, 121). The acceleration of induced tumor formation in rats by high fat diets could be inhibited by restricting the caloric intake (Dunning *et al.*—*Cancer Res.* 9, 354).

Rossini (*Russ. studi psichiat.* 38, 8) found that the iodine number of blood lipid was affected in some nervous diseases. Delayed emptying time of the stomach in many diseases was attributed to decreases in gastric lipase (Veghelyi—*Orvosok Lapja Nepegszegugy* 3, 590). Intestinal fat absorption in the diarrhea of patients with Addison's disease was practically normal (Diaz *et al.*—*Bull. Inst. Med. Res. Univ. Madrid* 1, 211).

In a discussion on treatment of obesity and leanness, Gastineau *et al.* (*J. Am. Med. Assoc.* 139, 86) considered the conditions as disproportions between caloric intake and caloric requirements; various endocrine medications and exercise were considered ineffective or inefficient means of disposing of excessive fat.

CHOLESTEROL METABOLISM. The investigations on the physiology and the biochemistry of cholesterol involved its *in vivo* behavior, factors involving changes of cholesterol content of blood and tissues of the body, and the relation of these to health and disease.

Lippi & Argiolas (*Boll. soc. ital. biol. sper.* 24, 773) recorded that the blood cholesterol of healthy persons varied appreciably, all showing three maximums during the day. In another investigation on normal men, no correlation was found to exist between basal metabolic rates and blood cholesterol value (Peeler *et al.*—*Federation Proc.* 8, 365). Mice fed vegetable fats had increased blood cholesterol values and fat depots diminished; whereas, when fed animal fats there was no hypercholesterolemia, and the animals gained weight (Schettler—*Biochem. Z.* 319, 349).

A high cholesterol content and a low phospholipid content of the blood of patients with thromboangiitis obliterans was interpreted to suggest that the adrenal cortex has a role in the regulation of both cholesterol and phospholipid metabolism (Pagliardi & Marossero—*Minerva med.* 39, I, 461). The posterior lobe of the pituitary gland was also involved in the metabolism of cholesterol, for three fractions chromatographically

separated from its extracts, respectively, (a) lowered serum ester-cholesterol, (b) lowered free cholesterol, and (c) lowered both esterified and free cholesterol (Wachtel—*Nature* 163, 254). Insulin decreased both serum cholesterol and fat levels (Harris *et al.*—*Lancet* 257, 283). The above reports were discussed in connection with metabolism of lipids and effects of diseased conditions.

The interpretation of investigations on cholesterolases concerned synthesis and metabolism of cholesterol. The enzyme system from the liver of rats whose blood was removed with physiological salt solution was found to hydrolyze, not synthesize, cholesterol esters (Metzger & Favarger—*Helv. Physiol. et Pharmacol. Acta* 6, C-59). This suggested that cholesterol esters were formed in the blood plasma and not in the liver. Benard *et al.* (*Bull. soc. chim. biol.* 31, 170) also suggested that enzymes in the blood form cholesterol esters, but found that in several liver diseases this activity of blood was considerably lowered. Nieft & Deuel (*J. Biol. Chem.* 177, 143) and Nieft (*Ibid.* 151) extracted enzymes from both liver and intestines of rats that could esterify cholesterol and enzymes that could split cholesterol esters. The esterifying systems required the presence of phosphate ion and a fatty acid source.

New data was developed on modifying blood cholesterol content and on abnormal depots of cholesterol. The hypercholesteremia induced in normal rats by intravenous administration of cholesterol is gradually overcome within 36 hours (Byers & Friedman—*J. Biol. Chem.* 177, 841). The effect of feeding cholesterol to chickens was recorded by Stamler *et al.* (*Am. J. Physiol.* 155, 470). All preparations increased plasma cholesterol, but the cholesterol dissolved in oil caused the greatest increase. Atherosclerosis of the aorta and large vessels was greatest in groups fed cholesterol oil. They suggested that the occurrence of the atheroma might be related to four factors: hypercholesterolemia, disturbance in ratio of plasma lipids, organ lipoidosis, and disturbance in normal tissue cholesterol esterification ratio. Kuntz & Sulkin (*Arch. Path.* 47, 248) fed cholesterol to rabbits and described the lesions produced. This production of atherosclerosis in rabbits by feeding cholesterol was accelerated when kidney damage was induced by pharmacological means (Moses *et al.*—*Federation Proc.* 8, 114). A deficit or excess of vitamin E did not alter blood cholesterol levels, but did respectively produce fat infiltration of the wall of the aorta and deposition of cholesterol in the aorta (Marx *et al.*—*Arch. Path.* 47, 440). The latter was inhibited by high-cholesterol and bile-salt diet. The presence of surface active agents in the diet of rabbits increased the cholesterol content of the blood, but in spite of this hypercholesterolemia the animals had significantly less atherosclerosis (Kellner *et al.*—*Federation Proc.* 8, 359, 360). Intravenous detergents were ineffective in resorption of atherosclerosis produced in this series of tests of cholesterol feeding. The results were interpreted to suggest that the cholesterol-phospholipid ratio in the blood was an important factor in experimental atherosclerosis. Becker *et al.* (*Science* 110, 529) suggested that atherosclerosis development in persons over 50 years of age might be inhibited by administration of lipase or of a detergent with fat meals. They reviewed data from the literature to support this belief. Experimentally pro-

duced atherosclerotic lesions in chickens regressed somewhat with cessation of cholesterol feeding, thus suggesting that there must be a regression mechanism in animals and that the principles of lipid metabolism should be studied for the mechanism (Horlick and Katz—*J. Lab. & Clin. Med.* 34, 1427). Old hens, nephrotic patients, and hypercholesterolemic rabbits had a higher amount of cholesterol extractable with chloroform in their serum than did normal persons and young chickens (Forbes *et al.*—*Proc. Soc. Exptl. Biol. & Med.* 71, 26; *Federation Proc.* 8, 198).

The effect of some fat metabolism modifying agents were determined on cholesterol metabolism and abnormal cholesterol pathology. Oral administration of lipoic acid, vitamin E, and lecithin were without effect on the cholesterol or total plasma lipids of nine patients with hypercholesteremia (Delevett & Bruger—*Arch. Internal Med.* 81, 859). Experimentally produced atherosclerosis in rabbits was reabsorbed in 74% of the animals by subsequently feeding one gram of choline daily for 185 days (Morrison & Rossi—*Proc. Soc. Exptl. Biol. & Med.* 69, 283). Choline and inositol failed to modify atherosclerosis in cholesterol-fed chicks, and actually intensified the hypercholesterolemia induced by cholesterol administration (Bolene *et al.*—*Federation Proc.* 8, 13). Lack of biotin in the diet reduced the deposition and storage of cholesterol in the liver of rats when fed whole egg (Okey *et al.*—*Federation Proc.* 8, 234). Alloxan diabetes retarded the development of cholesterol atherosclerosis in the rabbit (McGill *et al.*—*Ibid.* 8, 361).

MICROBIOLOGY AND LIPIDS. Several communications reported that various fatty acids could be substituted for biotin for the nutrition of various microorganisms (Axelrod—*J. Biol. Chem.* 175, 265; Hodson—*Ibid.* 179, 49; Williams *et al.*—*Ibid.* 177, 739, 745; Tomarelli *et al.*—*Ibid.* 181, 879; Shull *et al.*—*Arch. Biochem.* 20, 227). Surface active agents enhanced this property of fatty acids. The efficiency of the various fatty acids for promoting the growth of microorganisms was recorded. The *cis* forms of unsaturated fatty acids had a smaller stimulating effect on respiration on *Mycobacterium rubrun* and *M. phlei* than did the *trans* forms (Franke *et al.*—*Biochem. Z.* 319, 263). The acids seemed to be attacked near the carboxyl group. The higher fatty acids inhibited oxygen uptake of *Blastomyces dermatitidis* (Levine & Novak—*J. Bact.* 57, 93).

The references to communications on synthesis have already been presented in the first section of this review.

Composition and Characteristics

The communications limited to methods of analysis, composition, or characteristics of fats and oils are being cited in this section of the review. Some papers cited in other sections of this review contain information on methods of analysis, composition, and characteristics as related to the main text of the individual papers. For example, some of the comprehensive treatises on fats and oils cited in the first section contained analyses; the section on deterioration contained references to methods for testing for spoilage and rancidity; the biochemical work cited in the next section contains methods and analyses incidental to that work; and the analytical methods for soap products are included in the detergent section.

FAT ACID COMPOSITION

Oil or Fat Source	Common Saturated Acids			Common Unsaturated Acids			Other Fatty Acids
	C ₁₄ Myristic	C ₁₆ Palmitic	C ₁₈ Stearic	C ₁₈ (-2H) Oleic	C ₁₈ (-4H) Linoleic	C ₁₈ (-6H) Linolenic	
<i>Abrus precatorius</i> seed ¹	—	1.1	4.7	46.0	12.6	18.5	C ₂₀ 5.1, C ₂₂ 4.4, C ₂₄ 2.5
Alfalfa leaf meal ²	—	19.9	—	31.0	16.9	32.2	—
Triglyceride fatty acids	—	13.3	—	36.8	14.7	35.2	—
Phospholipid fatty acids	—	2.8	1.6	57.6	38.1	—	—
Allium cepa seeds ³	0.3-1.0	17.5-24.7	0.4-1.3	42.2-55.3	15.7-18.5	—	C ₂₀ 0.1, C ₂₂ 0.2, C ₂₄ 0.4-0.8, C ₁₈ (-2H) 5.0-8.3, C ₂₂ (-2H) 2.6-9.0
Avocado pear pulp (Argentina) ⁴	1.0	55.1	6.4	31.7	2.3	—	C ₂₀ 0.3, C ₁₈ (-2H) 3.2
Bacury seed ⁵	—	—	—	—	—	—	—
Badhara seed ⁶	—	—	—	—	—	—	—
<i>Gmelina asiatica</i>	—	9.4	19.3	33.0	25.2	—	Ricinoleic 11.0
<i>Benincasa cerifera</i> seed kernel ⁷	—	8.5	4.0	19.2	68.3	—	—
Capeberry fruit coat fat ⁸	—	—	—	—	—	—	—
<i>Myrica coralyolia</i>	47.0	51.8	0.3	0.6	57.1	3.5	C ₁₂ 0.3
<i>Courbonia virgata</i> root ⁹	—	1.7	—	37.7	—	—	—
Dhupa seed ¹⁰	—	—	—	—	—	—	—
<i>Yucca indica</i>	—	9.7	40.7	42.2	2.3	0.5	C ₂₀ 4.6, C ₁₈ (-2H) trace
<i>Dodonaea viscosa</i> seed ¹¹	—	13.1	16.5	23.9	29.8	—	C ₂₀ 5.4, C ₂₂ 2.2, C ₂₄ (-2H) 7.2
Filbert kernels (Barcelona variety) ¹²	—	2.3	1.6	56.2	16.8	—	C ₂₀ 1.4, C ₂₂ (-2H) 20.6, C ₂₄ (-2H) 0.4
Filbert kernels (Du Chilly variety) ¹³	—	0.5	0.8	64.8	15.2	—	C ₂₀ 3.1, C ₂₂ (-2H) 15.3, C ₂₄ (-2H) 0.0
<i>Ipomoea muricata</i> seed ¹⁴	—	7.9	15.3	20.4	37.4	14.7	C ₂₀ 4.6
Italian millet seed ¹⁵	—	—	—	—	—	—	—
<i>Sataria italica</i>	—	10.6	14.0	20.8	36.4	6.1	C ₂₀ 6.3, C ₂₂ 1.2
<i>Luffa aegyptiaca</i> seed kernel ¹⁶	—	9.5	7.4	40.5	42.6	—	—
Mustard seed (Indian) ¹⁷	—	—	—	—	—	—	—
Black	0.8	0.7	0.4	20.7	18.0	6.5	C ₂₀ 0.5, C ₂₂ 2.3, C ₂₄ 1.8, C ₃₀ (-2H) 8.1, C ₂₂ (-2H) 40.6
Yellow	0.4	1.5	0.4	22.0	14.2	6.8	C ₂₀ 0.5, C ₂₂ 2.0, C ₂₄ 1.0, C ₃₀ (-2H) 7.0, C ₂₂ (-2H) 44.2
Patana fruit pulp ¹⁸	—	—	—	—	—	—	—
Oenocarpus batava	—	—	—	—	—	—	—
Rape seed ¹⁹	0.6	1.0	0.2	79.2	8.8	—	C ₂₀ 0.4, C ₂₂ 2.6, C ₂₄ 1.2, C ₃₀ (-2H) 3.1, C ₂₂ (-2H) 50.4
Seal (common) ²⁰	—	—	—	—	—	—	—
Blubber	2.2	10.6	4.4	—	33.7(-2.4H)	—	C ₂₀ 0.3, C ₁₄ (-2H) 2.2, C ₁₆ (-2.1H) 20.8, C ₃₀ (-7.2H) 13.6, C ₂₂ (-11.0H) 12.2
Liver	0.2	11.4	7.8	—	27.9(-2.4H)	—	C ₂₀ 0.1, C ₁₆ (-2.0H) 8.6, C ₂₀ (-6.2H) 23.7, C ₂₂ (-11.0H) 20.3
Sheep (body) from India ²¹	2.9	27.8	27.7	33.0	3.4	—	C ₂₀ 1.5, C ₁₄ (-2H) 0.4, C ₁₆ (-2H) 2.7, C ₃₀₋₃₂ (Unsat.) 0.6
Soybean (data on 20 varieties) ²²	—	16.4-19.2	—	11.4-22.1	49.3-58.6	6.2-8.5	C ₂₀ (-8H) 0.01-0.09
Tangerine seed ²³	—	19.6	5.2	22.5	46.6	2.1	C ₂₀ 1.1, hydroxy acids 2.9
Wheat germ (of Argentina) ²⁴	—	19.7	—	21.6	41.6	9.2	—

