

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

## Report of the Literature Review Committee\*

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### Deterioration

**REVIEWS.** The reviews on deterioration of fats and oils varied in scope. Loury (*Corps gras, savons* 2, 137; *Inds. corps gras* 1, 14; *Bull. matières grasses inst. colonial Marseille* 28, 138) prepared a brief but comprehensive review of fat deterioration and means of preventing it. In a lecture on recent British antioxidant studies, Lovern (*Oil & Soap* 23, 40) discussed a rapid test for evaluating rancidity inhibitors and the efficiencies of several of these for protection of fat and carotene. Other reviews on antioxidants included a short, comprehensive review (Ripert & Sisley—*Inds. corps gras* 2, 104), one on natural active material (Dubouloz—*Ibid.* 1, 68), a discussion on soap preservatives (Better & Davidsohn—*Soap, Perfumery Cosmetics* 19, 132), and a card catalog of abstracts of patents and literature on antioxidants (Anon.—*Quartermaster Food and Container Inst. Armed Forces J.Q.M.D.* 600, about 700 cards).

**METHODS FOR TESTING.** Modifications were suggested for improving the accuracy of the peroxide value determination for fats and oils. Lea (*J. Soc. Chem. Ind.* 65, 286) designed apparatus and a method to reduce the oxidation of the test sample by dissolved oxygen during the determination. The procedure permitted analysis of small samples without obtaining the increase in values as was heretofore common on reducing sample size. For a similar purpose, Stuffins & Weatherall (*Analyst* 70, 403) bubbled inert gas through the reaction mixture. Hills & Thiel (*J. Dairy Research* 14, 340) improved the sensitivity of the ferric thiocyanate method for estimating the peroxides of fats by substituting a benzene-methanol mixture for the acetone. The reagent prepared with the new solvent had greater stability. A new method for assessing the quality of butter depended on the fluorescence of the serum (Brant & White—*Can. J. Research* 24F, 461). The fluorescence value of the phospholipid fraction of egg powders was suggested as a measurable criterion for palatability of the products (Dutton *et al.*—*Ind. Eng. Chem.* 38, 1082).

Several investigations compared rancidity tests and their limitations. Watts & Major (*Oil & Soap* 23, 222) observed that the temperature of fat storage had a greater effect on the Kreis test than on the peroxide value, *i.e.*, increasing the temperature caused a decrease, not only in the peak values of the former, but also in the ratio of Kreis to peroxide values. McConnell & Esselen (*Ibid.* 369) observed the changes in various tests on aging corn and cottonseed oils in sealed containers. The changes in methylene blue fading depended on presence of air and high storage temperatures. Aldehydes developed slowly even with extensive rancidity. Film pressure changed only with exposure to light or aeration. The length of the induction period and changes in chlorophyll and fluorescence depended on exposure to light. Light

transmission and the ultra-violet and infrared spectra were unchanged on rancidification. Grant & Lips (*Can. J. Research* 24F, 450), in similar work, recorded the following correlation coefficients between odor scores and the logarithms of the chemical tests: iodometric peroxide value —0.90,  $\alpha$ -dicarbonyl test —0.85, Stamm reaction —0.82, Kreis test —0.81, ferrometric peroxide value —0.80, fluorescence —0.79, free fat acids —0.10. Rancidity was best rated chemically by measuring the  $\alpha$ -dicarbonyl compounds, for they were the most stable products developed during spoilage. Støren & Dovle (*Meldinger Norges Landbruks-hogskole* 24, 360) estimated peroxides, aldehydes, and quality of 226 butter samples. Defects in flavor were evident in 85% of the samples having a peroxide value of 0.5 or more and an aldehyde value of 0.40 or more. Beadle (*Oil & Soap* 23, 33) reviewed the various rancidity tests and discussed their limitations. All the investigators of deterioration criteria pointed out that, as yet, there is no reliable chemical measure for rancidity.