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For increased convenience of presentation some analytical data are tabulated in charts appended to this section of the review.

Some communications are reviews, compilations of data, discussions of composition and methods, or standards which are being merely cited, giving the scope of the text. Lovern (*Ann. Rev. Biochem.* 18, 97) selectively reviewed the work published in the second half of 1947 and the first nine months of 1948 on analytical methods, structure of lipids, and lipid protein complexes. Hilditch (*J. Am. Oil Chemists' Soc.* 26, 41) favored the theory that acids of natural animal fats tend to be evenly distributed among the glycerides. He suggested that although random distribution occurs in fats produced by high temperature synthesis, those synthesized by yeast should differ in this regard. A study of various horse depot fats indicated that they are close to the vegetable oils, such as olive and peanut oils, in acid composition (Holmberg & Rosenqvist—*Svensk Kem. Tid.* 61, 89). Winterized horse fat was found suitable as a salad oil without further treatment. In a communication on fish oils, Lovern (*J. Oil & Colour Chemists' Assoc.* 32, No. 345, 113) reviewed the composition, pointing out the general overall pattern exhibited by certain species and special features such as presence of certain uncommon acids in specific species. Analyses of 554 samples of herring oils were recorded (Norw. Fisheries Res. Lab. & Res. Lab. Norw. Canning Ind.—*Tids. Hermetikind.* 35, 209). A review of the milk fats of various animals indicated that striking differences occur; thus the milk fats of goat and sheep were richer in water-soluble fatty acids than those of the cow and buffalo (Sadgopal—*Indian J. Dairy Sci.* 1, 23). Milk fat from the ass, camel, and mare had no butyric acid, and those from the elephant and sow were completely free of volatile fatty acids. Specifications were issued for Brazilian tung oil (Neto—*Anais assoc. quim. Brasil* 7, 89), and British-produced bone grease (*Brit. Standards* 1483, 21 pp.) and tallow (*Ibid.* 1482, 20 pp.). A comparison of Palestinian with Mediterranean olive oils showed that the former were usually of poorer quality (Puffeles—*J. Soc. Chem. Ind.* 68, 219). The yields of oil from different varieties of olives were recorded (Pantaneli & Brandonisio—*Olearia* 3, 77; Castorina—*Ibid.* 88). Excess irrigation in the production of rapeseed lowered quantity but improved quality (Gattorta & Tombesi—*Ibid.* 1948, 567). The composition of the fatty acids of various palm oils was published (Servant & Valantin—*Oleagineux* 4, 16).

Comprehensive and general reports were written also on methods of analysis of fats and oils. The reports of the following American Oil Chemists' Soc. Committees covered developments on standardizing practically all types of fat analysis methods: Uniform Methods Committee (King *et al.*—*J. Am. Oil Chemists' Soc.* 26, 307), Meal Analysis Committee (Hopper—*Ibid.* 353), Commercial Fat and Oil Analysis Committee (Mehlenbacher—*Ibid.* 345), and Smalley Foundation Report (Bates *et al.*—*Ibid.* 337). The minutes of the meeting of the International Commission on Fats and Oils were translated for the American readers by Hoffpauir (*Ibid.* 61, 106, 151, 692). The fat analyses reports of the Association of Official Agricultural Chemists' contained a collaborative investigation of three methods of preparing butter samples (Meuron—*J. Assoc. Offic. Agr. Chem-*

ists' 31, 318) and slight changes in determination of the fat and fat number of bread (anon.—*Ibid.* 32, 85), and the Bellier test (anon.—*Ibid.* 97). The new fat analysis contributions to the Austrian pharmacopeia were reviewed by Jekel (*Österr. Apoth.-Ztg.* 3, 29). The methods of testing drying oils were discussed from the viewpoint of correlation of evaluation data (Houston *et al.*—*J. Am. Oil Chemists' Soc.* 26, 267). Information concerning microchemical determination of most of the chemical constants and constituents of fats were published (Gorbach—*Mikrochemie ver Mikrochim. Acta* 31, 302; Witoszynskyj—*Österr. Apoth.-Ztg.* 2, 260). Hatt (*Australian Chem. Inst. J. & Proc.* 15, 309) critically reviewed various methods for determining the composition of unknown mixtures of fatty acids. The application of statistics to analysis of oils, fats, and surface active agents was reviewed by Sexton (*Ann. Repts. on Progress Chem.* 44, 287).

ANALYSIS OF FAT SOURCES. Quick methods for determining moisture in oil seeds were compared with standard procedures by Francois (*Bull. mens. ITERG* 3, 257). An electronic method, standardized for corn, required development of factors to correct results when applied to other seeds. The use of the oven procedure at 170° gave results within one per cent of those of the standard method. When using infrared rays the distance from the lamp had to be established with each kind of seed to complete the drying in 15 minutes.

A rapid method for determination of fat in copra was based on fine grinding and agitation in a Waring blender during extraction to prevent channelling (Pinto & Enas—*J. Am. Oil Chemists' Soc.* 26, 723). This type of procedure was also designed for fat analysis of safflower and sunflower seeds (Kennedy & Unrau—*Agron. J.* 41, 93). A rapid control procedure for olive pulp was based on the difference in the specific gravity of the pure solvent and a standard solvent-oil extract from a sodium sulfate dehydrated sample (Bonino—*Anales soc. cient argentina* 146, 375). Rapidity in estimation of grease in sewage sludge was obtained by dehydrating with magnesium sulfate and using one extraction with light petroleum (Stephenson—*Analyst* 74, 257). This single operation extracted approximately 97% of the grease, hence a correction was made on this basis. In a single shaking extraction procedure for fat in food, weighing was also eliminated by use of a specially designed solvent evaporation tube (Borisov—*Gigiiena i Sanit.* 13, No. 11, 38). After evaporation of solvent the tube was inclined in a definite position, and the volume of fat was read directly from the calibration on a side tube. Empirical data and equations were published for calibration and control of several fat determinations by refractometric reading on extracts (Jakobey—*Magyar Kem. Lapja* 3, 636). This method when applied to avocados did not give a straight line relationship between ether extractable material and refractive index (Shannon—*Calif. Dept. Agr. Bull.* No. 38, 127). A method for determination of castor oil in lipstick was based on isolation of the fat, acetylation of the free hydroxyl groups, transesterification of acetylated material, and subsequent saponification of the ethyl esters (Newburger—*J. Assoc. Offic. Agr. Chemists'* 32, 658). The cottonseed oil film on tin plate was determined by transferring it to a water surface by repeatedly dipping the sample in water,

and then measuring it by surface balance technic (Donelson & Neish—*Anal. Chem.* 21, 1102).

The lipid extraction work of Neu (*Süddeut. Apoth.-Ztg.* 88, 239) indicated that wide variations were obtained from wax-containing material. This had previously been demonstrated with coffee oil, and in continuing the work in the same plant family various solvents gave the following lipid yields with *Galium verum*: petroleum ether 3.12, ether 6.36, benzene 6.96, chloroform 7.62, ethyl acetate 13.95, acetone 17.16, and alcohol 27.59% extract. The differences were due to the different capacities of the solvents for dissolving waxes and unsaponifiable material.

The acid used in the Babcock milk fat determination was replaced with detergent solutions that release the fat from the protein solution (Schain—*Science* 110, 121). Milk samples in Babcock bottles were first treated with a solution of "Oil Red O" dye and polyoxyethylene sorbitan monolaurate in isopropyl alcohol, a solution of dioctyl sodium phosphate was added, the mixture was heated at 180°F. for five minutes, brought to within the calibrated range of the bottles with hot water, centrifuged, and the fat content read. An investigation on the Rose-Gottlieb butterfat test showed that when ether containing peroxides was used high values were obtained (Muers & House—*Analyst* 74, 85). Removal of peroxides from the solvent by treatment with a zinc-copper couple was recommended. In an investigation on factory losses of butterfat McDowall & McDowell (*J. Dairy Res.* 16, 227) attributed discrepancies of other investigations on such calculations to failure to determine the fat washed out in the wash water. When the quality of the milk fat was to be determined or the effect of lipase in milk measured, solvent extraction yielded more of the free fatty acids with the fat than did churning (Johnson & Gould—*J. Dairy Sci.* 32, 435, 447). To insure most complete extraction of the lower fatty acids, the milk was acidified to a pH of 2.0 with sulfuric acid. A rapid control moisture procedure for butter made use of a free flame for drying and additional rapidity was gained by reweighing while the sample was still hot (Schütz—*Z. Lebensm.-Untersuch. u. Forsch.* 89, 250). Empirical corrections were developed to bring the results in close agreement with those of standard methods. In the standard fat and moisture procedure for cheese, the sand carrier for the sample was replaced by glass beads (Horwitz & Knudsen—*J. Assoc. Offic. Agr. Chemists'* 32, 303).

The methods for determining fat in clinical work were designed to suit the investigations. The use of Mojonnier extraction flasks was considered suitable and rapid for the fat determination in feces (Soderhjelm & Soderhjelm—*J. Lab. & Clin. Med.* 34, 1471). Another simple method comprised acidification, shaking with ether, centrifuging to separate the fat solution, and then determining the fat in the ether (Zuckerman *et al.*—*Ibid.* 282). If fatty acid determinations were desired the fat residue was dissolved in alcohol and titrated. A new method for the colorimetric determination of total esterified fatty acids in human sera was based on the conversion of the fatty acid into hydroxamic acids and the subsequent development of colored ferric salts (Bauer & Hirsch—*Arch. Biochem.* 20, 242). To determine the radio active C¹⁴ in fat acids the fat extraction solution was spread on a piece of lens paper covering one side of an aluminum disk, the solvent evaporated, and the

measurement was made with a Geiger tube (Entenman *et al.*—*Proc. Soc. Exptl. Biol. & Med.* 70, 364).

QUALITY TESTS. The general problem of taking a representative sample from a tank car containing fats was discussed by Freyer (*J. Am. Oil Chemists' Soc.* 26, 408). The application of geometry of the form of cars was the basis for a method proposed by the author.

The interest in determining color of oils dealt principally with spectrophotometric methods. Such a method using readings at 525 and 550 m μ . was recommended for official adoption by the American Oil Chemists' Society (Agee *et al.*—*J. Am. Oil Chemists' Soc.* 26, 312). Presnell (*Ibid.* 13) recommended that the color be evaluated from transmittance values at three wave lengths. Reasonably accurate Lovibond color values for oils were obtained also by using photoelectric tristimulus colorimetry with three color filters (Bronell—*Ibid.* 427).

An accurate method for determination of the acid number of stand oils made use of a 3:1 mixture of toluene and 96% ethanol as the solvent (Commissie Chem. Analysemethoden—*Verfkroniek* 21, 208). The solvent adequately dissolved the sample, and appearance of turbidity during titration could be inhibited by adding more toluene.

The American Oil Chemists' Society Committee on the refining test for oils recommended that tentative methods be established for degummed expeller soybean oils in which the excesses of 12° lye used were 0.15 and 0.20% (James—*J. Am. Oil Chemists' Soc.* 26, 277). A modified centrifugal method, first introduced by Swift & Co. laboratories, was described (Johnson & Bauer—*Ibid.* 435).

The microscopic examination of edible fat preparations for bacteria was improved by addition of detergents to the sample (Lord & Smull—*Food Res.* 14, 241; Capps *et al.*—*Food Tech.* 3, 260). A synthetic detergent made possible the homogenization of samples on mechanically cleaned slides for the microscopic examination.

CHEMICAL CHARACTERISTICS. The work on procedures for determining the unsaturation of oils was principally to determine their limitations, to increase the rapidity, and to make them suitable for specific difficult-to-analyze oils. The Wijs, the Hanus, and these same methods, modified by using small quantities of mercury acetate as a reaction catalyst, were found to be unreliable for oils containing conjugated double bonds (Francois & Bourignon—*Bull. mens. ITERG* 1948, No. 8, 33). Latzenhofer & Ruziczka (*Mitt. chem. Forsch. Inst. Österr.* 1, No. 1, 17) recommended the method of Margosches, Hinner, and Friedman for the drying oils and listed precautions on the details for insuring good results. A new rapid direct titration iodine number procedure made use of a solution of chlorine and mercuric acetate in acetic acid as the reagent, a mixture of chloroform, acetic acid, and alcohol as the solvent, and a saturated solution of helianthine in alcohol as the indicator (Reutenauer & Regent—*Oleagineux* 3, 379). The method was much more rapid than the Wijs method and gave results agreeing with those of the Wijs procedure. Like the Wijs method it gave low results with tung and oiticica oils. The Wijs method was modified for determinations on very small quantities of sample (Phillips & Wake—*Analyst* 74, 306). The micromethods designed for the fat from biological

material were a modified Hanus method (Viollier—*Helv. Physiol. et Pharmacol. Acta* 7, C26) and a method using a dibrominated pyridine sulfate solution as the reagent (Giraut-Erler & Grimberg—*Ann. biol. clin., Paris*, 7, 127). Apparatus was designed for determination of iodine value of oils by microhydrogenation (Ogg & Cooper—*Anal. Chem.* 21, 1400).

The iodine values of both conjugated and nonconjugated drying oils with the Wijs, Hoffman-Green, Kaufmann, and Wolburn methods were recorded (Carrick *et al.*—*Am. Paint J.* 32, No. 11, 72). The most interesting observation on these figures was that the difference between the Wolburn and Wijs values gave an approximate measure of the diene value; *i.e.*, a measure of conjugation of the oils. A critical review on the determination of linoleic and linolenic acids by thiocyanometry was prepared by von Mikusch (*Farbe u. Lack* 54, 270, 296).

The determination of the saponification value of fats was followed potentiometrically to evaluate the influence of variations in details on the results (Englis & Reinschreiber—*Anal. Chem.* 21, 602). Dilution of the ethanol content to as low as 35%, the change in the pK value for the indicator with change in the solvent composition, and exposure of the samples to air for one hour did not significantly alter the results obtained. Elsner (*Pharmazie* 3, 401) favored the use of propyl alcohol in place of ethanol as the solvent for the determination, because it gave clearer solution and the saponification time could be cut in half. Methanol was unsatisfactory because of low solvent capacity and slow rate of saponification.

The hydroxyl value determination was also investigated for the influence of variables on the results (Hawke—*J. S. African Chem. Inst.* 1, 85). This data was used as a basis for designing a modification of the procedure in which a mixture of pyridine and acetic anhydride was the acetylation agent. Johnson (*Anal. Chem.* 20, 777) simplified the hydroxyl determinations with acetyl chloride as the acetylation reagent by using toluene as the solvent and an apparatus comprising a 500-ml. Kjeldahl flask with a finger condenser in the neck.

PHYSICAL TESTS. The new physical data on fatty acids and fats were developed for fundamental and applied purposes. The solubilities of the C₈ to C₁₄ saturated fat acids in water were redetermined, because the literature contained discrepancies in these data (Eggenberger *et al.*—*J. Org. Chem.* 14, 1108). The correlations between molecular weight and solubility in liquid propane already published for fatty acids and their esters was supplemented with similar information on the fatty amines and nitriles (Bogash & Hixson—*Chem. Eng. Prog.* 45, 597). The aim in this work was to develop basic data on usefulness of propane as a selective solvent for fractionation of fatty material. Vapor-liquid equilibrium data on myristic acid-palmitic acid and palmitic acid-stearic acid systems at four millimeters' pressure were developed to serve as a basis for segregating these acids by distillation (Williams & Osburn—*J. Am. Oil Chemists' Soc.* 26, 663). In the distillation of these mixtures the bubble cap column was more satisfactory than a fibrous glass packed column.

Fresh data appeared on the polymorphic properties of fatty material. Baur *et al.*'s (*J. Am. Chem. Soc.* 71, 3363) and Jackson & Luton's (*Ibid.* 1976) new data on the polymorphism of saturated diglyce-

rides and triglycerides failed to confirm the presence of as many forms of these compounds as reported in the past by Malkin. The divergence of Malkin's results from those of the above group was sustained in a new publication recording the existence of five modifications of monounsaturated-disaturated glycerides (Malkin & Wilson—*J. Chem. Soc.* 1949, 369). A new technique of observing transformations of fat to different polymorphic forms was by noting changes in interference color or extinction angle of a crystal under the microscope (Ravich & Vol'nova—*Doklady Akad. Nauk S.S.S.R.* 66, 417). In this work data on stearic acid was recorded.

X-ray and solidification point data on 9,10- and 10,12-linoleic acids were developed to serve as a basis for analyses of mixtures of these acids (Witnauer *et al.*—*J. Am. Oil Chemists' Soc.* 26, 653). The information could define the composition of any mixture to within three per cent. Fundamental x-ray data was also presented on some fatty acid mixtures (Yoshida—*Proc. Imp. Acad., Tokyo*, 18, 377), amides of saturated fatty acids (Wurz & Sharples—*Anal. Chem.* 21, 1446), and silver salts of fatty acids (Vand *et al.*—*Acta Cryst* 2, 398).

The monolayers of several fatty acids were studied to ascertain the effect of various structures (Schneider *et al.*—*J. Physical & Colloid Chem.* 53, 1016). Limiting area increased with increase in double bonds of natural fats; trans-isomers had smaller limiting area than cis-isomers in non-conjugated series; in conjugated acids the trans-isomers formed condensed monolayers; and hydroxy- and ketoisomers also showed distinguishing characteristics in monolayers. The data indicated the possibility of isomerism in some acids which was not reflected in melting point differences. A high film rigidity found in films of mixtures of fatty oils and petroleum oils as compared to that of the individual oils was explained on the basis of formation of duplex-like films (Aron & Frenkel—*Zhur. Fiz. Khim.* 22, 1246).

The surface tensions of eight fatty oils over a temperature range from 20 to 130° were determined (Halpern—*J. Physical & Colloid Chem.* 53, 895). The data was needed in connection with future investigations on emulsions. The interfacial tensions of paraffin and dilute solutions of oleic and stearic acids were also determined over a wide temperature range (Trillat & Brignonnet—*Compt. rend.* 226, 803).

The dielectric behaviors of methyl palmitate (Dryden & Jackson—*Nature* 162, 656) and several fatty alcohols (Hoffmann & Smyth—*J. Am. Chem. Soc.* 71, 431) were determined and the data were related to changes in phases and crystal structure.

COMPOSITION OF FATS. In new methods for determination of polyunsaturated fatty acids in mixtures by isolation as polybromides the empirical nature of the polybromide yields was recognized, and the results were determined by interpolation from standard yield curves from known wide ranges of mixtures (White & Brown—*J. Am. Oil Chemists' Soc.* 26, 133, 385; White *et al.*—*Ibid.* 85). The method was successfully applied for estimation of arachidonic acid in adrenal phosphatides and linoleic acid in concentrates from corn oil, safflower oil, and butter fat. The amides of many unsaturated fatty acids were prepared, and the properties determined to serve as fundamental data for the separation and identification of the acids (Swern *et al.*—*J. Am. Chem. Soc.* 71, 3017). A novel

analytical system was suggested for the analysis of Chaulmoogra oil (Hoi & D-Xuong—*Bull. soc. chim., France, 1948, 751*). The conjugated diethylenic acids were removed by diene synthesis with maleic anhydride, the mono-ethylenic by arylation with phenylmethane and the saturated were separated by means of metallic soaps.

The collaborative investigation on determination of the unsaturated constituents of oil by spectrophotometry before and after isomerization by an American Oil Chemists' Society Committee (*J. Am. Oil Chemists' Soc. 26, 399*) has resulted in the development of a procedure yielding data with only minor variations between individual laboratories. The full procedure, method of calculation, and collaborative results from eight laboratories on soybean, linseed, and cottonseed oils, and lard were published. An apparatus for isomerization of oils before spectrophotometric analysis was designed by Lips & Tessier (*Ibid. 659*). Absorption spectra were used in the discovery of a new fatty acid in stillingia oil (Huang *et al.*—*Ibid. 405*). This component had maximum absorption at 2600 and an inflection at 3100Å.