Two devices were designed to measure stability of fats. Eekey's (*Oil & Soap* 23, 38) equipment recorded the time required for one gram of fat to absorb three milliliters of oxygen. For a similar

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### TABLE OF CONTENTS

- A. INTRODUCTION.  
Statistics, outlook, fostering individual sources, new literature.
- B. PRODUCTION PROCESSES.  
Animal fats, vegetable fats, refining, tall oil, medicinal oils, fat acid fractionation, hydrogenation, interesterification.
- C. PRODUCTS (except detergents).  
Table and cookery fats, emulsifiers, protective coatings, resins, synthesis of acids, alcohols, ketones and esters, fat derivatives containing nitrogen, soaps of polyvalent metals, lubricants, fuels, miscellaneous products.
- D. DETERIORATION.  
Reviews, methods for testing, mechanisms, factors, influencing autoxidation, flavor reversion, biochemical rancidity, radiochemical changes in fats.
- E. BIOCHEMISTRY AND PHYSIOLOGY.  
Desirability of fat in the diet, relative dietary values, adsorption, mobilization, fat soluble vitamins.
- F. CHARACTERISTICS AND COMPOSITION.  
Methods of testing, new data.
- G. DETERGENTS.  
Soap manufacture, testing, list of patents on nonsoap detergents.

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purpose the equipment of Gilmont *et al.* (*Ibid.* 248) recorded the volume of oxygen absorbed under constant pressure. The rate was plotted so that the induction period could be distinguished by the break in the curve. Bickoff & Williams (*Ibid.* 65) advised that since antioxidant protection was better at lower temperatures, rapid high temperature tests were less accurate indicators of stability.

The stability test for fatty textile lubricants, Mackey test, was improved by ventilating the test samples with measured volumes of compressed air (Schmidt-Nielsen & Naerland—*Kgl. Norske Videnskab. Selskab., Forh.* 16, 83, 125; 17, 47, 134, 157, 169). The authors investigated this test with marine oils and rated the influence of acid value, peroxide value, pro-oxidants, antioxidants, and per cent unsaponifiable.

**MECHANISM.** Two spectroscopic investigations by Bergström (*Arkiv. Kemi, Mineral. Geol.* 21A, No. 14, 1; No. 15, 1) and Holman *et al.* (*Oil & Soap* 23, 10; *J. Am. Chem. Soc.* 68, 562; *Arch. Biochem.* 10, 519) agreed that, in general, the autoxidation of unsaturated fat acids and the lipoxidase oxidation of the same led to the identical primary oxidation products. Bergström's evidence for this statement was the isolation of 9- and 13-hydroxystearic acid from the hydrogenated products of methyl linoleate oxidized by both methods. He believed that autoxidation started at the methyl group between the double bonds. Holman and coworkers' reports pointed out the similarities in developments of various chromophores as related to both types of oxidation of linoleic, linolenic, and arachidonic acid esters. Much of their work and discussion pertained to the degree of conjugation produced and compounds formed during the process; however, they felt that additional data on pure compounds would be necessary to confirm their suggestions and those of others along these lines. Swift *et al.* (*Oil & Soap* 23, 355) made a step in this direction when they isolated methyl hydroperoxido oleate in 85-95% purity from partially oxidized methyl oleate. Analysis by hydrogenation, oxidative fission, and reduction indicated a mixture of 8- and 11-hydroperoxido C<sub>18</sub> acids. The spectral absorption of the products was recorded. Gunstone & Hilditch (*J. Chem. Soc.* 1946, 1022) also studied oxidation of methyl oleate. The process was accelerated by the presence of small amounts of methyl linoleate. They discussed some of the above interpretations and other recent papers in connection with the suggestion that the initial association of oxygen was with a double bond and not with an adjacent methylene group. All the papers referred to above on the mechanism of the autoxidation are concerned both with rancidification and with initial bodying or polymerization reactions.

**FACTORS INFLUENCING AUTOXIDATION.** Commercial common salt was found to accelerate oxidation of many fats (Hills & Conochie—*J. Council Sci. Ind. Research* 18, 355). This pro-oxidant action was attributed to the magnesium chloride impurities and the action could be greatly reduced by grinding the salt with one-half per cent of sodium carbonate. This same group (*Ibid.* 19, 1) suggested that the effect of the salt solution may arise from the liberation of active oxygen from water through formation of hypochlorous acid. Wiesman (*Food Industries* 18, 1863) avoided early advent of rancidity in frozen pork sausage by omitting salt from the formula.