The other work with spectral analysis methods concerned the development of new compounds during processing of oils. A comprehensive study on the subject showed that the influences of conjugation and oxidation were evident between 220 and 320 m μ .; at 320 to 500 m μ . removal of pigments by refining and bleaching may be observed; and by absorption above 500 m μ . all characteristic absorptions were attributed to pheophytin A (O'Connor *et al.*—*Ibid. 710*). Similar work was done combining spectrophotometry with chromatographic fractionation (Holm & Wode—*Arkiv. Kemi, Mineral. Geol. 26A, No. 29, 10 pp.*). Peroxide adsorption on alumina and spectrophotometric observations made possible accurate determination of the peroxide value of fat with low peroxide content. Other observations were that chromatographically absorbed portions of oils contained most of the diene compounds, refining and bleaching decreased dienes and increased trienes, and deodorization decreased both. Swain & Brice (*J. Am. Oil Chemists' Soc. 26, 272*) demonstrated that tetraenoic and trienoic conjugations formed from oxidation products of linolenic and linoleic acids could be differentiated from similar conjugations produced by alkali-isomerization of arachidonic and linolenic acids, respectively, by spectrophotometric examination of the sample after heating in neutral ethylene glycol. The differentiation was based on essentially equal amounts of conjugation being formed from fatty acid oxidation products on heating and alkali-isomerization whereas no conjugation was obtained from arachidonic and linolenic acids on heating in the absence of alkali. The data obtained in this work were discussed from the standpoint of analysis, autoxidation behavior, and structure of oils. A report of spectral investigation on autoxidized methyl linoleate attributed a band at 3430 cm.⁻¹ to —OOH groups associated by hydrogen bridging, and bands at 3467 and above 3500 cm.⁻¹ were attributed to —OH groups (Dugan *et al.*—*Ibid. 681*). A maximum absorption band at about 968 cm.⁻¹ in methyl isolinoleate was associated with the presence of double bonds with *trans*-configuration (Lemon & Cross—*Can. J. Res. 27B, 610*).

Chromatographic adsorption technic was used in various modifications for analysis of fats. Use of a

column of silicic acid with mixtures of furfuryl alcohol and 2-aminopyridine as immobile solvent and hexane as mobile solvent permitted separation of straight chain even-numbered or odd-numbered higher acids from one another but did not effect separation of close even- from odd-numbered acids (Ramsey & Patterson—*J. Assoc. Offic. Agr. Chemists' 31, 441*). Holman & Hagdahl (*J. Dairy Sci. 32, 700*) suggested use of this type technic for the analysis of butter fat. Riemschneider *et al.* (*J. Am. Oil Chemists' Soc. 26, 371*) used the technic to isolate pure natural linoleic and linolenic acids and recorded their chemical, physical, and spectrophotometric characteristics. Adsorption of tall oil from petroleum ether on alumina and elution with ether resulted in an eluate containing the neutral components, excepting phytosterol (Jensen—*Finnish Paper Timber J. 31, 225*). The values for neutral components so determined compared fairly well with those of standard methods. In work on removal of free fatty acids from perilla oil by passage through alumina, the adsorbate after neutralization and re-adsorption on alumina showed partition in six fluorescent zones when observed under ultraviolet light (Tischer & Tögel—*Z. physiol. Chem. 282, 103*). The first two zones were identified as mono- and diglycerides, the third as di- and highly unsaturated triglyceride, next appeared triglycerides of increasing order of saturation, sterol esters, and finally waxes and carbohydrates. Direct chemical methods were superior to chromatographic adsorption for determination of neutral substances in oils and fats of high acidity (Balestrini—*Olearia 1948, 547*).

Efforts were made to make potentiometric titration technics useful for analysis of fatty materials. Wolff (*Oleagineux 3, 607*) believed that if the titrations were made in organic solvents, useful applications could be devised for investigations on synthetic detergents and soap. His tests indicated that the acidimetric properties of various fatty acids were too close for use as distinguishing characteristic. Harva & Ekwall (*Acta Chem. Scand. 2, 713*) estimated individual C₁₀ to C₁₈ saturated "even" fatty acids potentiometrically by titrating with silver nitrate solution when the mixture contained two components, and if successive homologues were not present three components could be determined. A comparison of potentiometric and conductometric analysis for fats, oils, and soaps indicated that the latter could be more readily applied (Maron *et al.*—*Amal. Chem. 21, 691*).

Wick (*Ibid. 1511*) cautioned that in work on comparing the radioactive components of fat with other material for biological investigations, the physical form and density should be comparable in order to obtain reproducible results.

A procedure for the determination of the amount of α -monoglyceride in oils based on oxidation with periodic acid was published (Musumi—*J. Nippon Oil Technol. Soc. 2, Nos. 2/3, 69*).

Hopkins *et al.* (*Can. J. Res. 27B, 35*) isolated *cis*-*n*-11-eicosenoic acid from cod liver oil. Other workers had only found the 9-isomer in this oil. Schnette & Baldinus (*J. Am. Oil Chemists' Soc. 26, 530*) confirmed the presence of C₂₀, C₂₂, and C₂₄, alcohol and acids in candelilla wax. Kaufmann & Keller (*Chem. Ber. 81, 152, 159*) detected the presence of acids containing conjugated double bonds in various natural fats and suggested that conjugated bonds may be of biological significance in the synthesis of fats from

carbohydrates in seeds and in the breakdown of the fat on germination.

Several communications on composition of fats dealt with the components other than the fatty acids and glyceride-fatty acid esters. Gossypol was determined in cottonseed oil by extraction with acetone and colorimetric measurement of the reaction between the gossypol and *p*-anisidine (Pons & Guthrie—*J. Am. Oil Chemists' Soc.* 26, 671). Cholesterol was determined in sera by extraction with chloroform and development of color according to the Lieberman-Burchard reaction (Kingsley & Schaffert—*J. Biol. Chem.* 180, 315). Cholesterol in fats was determined by adsorption on alumina from carbon tetrachloride, and elution with chloroform (Kruckenberg—*Z. physiol. Chem.* 283, 68). In an investigation on the lipids of fish, a lower content of cholesterol in the arctic fish as compared to nonarctic fish was correlated with the resistance of the fish to freezing injuries (Wilber & Del Pomo—*Proc. Soc. Exptl. Biol. & Med.* 72, 418). The lipoprotein particles of yeast cells contained 22 to 26% lipid material (Nyman & Chargaff—*J. Biol. Chem.* 180, 741). Data were recorded on the various lipids present in brains of infants and adults (Johnson *et al.*—*Biochem. J.* 44, 494), in the peripheral nerves of animals (*Ibid.* 43, 578), and tissues of the dog (McKibbin & Taylor—*J. Biol. Chem.* 178, 17).

A new tetracyclic diethenoid alcohol was isolated from shea nut fat (Heilbron *et al.*—*J. Chem. Soc.* 1949, 444).

The amount of sulfur in the seed and oil from several oil-producing plants was recorded (Andre & Kogane-Charles—*Ann. agron.* 18, 462). With solvent-extracted oils some sulfur was lost during the evaporation of the solvent. The newly published analytical procedures for metallic impurities in fats were a colorimetric procedure for determination of iron (Kuthanova—*Chem. Obzor* 24, 69), and polarographic procedures for nickel and copper (Korshunov & Kirillova (*Zavodskaya Lab.* 14, 498).

In work on vitamins in fishery products at the Fish and Wildlife Service Laboratories the efficiency of solvents for extraction of the oil from livers was evaluated (Sanford & Manalo—*Com. Fisheries Rev.* 11, No. 2, 18). The spectroscopic E value ratios for the liver oils of various fish were being compiled to serve for evaluation of results obtained by the spectrophotometric method of vitamin A analysis (Sanford *et al.*—*Ibid.* No. 3, 19; No. 9, 11). The vitamin A in the liver of Alaska fur seal was too low for commercial use as a source (Sanford *et al.*—*Ibid.* No. 4, 9). The seasonal vitamin A and D potencies of the liver oils of Pacific cod were recorded (Sanford & Nilson—*Ibid.* No. 3, 13). The vitamin A content and its seasonal variation were compiled for the liver oils of fish of Indian waters (Das *et al.*—*Proc. Indian Acad. Sci.* 29B, 13).

The various methods of assaying for vitamin A were standardized in terms of a new U.S.P. vitamin standard and the international standard (Ellenberger *et al.*—*J. Nutr.* 37, 185; Chilcote *et al.*—*Anal. Chem.* 21, 1180). A report on determination of vitamin A in whale liver oils favored the activated glycerol dichlorohydrin method (Braekkan—*Ibid.* 1530). Kitol and some closely related compounds did not interfere with the reaction. Other reports on whale oil analysis dealt with chromatographic adsorption technic. Barua & Morton (*Biochem. J.* 45, 308) extracted the oil with

alcohol, adsorbed the extract on alumina, and selectively extracted the column to get a clean separation of vitamin A and kitol for evaluation of the vitamin A spectroscopically. A similar procedure attained separation of kitol from the vitamin A by segregating the vitamin A zone in a chromatographic column containing the adsorbed concentrate (Gridgeman *et al.*—*Analyst* 73, 662). Both spectrophotometric and antimony chloride procedures for vitamin A in margarine were slightly revised to eliminate weaknesses found in collaborative investigations of the procedures (Wilkie—*J. Assoc. Offic. Agr. Chemists'* 32, 455).

IDENTIFICATION OF FATS. A new method for estimating butter, coconut oil, and palm kernel oil in margarines was a modification of the Bolton, Richmond, and Revis graphical method in which Reichert, Polenske, and Kirschner values were determined, and the composition was interpolated from relations of these values to curves of values determined on known mixtures (Williams—*Analyst* 74, 508). The measurement of rate of action of castor-bean lipase on samples was recommended as a criterion for the detection of adulterant fats in butter (Vitali & Caccia-Bava—*Ann. chim. applicata* 39, 121). Alfa butter could be distinguished from churned butter microscopically by the shape of the globules and crystal structures in the globules, and macroscopically alfa butter showed a smooth-cut surface, whereas churn products were crumbly and difficult to cut (King & Fritz—*Milch-wissenschaft* 3, 2, 36). Analytical data on the Indian butterfat product "ghee" and its most common adulterants indicated that adulteration could most often be detected from the molecular weight (Phatak *et al.*—*J. Univ. Bombay* 17A, No. 24, 29).

Several adulterant detection tests for olive oils were investigated. The component of the Pitelson color reaction which permits the detection of tea seed oil in olive oil was isolated and named theasin (Hadorn & Jungkunz—*Mitt. Gebiete Lebensm. Hyg.* 39, 259). Tea seed oil contains 0.2 to 0.3% of theasin, whereas olive oil contains 0.03%. A new procedure for detecting adulteration of olive oil depended on the determination of squalene as squalene hexahydrochloride (*Ibid.* 40, 61). The squalene content of olive oil was approximately ten fold that of other vegetable oils except Brazil nut oil which was intermediate between olive and other vegetable oils. The method of Bellier for detecting peanut oil was adapted to detection of adulteration of African olive oils with peanut oil (Blanc—*Ann. fals. et fraudes* 41, 362). In a modification of the Bellier method, methanol was substituted for ethanol in the saponifying solution (Lacerda—*Rev. soc. Brasil Quim.* 16, 153). This report also listed Bellier values of Brazilian oils. A new method for detecting peanut oil in olive oil was similar in principle to the Bellier method in that the turbidity point of the free fatty acids was the criterion (Spiteri—*Chim. anal.* 31, 196).

A test for detection of tung oil in poppyseed, linseed, and rape oils comprised adding one drop of concentrated sulfuric acid to 10 drops of sample (Thymian—*Pharmazie* 4, 140). A typical agglomeration to a firm black mass with a raised indented periphery occurs in the presence of tung oil. The examination with Wood light of the green fluorescences produced in the alcohol extract of hydrochloric acid-treated linseed oils and comparison with stand-

ards made possible classification of samples into first, second, and third pressings, and solvent extracted oils [Foschini—*Chimica (Milan)* 4, 265].

Methods for determination of mineral oils in edible fats were based on chromatographic separation of the hydrocarbons from the unsaponifiable material Hadorn & Jungkunz—*Mitt. Gebiete Lebensm. Hyg.* 40, 96; Williams—*J. Assoc. Off. Agr. Chemists'* 32, 668).

Detergents

MANUFACTURE. The innovations in continuous soap making dealt principally with improvements in systems developed in previous years. The improvements in the continuous process of Joseph Crosfield & Sons, Ltd. (*Brit.* 605,995, 612,014, 623,224) were equipments for emulsifying the fatty raw material, for continuously washing soap curd, and for separating niger and molten neat soap. The latter process was based on slow travel of the soap through troughs containing weirs. The use of caustic and non-caustic alkali and of reaction temperatures as high as 450°F. were the improvements in another continuous system (Clayton—*U. S.* 2,483,002). In the Sharples Corp. system, continuous centrifugal separation of soap and aqueous phases was most suitable when the degree of fat saponification was over 85 or below 70% (Sharples Corp.—*Brit.* 608,943). Between 70 and 85% emulsions too difficult to separate centrifugally existed. A continuous system patented by Bradshaw (*U. S.* 2,452,724-5) contained the steps of catalytic ester interchange of the fat with methanol and saponification of the methyl esters. A French continuous soap system was improved with invention of a new alkali-control device (Lachampt—*U. S.* 2,485,205), and of means for continuously washing the soap with salt solution (Lachampt—*U. S.* 2,485,204). In connection with this system of soapmaking Lachampt *et al.* (*Research, Surface Chem. Suppl.* 1949, 165) recorded the rates of saponification with caustic soda that take place in water-in-oil emulsions. The rapidity of the Mazzoni continuous soap making was increased by use of improved mixers (Mazzoni & Mazzoni—*Ital.* 428,584).

Modifications in batch soap making processes were made for various purposes. Rapidity of saponification was gained in the cold process by mechanically emulsifying the reaction mixture (Razis—*Brit.* 605,653). In two new processes, saponification was carried out under conditions of operational sequence, temperature and pressure, and in the presence of organic solvents so that the soap was produced in powdered form without the necessity of mechanical grinding (Tabor *et al.*—*U. S.* 2,469,753; Holmberg—*Swed.* 120,961). When equal quantities of coffee oil soap were added to a wool-fat soap emulsion the cholesterol and other unsaponifiable matter separated and could be skimmed off (Szücs—*Swiss* 223,076). Another method of removing unsaponifiable matter from wool-fat soap was by extraction with alcohol (Bottaro—*Ital.* 421,630). Hydrocarbons were removed from tall oil soap solutions by distillation (Gayer—*U. S.* 2,492,038). A method of making tall oil soap of reduced unsaturation included a step in which the soap was heated at 230-300° with 100% excess alkali (Segessemann & Molnar—*U. S.* 2,481,356). Oxidized palm oils of peroxide value 125 yielded soaps which could not be grained out, but gave jellies which retained all added salt (Allard *et al.*—*Bull. mens. ITERG* 3, 73). Non-irri-

tating soaps were made from coconut fat when the fatty acids of less than twelve carbon atoms were removed (Dreger & Ross—*U. S.* 2,462,831).

Holmberg (*Svensk Kem. Tid.* 60, 267) recorded analytical data on the fractionation of soap during formation of different layers when in the kettles. Acids up to 16-carbon atoms were increased in the lye and nigre. Higher acids were increased in the neat.

The literature on synthetic detergents contained a discussion on making polyoxyethylene derivatives of fatty acids derived from oxidized paraffin (Griesinger *et al.*—*J. Am. Oil Chemists' Soc.* 26, 241) and a record of experiences on sulfonating Indian vegetable oils (Naik & Desai—*J. Sci. & Ind. Res. India* 7B, 193, 195).

Many communications on synthetic detergents were new patents which are listed here under the assignee or patentee for the convenience of presentation:

Allied Chem. & Dye Corp.

Stabilized alkylaryl sulfonates (*U. S.* 2,469,376-8)

American Cyanamid Co.

Quaternary ammonium compounds (*U. S.* 2,459,062, 2,459,088). Unsaturated guanamines (*U. S.* 2,461,943). Acylguananylthioureas (*U. S.* 2,463,819). Polyoxyethylene esters of polymeric acids (*Brit.* 621,104).

Applicazioni Costruzioni Tec. Antigas

Solid sulfonated castor oil product (*Ital.* 431,953).

Atlantic Refining Co.

Polyisopropylbenzene sulfonate (*Can.* 453,974).
Cyclic nitrogen base sulfonate (*U. S.* 2,472,583).

Atlas Powder Co.

Mixed alkylaryl and polyoxyethylene-tall oil ester sulfonates (*U. S.* 2,469,493).

N. V. Bataafsche Petroleum Maatschappij

Decolorized sulfonates (*Dutch* 63,505).

Bersworth, F. C.

Special mixture of synthetic and inorganic detergents (*U. S.* 2,474,412).

Botson, R.

Alkylaryl sulfonates and sulfo-halogens (*Belg.* 475,010).

Bray, U. B.

Desalted petroleum sulfonates (*U. S.* 2,453,690).

California Res. Corp.

Alkali salts of sulfonated phenyl-substituted alkanes (*U. S.* 2,477,383).

Chemische Fabrik G. Zimmerli A.-G.

Condensation product of cetyl chloromethyl ether and dicyanodiamide (*Swiss* 246,253).

Ciba, Ltd.

Fatty acid amides and their derivatives (*Brit.* 611,215, 615,665, *Swiss* 238,329-30, 238,440, 239,207, 241,142-4, 242,153, 242,782, 243,597, 244,507, 245,276-9, 246,668, 247,918-19, 248,209, 249,001-4, 252,068-71, 256,763-7, *U. S.* 2,448,125). Salts of amino esters (*Swiss* 238,948-50, 240,104-7, 243,331, 246,418-21, 253,256). Dicaprylyl derivatives (*Swiss* 238,441-3, 243,598). Esters of hydroxycarboxylic acids and cycloaliphatic alcohols (*Swiss* 250,061). Sulfonated hydrazide derivatives (*Swiss* 243,596, 248,683). Condensed products of fatty acid mercaptans and various organic compounds (*Swiss* 247,984). Quaternary nitrogen

- compounds (*Swiss 249,005-7*). Sulfonated ethers (*Swiss 242,834, 247,920*). Condensation product of eymene sulfonate and sodium formaldehyde-sulfoxylate (*Swiss 253,011*).
- Chlorox Chem. Co.
A mixture of inorganic and synthetic detergents (*U. S. 2,471,645*).
- Colgate-Palmolive-Peet Co.
A mixture of mineral oil and fatty sulfonates (*U. S. 2,462,758*).
- Duncan, G. W. & Zimmer, J. C.
Desalted sulfonates (*U. S. 2,480,638*).
- E. I. duPont de Nemours & Co.
Aliphatic sulfonate (*U. S. 2,462,341*). Alkyl substituted aromatic sulfonates (*U. S. 2,467,130-2, 2,467,170*). Condensation products of citric acid and fatty alcohols (*U. S. 2,473,460*).
- Eastman Kodak Co.
Fatty esters of phosphoric acid (*U. S. 2,466,393-4*).
- J. R. Geigy A.-G.
Sulfonated, condensation products and other derivatives of amide, amine, biguanide, and urethan derivatives (*Brit. 604,351, Swiss 226,853-4, 230,904, 231,697, 232,132, 232,277-84, 234,350, 234,581, 235,189-90, 238,944-6, 239,000, 239,478-80, 240,218, 240,353-9, 244,766-9, U. S. 2,447,175, 2,451,432*). Hydrazine derivatives (*Swiss 220,926, 242,985, 243,098, 244,579, U. S. 2,473,475*). Fat-aromatic quaternary ammonium compounds (*Swiss 245,642*). Hydrogenated stilbene derivatives (*Swiss. 237,393*).
- General Mills, Inc.
Beta-amino propionates (*U. S. 2,468,012*).
- Hanssens, G. & Hanssens, R.
Soap and sulfonated detergent mixture (*Belg. 476,465, 476,488-9*).
- Hyalsol Corp.
Quaternary nitrogen compounds (*U. S. 2,445,319*).
- Houghton & Co.
Detergent and polyethyleneglycol ester blends (*U. S. 2,471,945*).
- Imperial Chemical Industries, Ltd.
Sulfated ethanolamides of fatty acids (*Brit. 573,524*).
- Kester, E. B.
Sodium fatty glutamates (*U. S. 2,463,779*).
- Malkemus, J. D.
Mineral oil sulfonate and 1-(β -hydroxy ethyl)-piperazine mixture (*U. S. 2,491,992*).
- Matsumoto, T.
Whale oil fatty acids condensed with naphthalene or phenol (*Japan 158,765*).
- Mitchell, J. E.
Mineral oil sulfonate (*U. S. 2,480,592*).
- Montclair Research Corp.
Quaternary ammonium compounds (*U. S. 2,474,202*).
- National Drug Co.
Benzoates of 2-dialkylaminoethyl 2-hydroxyethyl sulfide (*U. S. 2,458,823*).
- Novag, A.-G.
Polyhydroxynaphthalene sulfonic acid salts (*Swiss 229,305*).
- Phipps Products, Inc.
Metal-wool cleaning pads impregnated with synthetic detergents (*U. S. 2,483,135*).
- Pure Oil Co.
Sulfonated hydroaromatic compounds (*U. S. 2,455,811*).
- Rohm & Haas Co.
Sulfonated condensates of urea, formaldehyde, and fatty alcohols (*U. S. 2,486,459*).
- Sandoz A.-G.
Condensation product of hydroxyethylthiourea and formaldehyde (*Swiss 229,125*). Condensation product of fatty acid amides and sodium salt of 3-chloro-2-hydroxypropane sulfonic acid (*Swiss 254,537*).
- Sinclair Refining Co.
Concentrated mahogany sulfonates (*U. S. 2,461,371*).
- Shell Development Co.
Aromatic sulfanyl sulfones (*U. S. 2,452,949*).
- Soc. Anonyme dite
Aliphatic sulfonic acids (*U. S. 2,460,968*).
- Soc. pour l'ind. chim. a Bâle.
Amide derivatives (*Brit. 573,903, 616,247, 616,694, Swiss 221,923, 222,451-4, 224,857-60, 225,555, 227,291, 230,409, 233,159, 237,621*). Benzimidazole derivatives (*Swiss 221,823*). Stearoylaminobenzo-hydrazide derivatives (*Swiss 230,842*). Reaction products of dipentene, phenol, formaldehyde and hydrochloric acid (*Swiss 231,061*). 2,3-bis-(caproyloxy) propane sulfonic acid (*Swiss 235,563*). Organic hydrazine derivatives (*Brit. 572,954, Swiss 223,538, 225,778, 230,841, 230,843*). Mixtures of aliphatic-aromatic alcohols, soaps and other detergents (*Swiss 234,082*). Reaction products of chloromethylcumene with alkylaryl sulfonates (*Swiss 232,270*). Reaction products of pyridinium chloride and aromatic compounds (*Swiss 226,833-4*). Sodium salt of the reaction product of 1-(4-sulfophenyl)-3-methylpyrazolone with 6-(chloromethyl)-1,2,3,4-tetra-hydronaphthalene (*Swiss 230,679*). Thiourea derivatives of various organic compounds (*Swiss 222,958, 225,357, 233,338*).
- Socony-Vacuum Oil Co.
Sulfonated aliphatic and aromatic compounds (*U. S. 2,448,370, 2,463,497*).
- Standard Oil Development Co.
Sulfonated alkyl aromatic hydrocarbons (*U. S. 2,450,585, 2,467,846*).
- Suter, A.
Reaction product of trisulfuric acid ester of glycerol and coconut oil (*Swiss 256,479*).
- Svenska Oljeslageriaktiebolaget
Diocetyl sodium sulfosuccinate (*Swed. 123,268*).
- Thomas Swan & Co., Ltd.
Amidoamino soap (*Brit. 618,767*).
- Universal Oil Products Co.
Sulfonated alkylated benzene hydrocarbons (*U. S. 2,456,119*).
- Virginia Smelting Co.
Blends of sodium di-tert-butyl-naphthalene sulfonate and cyclic ketones (*U. S. 2,453,022*).
- Wallace & Tiernan Products, Inc.
Alkyl quaternary ammonium derivatives (*U. S. 2,479,850*).