Lea's (*J. Soc. Chem. Ind.* 65, 136) investigation on the effect of the container on butterfat showed that tin induced deterioration more rapidly than lacquered or glass surfaces. Work by Barnicoat (*New Zeal. J. Sci. Technol.* 27A, 343), on effect of storage temperature on grade of butter, showed the rate of deterioration rose rapidly from 14° to 60°, while the difference between -5° and 14° was too slight to warrant the extra expense involved in storing at temperatures below 14°. In regard to the effect of the concentration of oxygen in the atmosphere, Schaffer *et al.* (*J. Dairy Sci.* 29, 145) markedly increased the keeping qualities of butterfat only after decreasing the oxygen concentration below five per cent.

Most of the work on susceptibility of animal fats to oxidation was on hog fats. According to Tuck *et al.* (*New Zeal. J. Sci. Technol.* 27A, 212) the flare and outer back fat of pigs fed solely on buttermilk was less stable to oxidation than the inner back fat. They found no correlation between the rate of growth of the fatty tissue and its susceptibility to oxidation. Watts *et al.* (*Oil & Soap* 23, 254) demonstrated that the diet of hogs affected the stability of the fat produced. However, feeding of tocopherol, the natural antioxidant of lard, resulted in only small increases in stability; subcutaneous injections added no protection. Soft lard, of high iodine number, produced as the result of soybean feeding, had less stability than firmer products (Brady—*J. Animal Sci.* 5, 358). Crude expeller process cottonseed oil was a better stabilizer for this product than nordihydroguaiaretic acid (N.D.G.A.). In the work of Burr *et al.* (*Ibid.* 382) on the effect of diet on stability of fats, rats were used as experimental animals. It was found that the protein level was unimportant; dietary fat was important; and dietary tocopherols improved stability of the fat deposited, while hydroquinone and wheat germ oil had a deleterious effect.

In work on stability of fat in salt herring, Lundborg (*Svensk Kem. Tid.* 58, 172) observed deterioration was slower as the amount of fat in the fish increased. Other work on fish oils pertained to destruction of vitamin A by rancidification reactions. Rao (*Indian J. Med. Research* 32, 155, 161; 33, 63, 69) rated the effect of temperature and metallic pro-oxidants on development of peroxide and acid values and on destruction of vitamin A in shark-liver oil. In a search for stable oil carriers for fish liver vitamin concentrates, Sen-Gupta (*J. Indian Chem. Soc.* 23, 233) observed that vitamin A was rapidly destroyed in sesame oil. Similar work on  $\beta$ -carotene, the provitamin A from plants, by Euler & Euler (*Ark. Kemi Mineral. Geol.* 18B, No. 12, 8 pp.) showed greatest destruction in coconut oil, palm kernel oil, lard, and tallow, while oils of higher unsaturation had greater protective effects as solvents for the carotene.

In nutritional experiments with diets containing linseed oil and carotene, Tomarelli & György (*J. Biol. Chem.* 161, 367) obtained synergistic action for a tocopherol antioxidant with rice bran extracts. In work on the kinetics of antioxidants and synergists, Golumbic (*Oil & Soap* 23, 184) rated the efficiency of many mixtures for preservation of lard. The most effective combinations of antioxidant and synergist were found to have an oxidation potential between 848 and 484 millivolts.

Some work was recorded on rating antioxidant action on specific products. According to Rac [*Kem. Vjestnik* (Zagreb) 15/16, 67] autoxidation of linseed oils was inhibited by reducible aniline dyes; ascorbic and barbituric acids had a mild effect, and barely flour exhibited a very slight effect in the tests. In similar work with common antioxidants by Smith & Stotz [*N. Y. (Geneva) Agr. Expt. Sta. Tech. Bull.* 276, 30 pp.], the presence of copper in the linseed oil affected the degree of protection of the various antioxidants differently. In work on correlating the keeping properties of shortening with the keeping quality of biscuits, Maveety (*Oil & Soap* 23, 25) observed effective influences by lecithin, molasses, and spices. A test on 19 aromatic chemicals for preservation of soap revealed that vanillin, eugenol, coumarin, and methyl anthranilate were most desirable (Better & Davidsohn—*Ibid.* 245, 283).