Zovody, S. T., & Podnik, R.

Purified sulfonates (*Brit.* 621,765).

New final mechanical operations were developed to improve the finished detergents. Extruded bar soap had a reduced tendency to develop cracks, when, prior to extrusion, and while in a grainy structure and partially in the neat phase, it was passed through a grid of not less than three-mesh per linear inch (Knoll—*U. S.* 2,484,098). The soap bar stamping operation was expedited when the blank for the press was shaped to conform more closely to the die (Reddert & Sartorius—*U. S.* 2,465,398). To effect savings in packaging, small bars were pressed together to yield a long bar (Schulerud—*U. S.* 2,486,213). These could be easily divided for use. Detergents and water softeners were pressed into disks to expedite dissolving (Livingstone—*U. S.* 2,471,158). The patents on new forms of toilet soaps dealt with manufacture of a concave bar (Haskell—*U. S.* 2,489,639), bars with ingrained emblem (Knoll—*U. S.* 2,473,530), and porous packets providing soap for a single hand washing (Hermanson—*U. S.* 2,470,851). A sponge for cleaning was made from a combination of synthetic rubber and a mixture of synthetic detergent so that it would slowly exude the detergent when in use (Romaine—*U. S.* 2,466,826).

Ueno & Imade (*J. Nippon Oil Technol. Soc.* 2, No. 1, 5) determined the rate of drying of different forms of finished soap. In general, the decreasing order of rate was chips, cube, rectangle, pyramid, ball, and vertical column.

Charts and graphs were developed for soap-synthetic detergent-builder mixtures in hard waters to form a basis for selection of proportions to give effects desired (Morrisroe & Newhall—*Ind. Eng. Chem.* 41, 423). Alkylaryl sulfonate was the synthetic detergent, sodium stearate the soap, but several builders were used in the tests. Similar information was developed for soap-builder-polyoxyalkylene derivatives of fatty and rosin acids (Barker—*J. Am. Oil Chemists' Soc.* 26, 304). In this work improvements in detergency in hard waters were obtained when the polyoxyethylene derivatives replaced part of the soap in products containing carboxymethyl cellulose, sodium carbonate, or phosphates as builders. The use of reaction products of alkali cellulose with monochloroacetic acid to improve the detergent properties of soaps was patented (Oel- & Chemie-Werk A.-G.—*Swiss* 252,999). One investigation on such soaps indicated that the cellulose product did not increase cleansing power but increased the dispersion of the soil in water (Viertel—*Melliand Textilber.* 28, 345). Sodium tripolyphosphate and its thermal decomposition products were patented as builders for sodium salts of sulfonated fatty acid detergents (Byerly—*U. S.* 2,486,921; Strain—*U. S.* 2,486,922).

Many other materials were added to soaps for various purposes. Hydrogen peroxide or an organic hydroperoxide derivative was added to prevent the oxidation of the α -tocopherol in the soap to a pink color (Lange & Folzenlogen—*U. S.* 2,473,154). Lauric acid diethanolamine condensates (Young & Rubinstein—*U. S.* 2,483,253) and alkyl sulfates (Hanssens & Hanssens—*Belg.* 474,416-17) were added to ordinary soaps to inhibit precipitation of calcium and magnesium soaps when the products were used with hard water. A soap for use in the textile industry

contained 10 to 20% of organic solvent (Ind. Saponi Affini Milano ISAM—*Ital.* 421,586). The incorporation of ultramarine as a bluing agent in soap was patented (Nigra—*Ital.* 426,600). The new optical bleaches, i.e., laundry brightening agents, added to soap were *o*-alkoxybenzol derivatives of 4,4'-diaminostilbene-2,2'-disulfonic acid (Eberhart *et al.*—*U. S.* 2,468,431) and 1,2-di-2-benzimidazolylethylene (Ciba, Ltd.—*Swiss* 238,148). The dyes used and method of adding coloring to soaps were discussed by Ruemele (*Indian Soap J.* 14, 165). Comminuted soaps and detergents were rendered nondusting by incorporation or coating with starch, gum or glue (Miles—*U. S.* 2,456,437), hygroscopic alkaline earth metal chlorides (Bodman—*U. S.* 2,465,346), alkali metal silicates (Holuba—*U. S.* 2,480,579), and fatty acid mono-glycerides (Colgate *et al.*—*U. S.* 2,489,955). Patents were issued for use of halogenated 2,2'-dihydroxydiphenylmethanes as soap additives in the manufacture of germicidal soaps (Gump—*U. S.* 2,487,799; L. Givaudan & Cie—*Swiss* 223,543). Sulfonamides were also added to soaps for the same purpose (*Soc. italiana Commercio estero—Ital.* 421,241). Small amounts of guanidine stearate rendered soap less irritating and more easily soluble (Henderson—*U. S.* 2,459,818). Paraffin, waxes, sulfated albumin-fatty acid condensates, methylcellulose, casein, and starch were added to fatty alcohol sulfonates to inhibit their property of withdrawing fat from the skin (Novag A.-G.—*Swiss* 228,769). Soap was stabilized against discoloration and rancidity by the biguanide salt of mercaptobenzothiazole (Cook—*U. S.* 2,467,295). Foaming was inhibited during evaporation of detergents, and in use in dish or laundry washing by addition of phenoxyalkanols (Tremain & Bacon—*U. S.* 2,453,352), or butyltrichlorosilane (Scott—*U. S.* 2,462,999).

Many soap substitutes were made of combinations of inorganic salts. The purely inorganic detergent mixtures were a combination of alkali metal metasilicate and alkali metal polyphosphate (Robinson—*U. S.* 2,473,822), mixtures of the same salts made non-caking with hydrated magnesium phosphates (King—*U. S.* 2,468,448), alkali metal phosphates made noncaking with intimately bound organic detergents (Hafford & Parsons—*U. S.* 2,480,730), a combination of sodium silicate, sodium carbonate, and sodium hydroxide (Beckmann—*Ital.* 432,553), and a mixture of sodium hydroxide and sodium carbonate (Corrigan—*U. S.* 2,463,680). Soap substitutes containing limited amounts of common detergents were a combination of colloidal aluminum hydrate, sodium naphthalene sulfonate, and powdered soapwort roots (A. Wander A.-G.—*Swiss* 226,570) and the product of saponification with excess sodium carbonate of a mixture of beeswax or wool fat, a light hydrocarbon fraction, and oleic acid (Wainstain—*Fr.* 870,353).

Several special cleaners were patented. The new scouring cleansers were a combination of volcanic ash, soda, sodium alkylaryl sulfonate, and sodium carboxymethylcellulose (Bacon & Vaughn—*U. S.* 2,489,848), a mixture of fatty alcohol, borax, and sodium carbonate (Paulissen—*Belg.* 476,880), soap ground with kaolin or fuller's earth (Longchambon & Longchambon—*Fr.* 871,352), and mixtures of sulfated lauric amide, clay, and sodium carbonate, sodium sulfate, or sodium silicate (Chimiotecnic Union chim. du Norde et du Rhone—*Fr.* 868,793). A bottlewashing detergent contained sodium zincate, sodium hy-

dioxide, and trisodium phosphate (Bennett—*U. S. 2,455,648*). Sodium berylonates were added to bottle-washing detergents to reduce scuffing (Burkard & McNabney—*U. S. 2,474,392*), and alkylaryl sulfonates were added to ease removal of labels and increase microbe-kill (Tenney—*Food Inds. 21*, 159). A polishing composition contained soap, petroleum solvent, chalk, ammonia, and water (Fisher—*Can. 455,802*). A metal surface cleaner contained soap, higher fatty acid amines, polyhydric alcohol, and several organic solvents (Borus—*U. S. 2,466,632*).

Many communications on detergents provided information on common practices in the industry, and described raw material, or known products. These are most conveniently tabulated below under the subjects treated:

Raw Materials: Fatty acids, Rowe—*Soap Perfumery Cosmetics 22*, 257, Behrmann—*Soap Sanit. Chemicals 25*, No. 9, 40; tall oil, Ruemele—*Indian Soap J. 14*, 216; synthetic fatty acids, den Otter—*Chem. Weekblad 43*, 495; Schwen—*Angew. Chem. B19*, 72; sodium silicate, Merrill—*Ind. Eng. Chem. 41*, 337; soda ash, Treffler—*Soap Sanit. Chemicals 25*, No. 8, 25; sodium carboxymethyl cellulose, Nieuwenhuis—*J. Am. Oil Chemists' Soc. 26*, 51; whitening agents, Lesser—*Soap Sanit. Chemicals 25*, No. 11, 36.

Manufacture: Modern methods, Seaman—*Soap, Perfumery Cosmetics 22*, 698, Seaman—*Manuf. Chemist 20*, 530, Wells—*Indian Soap J. 14*, 246, Velharticky—*Chem. Obzor 24*, 35; Mon-savon continuous process, Webb—*Soap, Perfumery Cosmetics 22*, 150, 483; powdered soaps, Vallance—*Soap Sanit. Chemicals 25*, No. 3, 37; soft soap, Vallance—*Ibid.* No. 11, 40, No. 12, 44; chemistry and physics of saponification, Paleni—*Olearia 5*, 145.

Descriptions of Special Products: Specialty soaps, Lesser—*Soap Sanit. Chemicals 25*, No. 10, 33; liquid toilet soap, Brit. Standards Inst.—*Brit. Standards 1545*, 6 pp.; scouring powders, Vallance—*Soap, Perfumery & Cosmetics 22*, 158, glass polishes, James—*Ibid.* 149; cleaning compounds, Swan—*Org. Finishing 9*, 19.

Properties and Composition: Comparisons of synthetics vs. soap, Neu—*Seifen-Öle-Fette-Wachse 75*, 4, Slawson—*Soap Sanit. Chemicals 25*, No. 6, 49, Zeising—*Seifen-Öle-Fette-Wachse 74*, 193, 217, 237, Borghetty—*J. Am. Oil Chemists' Soc. 26*, 319, Flett—*Am. Perfumer Essential Oil Rev. 52*, 519, Palmer—*Standardization 20*, 130, chemical structure and performance, Scott & Roebuck—*Chemistry & Industry 1948*, 627; quaternary ammonium compounds, Cucci—*Soap Sanit. Chemicals 25*, No. 10, 129, "Sello-gens," Rao—*Indian Textile J. 59*, 346, non-ionic detergents, Dollinger—*Soap Sanit. Chemicals 25*, No. 1, 37; description of 700 synthetic detergents, McCutcheon—*Ibid.* No. 8, 33, No. 9, 42, No. 10, 40; synthetic detergents, Bright—*Chem. Eng. 56*, No. 9, 136, Morgan—*Ibid.* No. 5, 153, James—*Soap Perfumery & Cosmetics 22*, 272, 491, Chwala—*Mitt. chem.-Forsch.-Inst. Ind. Österr. 1*, 9, Larson—*J. Am. Water Works Assoc. 41*, 315; emulsification and detergency, Courtney-Harwood—*Chem. Products 12*, 162, Schwartz—*J. Am. Oil Chemists' Soc. 26*, 212, Lomas—*J.*

Soc. Chem. Ind. 68, 37; phase factors, Webb—*Soap, Perfumery & Cosmetics 22*, 838; composition of detergents, Lindner—*Seifensieder-Ztg. 72*, 174; specifications for soap, Das Gupta—*Indian Soap J. 14*, 311; evaluation of cleaners, Nielsen—*Org. Finishing 7*, No. 8, 9; testing germicide soap, Traub—*J. Soc. Cosmetic Chemists' 1*, 251; dermatological action of cleansers, Schwartz—*Soap Sanit. Chemicals 24*, No. 5, 33.

Uses: For Laundry, Smith—*Soap Sanit. Chemicals 25*, No. 3, 41, Bartlett—*Ibid.* 139, Harwood & Hill—*J. Textile Inst. 40*, 689; in the worsted industry, Monerief—*Soap Perfumery & Cosmetics 21*, 1114; in the fur industry, Smith—*Soap Sanit. Chemicals 25*, No. 5, 33; for food plant cleaning, Somers—*Food Ind. 21*, 295; for dairy plants, Lesser—*Soap Sanit. Chemicals 24*, No. 5, 40; on-location carpet cleaning, York Res. Corp.—*Soap Sanit. Chemicals 25*, No. 9, 37; in metal finishing, Leffingwell—*Metal Finishing 47*, No. 4, 68; metal cleaning, Reich & Snell—*Soap Sanit. Chemicals 25*, No. 2, 43, No. 3, 45; use as dispersing agents in aqueous and nonaqueous media, Lomas—*J. Soc. Chem. Ind. 68*, 37.

ANALYSIS FOR CONSTITUENTS. The Gerber method for determining the amount of fat in milk products was recommended for determining the amount of fatty acids in soaps (Truddaiu—*Ann. chim. applicata 38*, 338). A procedure for determining the amount of olive oil used in manufacture of pharmacopeia olive oil soap depended on determination of the fatty ingredients, hydrocarbons, and squalen number (Hadorn & Jungkunz—*Pharm. Acta Helv. 23*, 361).

The method for determination of potassium as potassium periodate was applied to the analysis of soap and caustic lye (Miller & Andrews—*J. Am. Oil Chemists' Soc. 26*, 309). The potassium was precipitated as the periodate, reduced, and the liberated iodine was titrated with thiosulfate solution. A semimicro determination of potassium depended on precipitating the sodium from a solution of the soap ash, weighing the precipitate, and calculating the potassium by difference (Blank & Rotolo—*Ibid.* 102).

A procedure for determining free alkali and sodium carbonate in soap depended on titrating a sample to a green color in alcohol solution with thymol blue as the indicator for the free alkali, diluting with equal amount of water and titrating to a yellow color for the sodium carbonate (Wolff—*Oleagineux 4*, 662). A method of determining bicarbonate and carbonate comprised gravimetrically estimating the carbon dioxide liberated by heating and calculating to sodium bicarbonate, acidifying, and calculating the balance of the carbon dioxide to sodium carbonate (Kelley & Blank—*J. Am. Oil Chemists' Soc. 26*, 685).

Pohle *et al.* (*Ibid.* 376) discussed the use of the periodic acid reaction for determining free glycerol in soap. The direct iodometric method was applicable when no material which interferes with the periodic reaction was present; otherwise the glycerol must be extracted from the sample.

A colorimetric method for determination of iron in soap depended on the color development with sodium salicylate and comparison with colors developed from standard solutions (Kathanova—*Chem. Obzor 24*, 69).

Several analytical methods pertained to estimation of non-soap detergents. For approximating the

amount of polyethylene glycol detergents, sulfates were removed with barium chloride and the amount of precipitate obtained by boiling with hydrochloric and phosphomolybdic acids was compared with similar precipitates obtained from known amounts of the same detergent (Oliver & Preston—*Nature* 164, 242). A quantitative test for cationic quaternary ammonium detergents comprised titration with anionic quaternary ammonium detergents in the presence of some water-immiscible organic solvent and dichlorofluorescein as the indicator (Cahn—*U. S.* 2,471,861). For the anionic compound the titration was with a cationic standard solution. A similar procedure made use of pinacyanol bromide as the indicator and was applicable for analysis of many synthetic detergent compounds (Salton & Alexander—*Research, London*, 2, 247). The use of Pontamine Fast Red 8, Lissolamine A, or Fixanol C, as indicators in a like method for determining soap and synthetic detergents, was also described (Wijga—*Chem. Weekblad* 45, 477). In attempts to develop a system for qualitative analyses of many commercial synthetic detergents, many qualitative tests for specific agents and mean of separation were given (van der Hoeve—*Rec. trav. chim.* 67, 649). A quantitative procedure for fatty acid amides sulfonates made use of a scheme for solvent fractionation into free fatty acids, fatty acid amides, fatty acid esters, sulfated fatty acid amides, sulfated fatty acid esters, and sulfated fatty acid fractions and analysis of the fractions (Desnuelle & Micaelli—*Oleagineux* 4, 353). Another scheme for the analysis of commercial detergent mixtures was based on similar technique (Parisot—*Olearia* 3, 13).

PHYSICAL PROPERTIES OF DETERGENTS. Hattiangdi (*J. Res. Nat'l Bur. Standards* 42, 331) recorded the x-ray data for 29 commercial soaps with a view to obtaining information on degree of crystallinity and phase nature and eventually to correlate the data with properties of soap solutions and washing action. Toilet and medical soaps seemed to consist essentially of β -sodium palmitate and to a small extent of ω -sodium oleate; soaps of high laurate-myristate content were of the ω -phase. However, better characterization of the soaps was attained with the electron microscope (Hattiangdi & Swerdlow—*Ibid.* 343). In this work the network of interlaced soap fibers and flagellar phase was described and discussed in relation to x-ray data and phase studies. X-ray observations were made on mixed crystals in soap and mixed micelles in soap solutions and discussed from the viewpoint of interferences caused by different lengths of the paraffin chains in the lattice arrangements (Hess & Kiessig—*Chem. Ber.* 81, 327). The spacings of palmitate, stearate, myristate, and arachidate soap hemihydrates were determined (Minor & Lingafelter—*J. Am. Chem. Soc.* 71, 1145). Part of the data were determined to check values already published, and the remainder to extend the series of this fundamental data to new acids.

Hattiangdi *et al.* (*J. Res. Nat'l Bur. Standards* 42, 361) in a general study on physical properties of soaps discussed the relationship of surface tension, electric conductance, pH, opacity, and foam formation to micelli formation and detergent action. The data was recorded on 30 commercial soaps and six popular soapless detergents. Chandler & McBain (*J. Phys. & Colloid Chem.* 53, 930) determined the diffusion and osmotic coefficients, conductivity, and

micellar charge as related to the concentration of solutions of detergents as a means of studying micellar composition. Data on light scattering in soap solutions was also related to formation of ions and micelles, and structure of the micellar layers (Debye—*J. Physical & Colloid Chem.* 53, 1; *Ann. N. Y. Acad. Sci.* 51, 575; *J. Colloid Sci.* 3, 407; Schulman & Friend—*J. Colloid Sci.* 4, 497; Thiele—*Kolloid-Z.* 112, 73).

X-ray diffraction studies were made on dispersions of organic solvents in aqueous detergent solutions (McBain & Hoffman—*J. Physical & Colloid Chem.* 53, 39; Mattoon & Mathews—*J. Chem. Phys.* 17, 496; Harkins & Mittlemann—*J. Colloid Sci.* 4, 367; Marsden—*J. Am. Oil Chemists' Soc.* 26, 57; Hartley—*Nature* 163, 767). These data were discussed with relation to phase changes, formation of micelles, and shape and surface structure of the micelle. Systems of organic solvent-soap solutions were observed for setting time of gels, syneresis, and gel strength (Hattiangdi & Adarkar—*J. Am. Oil Chemists' Soc.* 26, 364), solubilization of polar and nonpolar substances (Harkins & Oppenheimer—*J. Am. Chem. Soc.* 71, 808), size of the micelles in aqueous soap-benzene systems (Arkin & Singleterry—*J. Colloid Sci.* 4, 537), rate of solution of benzene in sodium oleate solution (Peregudova & Voyutskii—*Kolloid Zhur.* 10, 309), electrical behavior (Ralston & Eggenberger—*J. Physical & Colloid Chem.* 52, 1494), elastic properties (van den Berg & de Heer—*Proc. Koninkl. Nederland. Akad. Wetenschap* 52, 457), and viscosity characteristics (Neiman & Neiman—*Kolloid Zhur.* 9, 432). These data were discussed also with relation to character of the micelles formed. Viscosity and elastic behavior was measured also for aqueous oleate systems containing potassium chloride (de Jong & van den Berg—*Proc. Koninkl. Nederland. Akad. Wetenschap.* 52, 15, 99, 363, 377). These data were presented graphically. The nature of suspension of zein in aqueous detergent solutions as shown by viscosity, birefringence of flow, electrophoresis, and sedimentation indicated that the zein was molecularly dispersed and the detergent was bound tenaciously to the protein (Foster—*J. Phys. & Colloid Chem.* 53, 175). A review of the types of solubilization mechanisms that occur with soap was prepared by Kleven's (*J. Am. Oil Chemists' Soc.* 26, 456).

A study of the solubilities in aqueous potassium laurate-potassium silicate systems demonstrated that liquid soap containing 33% potassium laurate could be mixed in all proportions with potassium silicate solutions containing 38.7% solids (Merrill—*J. Physical & Colloid Chem.* 52, 1143). A depression of conductivity of colloidal detergent solutions with inorganic electrolytes was attributed to the depression in the ionization of the detergent (Ralston *et al.*—*J. Am. Chem. Soc.* 71, 2145).

Testing of solutions of detergents was also by measuring their capacity for dissolving water-soluble dyes (McBain & Huff—*J. Colloid Sci.* 4, 383; Arkin & Singleterry—*J. Am. Chem. Soc.* 70, 3965; Lambert & Busse—*J. Am. Oil Chemists' Soc.* 26, 289; Kolthoff & Stricks—*J. Physical & Colloid Chem.* 53, 424). These data rated some detergents for their solvent capacity for dyes, indicated concentrations under which maximum and minimum colloidal properties existed, indicated the critical concentrations for micelle formation and the effect of electrolytes on these

properties. The dye solubilization by detergents varied widely among different products.

The acidimetric properties of soap solutions of several fatty acids were recorded (Wolff—*Oleagineux* 4, 141). These results were discussed from the standpoint of solubility, hydrolysis, and formation of dimeric ions. Stainsby and Alexander (*Trans. Faraday Soc.* 45, 585) discussed hydrolysis of soap in aqueous solutions with the assumption that mixed micelle formation occurs between fatty acid mols, formed by hydrolysis and soap ions. Reduction in hydrolysis in presence of butyl alcohol was explained on the basis of a simple competition between fatty acid and alcohol molecules for the limiting number of sites in the micelles. An insight into the hydrolysis of soaps was also gained from analyses of foams from soap solutions. The foam from sodium oleate contained a 1:0.876 ratio of soap to free acid [Raison—*Research (Suppl. Surface Chem.)* 1949, 187]. Thus, hydrolysis occurred to enrich the foam with fatty acid and sodium hydroxide was enriched in the solution. Foam fractionation was used to purify certain detergents for surface tension measurements (Brady—*J. Physical & Colloid Chem.* 53, 56; Shedlovsky *et al.*—*J. Colloid Sci.* 4, 25). Reutenauer & Sicard (*Bull. mens. ITERG* 3, 262) recorded surface tension measurements on several pure soap and synthetic detergent solutions.

Harris (*Textile Res. J.* 18, 669), discussed the differences among various fibers for the adsorption of detergents. Cotton did not adsorb enough anionic or nonionic agents to form a monomolecular film whereas cationic agents were rapidly and tenaciously held. Wool adsorbed anionic and cationic agents in amounts exceeding those necessary for monomolecular film formation. The observations were discussed from the standpoint of electrostatic nature and chemical nature of the fibers. In adsorptions on carbon the concentration of the fatty acid component of soaps was always greater than that of the alkali component (Weatherburn *et al.*—*Can. J. Res.* 27F, 179; Reade *et al.*—*Ibid.* 426).

Values for the surface adsorption areas of various soaps were determined as a basis for determining particle size of polymerized latex particles by titration of the soap (Willson *et al.*—*J. Physical & Colloid Chem.* 53, 357).

PERFORMANCE TESTS. Most communications on evaluating the washing efficiency of detergents contained reviews of the tests available for the purpose, discussion of the limitations, and added some original fundamental data useful in testing or suggestions on modifications of the test. Bacon & Smith (*Ind. Eng. Chem.* 40, 2361) evaluated the amount of mechanical work in relation to cleaning efficiency. Utermohlen & Ryan (*Ibid.* 41, 2881) used black iron oxide as the soil and determined the amount of pigment left on pieces of washed cloth as a criterion of detergent capacity. Another test developed in the same laboratory made use of iron oxide and lamp black as the soil with oily and nonoily binders, and it was reported that ease of removal of solid soil and oily soil differed (Utermohlen *et al.*—*Textile Res. J.* 19, 489). A test designed by Powney & Feuell (*Research, London* 2, 331) made use of graphite and liquid paraffin to soil cloth, using a laboratory-type washing machine, and compared the washed cloths for residual dirt photometrically. Leonard & Beck (*Am. Dyestuff*

Reptr. 38, No. 8, 348) recorded the effects of temperature, concentration of detergent, and builder on the efficiency of cleaning wool raw stock. Leonard & Winch (*Rayon & Synthetic Textiles* 30, 79, 93) used various detergency tests to demonstrate the economy and effectiveness of using sodium carbonate as a soap builder. To determine the average reflectance of a number of cloth samples in detergency tests the samples were distributed on a turntable and read while rotating at 78 r.p.m. or faster (Thompson—*J. Am. Oil Chemists' Soc.* 26, 509).

In testing 160 detergents for army sea-water laundering, all but 41 were eliminated because of precipitation in sea water (Vaughn *et al.*—*Ind. Eng. Chem.* 41, 112). The remainder were studied for soil removal, whiteness retention of wash, pH, optimum concentration, optimum temperature of efficiency and loss of tensile strength of fabrics. On the basis of these data four detergents were approved and a new laundering formula was suggested.

LaFleur (*Am. Dyestuff Repr.* 38, 367) evaluated the wetting efficiency of 21 wetting agents by determining the sinking time of wool cloths in solutions of the agents. A new test for evaluating the wetting properties of surface-active agents was based on the time required for solutions of the agent to pass through the filter element of porous, sintered stainless steel filter crucibles (Finks & Petit—*Anal. Chem.* 21, 1101). Internal wetting of fibers was demonstrated to be a factor in detergency in tests observing microscopically the release of oil from fibers in detergent solutions (Powney—*J. Textile Inst.* 40, T519).

The dispersing powers of some detergents were evaluated. Greiner & Vold (*J. Physical & Colloid Chem.* 53, 67) recorded that nonionic detergents showed no suspending power whereas others did. The tests were based on ability to suspend manganese dioxide. Sodium carboxymethylcellulose, when used with soap and builder, prevented deposition of suspended matter on fabrics, but with soap alone it showed no advantage (Bayley *et al.*—*Laundry & Dry Cleaning J.* Dec. 1948, 4 pp.). In test with nonionic detergent the carboxymethylcellulose did have desoiling power (Feuell—*J. Textile Inst.* 40, T523). In another test when carboxymethylcellulose was used with sodium alkylaryl sulfonate and builders better carbon soil removal and whiteness were obtained (Vaughn & Smith—*J. Am. Oil Chemists' Soc.* 26, 733). Without the builder, carbon soil removal increased but whiteness was not improved nor was it decreased. The suspending action of potassium silicate in soaps was determined on various materials (Merrill & Getty—*Ibid.* 5). The silicate was more effective than potassium carbonate for suspending ilmenite. A mixture of soap and potassium silicate prevented deposition of iron oxide pigment on cotton cloth about equally as well as soap alone; and was more effective than soap for preventing deposition of raw umber. Silicate-soap mixtures were equal to soap in distilled water, but the former was superior in hard water. A simple method for measuring dispersing power of detergent solutions comprised mixing with lightly oiled umber soil, letting stand for two hours, and measuring the turbidity of the suspension (Snell & Reich—*J. Soc. Chem. Ind.* 69, 98).

In discussing the chemistry of detergency, Grunfest & Young (*J. Am. Oil Chemists' Soc.* 26, 236)

emphasized that there was a relation between critical concentration of the detergent required and the amount of dirt to be removed. In a theoretical discussion on detergency Goette (*J. Colloid Sci.* 4, 459) reviewed the relations of the structure of various soaps, anion detergents, and pH to wetting and washing action. In a discussion on washing with soap

Gardner & Smith (*J. Am. Oil Chemists' Soc.* 26, 194) suggested that an acid soap was adsorbed on the fabric which in hard water formed lime soap with strong adhering capacity. The lime soap was largely retained by the fiber and repeated washes accumulated substantial amounts.

Report of the Oil Color Committee April, 1950

SEVEN years ago with permission of the newly elected president, Lamar Kishlar, the Oil Color Committee of the Society was reorganized with the expressed objective of developing an instrumental method for measuring vegetable oil colors. The first step taken was the appointment of a small subcommittee consisting of Reid Milner, Procter Thomson, and R. C. Stillman, who undertook to study the best approach to the problem. The early work included studies of correlation between Lovibond colors and visual appearance or eye order of oils. Following this, the subcommittee investigated filter photometers as a possible type of instrument to be used.

Nearly three years ago the subcommittee began the investigation of a spectrophotometric method and at the annual meeting in May, 1949, reported a method which was adopted by the Society as a tentative one. It was recognized that this method was not entirely satisfactory, and so during the summer of 1949 thirteen men representing nine laboratories began an intensive study in which more than 30,000 individual spectrophotometric, Lovibond, and eye order measurements were made on 120 samples of vegetable oils. All of these voluminous data were tabulated and averaged, and statistical studies were made in four of the nine laboratories referred to.

Two meetings of the subcommittee, fully attended, were held in Chicago early in October, 1949, and late January, 1950. At this last meeting four different equations were presented as a result of these statistical studies and, after the fullest discussion, one of these was unanimously adopted for recommendation to the Oil Color Committee and to the Society.

The real purpose of this review is to pay tribute to the three men who inaugurated this work and to express my deep personal appreciation to them and to the members of the subcommittee for their splendid cooperation and their untiring efforts in carrying through the tremendous volume of work that they have done. It is my conviction that seldom, if ever before, has any committee of our Society completed such a large amount of work in so short a time and so successfully.

It is my pleasure and privilege to include in this expression of appreciation:

R. J. Buswell	Reid T. Milner
F. R. Earle	W. H. Schmidt
M. W. Formo	S. O. Sorensen
S. Goldwasser	R. C. Stillman
D. L. Henry	Procter Thomson
Duncan Macmillan	L. K. Whyte
V. C. Mehlenbacher	

I also wish to thank for the subcommittee and for myself Geoffrey Beall of the statistical department of

Swift and Company and Earl Follett of the statistical department of Archer-Daniels-Midland Company for their tabulation and studies of the data.

G. WORTHEN AGEE, chairman

Committee Report

AT THE 1949 Spring Meeting of the Society in New Orleans a tentative spectrophotometric method for the determination of color of vegetable oils was adopted. The tentative method of analysis contained a simple equation for converting spectrophotometric readings at 525 m μ to red color. The committee was not convinced that this simple equation was the best that could be derived for expressing the overall color of an oil in terms of its spectrophotometric readings. As a consequence a program involving the complete spectral analysis of 120 soybean, cottonseed, and peanut oils was undertaken. Nine different laboratories carried out the scheduled work.

It is impossible to include in this report a compilation of the data obtained. The committee plans to assemble all of the data, together with the statistical calculations later carried out on the data, in a separate report which will be made available to the members of the Society but which will not be published.

All of the data on the 120 oil samples were obtained prior to September 1, 1949. A number of the committee members then carried out statistical analyses of the data to determine what wavelengths best characterized the overall color of the oil. These statistical analyses were discussed at a meeting of the committee held in Chicago the week of October 2, 1949. At this meeting it was definitely decided that the primary wavelength for color measurement should be 550 m μ , that a green correction based on measurements at 620 and 670 m μ should be added, and that the possibility of a measurement at 460 m μ or nearby should be investigated. It was further decided that all correlations should be made using actual Lovibond colors as the primary correlating factor.

A second meeting was held in Chicago the week of January 30. At this meeting, which was attended by all of the collaborators, the results of the statistical treatment of the data were discussed. Of all of the equations derived for computing a red color correlating with Lovibond red, the best one involved transmittance readings at 460, 550, 620, and 670 m μ . The equation: photometric color = $1.29 \times D_{460} + 69.70 \times D_{550} + 41.2 \times D_{620} - 56.4 \times D_{670}$ gave a correlation of 0.993 with Lovibond red. Calculated photometric colors based on this equation are plotted against actual Lovibond red in Figure 1.