Some communications treated individual antioxidants. Wheat germ oil reduced the accelerating influence of copper in whole milk powder (Chapman & McFarlane—*Can. J. Research* 24F, 47). Commercial tannins (Spannuth *et al.*—*Oil & Soap* 23, 110) and N.D.G.A. (Fonyo—*Am. Perfumer Essent. Oil Rev.* 42, No. 8, 59) were recommended for animal fats. The fat in fish flesh was more stable after treating the fish with ascorbic acid (Tarr—*Fisheries Res. Board Can. Prog. Repts. Pacific Coast Stas. No.* 68, 52). A water-soluble constituent of liver was effective for ethyl esters of fat acids (Dubouloz—*Compt. rend. soc. biol.* 138, 670). The literature on the effectiveness of phosphatides was reviewed (Bibby—*Food Manuf.* 20, 441).

Some published information on antioxidants did not pertain to their protective activity. Fonyo (*Oil & Soap* 23, 75) discussed the availability of N.D.G.A. Gisvold *et al.* (*J. Am. Pharm. Assoc.* 35, 188) synthesized some  $\alpha, \omega$ -bis(3,4-dihydroxyphenyl) alkanes because their similarity to N.D.G.A. suggested possible usefulness as antioxidants. Evidently, Morris & Riemenschneider (*J. Am. Chem. Soc.* 68, 500) prepared fat alcohol esters of gallic acid for a like purpose. Lundberg & Halvorson (*Proc. Inst. Food Tech.* 1945, 115) demonstrated that the solubilities of hydroquinone, N.D.G.A. gallic acid, and propyl gallate in lard was greater than was required for most effective stabilization. The antioxidant, ascorbyl palmitate, retarded the growth rate of rats when fed at a five per cent level in the diet (Fitzhugh & Nelson—*Proc. Soc. Exptl. Biol. Med.* 61, 195). However, this dosage was a considerable multiple of that possible in diets containing fats protected by the antioxidant in effective amounts.

In the recently patented methods of stabilizing fats and oils the following antioxidants were used: thio di-fatty acids and their esters (O'Leary—*U. S.* 2,397,976), 3-hydrocarbon substituted  $\beta$ -mercapto-propionic acid (Gribbins *et al.*—*U. S.* 2,397,960), special sugar amines (Gubelmann—*U. S.* 2,396,097), *N, N'*-dicyclohexyl-*p*-phenylene-diamine (Cook & Thomas—*U. S.* 2,393,889), a partial carboxylic acid ester of gum guaiac (Black—*Can.* 431,986), reaction products of 1,2-dihydroquinolines and diarylamines (Gibbs—*U. S.* 2,400,500), a mixture of propyl gallate and  $\beta$ -amino-ethyl alcohol (Korner & Loomis—*U. S.* 2,394,456), a mixture of tocopherol with trypsin, pepsin, or clarase (Coe & Coe—*U. S.* 2,397,920), special alcohol extracts of vegetable oils (Buxton—*U. S.* 2,389,955, 2,396,679-

81), oil meals (Buxton & Konen—*U. S.* 2,401,293), and blackstrap molasses (Musher—*U. S.* 2,410,455). The newly patented soap antioxidants were polyethylene polyamines (McClain—*U. S.* 2,409,056) and stannic borate (Lofdahl—*U. S.* 2,411,443). The oil in roasted coffee was stabilized by treatment with infra-red rays (Chappell—*U. S.* 2,410,427). Procedures for obtaining pure N.D.G.A. by extraction followed by crystallization were patented (Gisvold—*U. S.* 2,382,475, 2,408,924). The tendency of discoloration with the use of gum guaiac as an antioxidant was reduced by the presence of acetic acid (Black—*U. S.* 2,408,904).

**FLAVOR REVERSION.** A comprehensive and critical review on this subject was prepared by Bailey (*Oil & Soap* 23, 55), who showed that the trend was to associate the phenomenon with the presence of polyunsaturated acids. Golumbic *et al.* (*Ibid.* 187, 380), in investigating this hypothesis, did not obtain reversion of the soybean type from a simulated soybean oil synthesized from purified fat acids. They suggested that the polyunsaturated acids contributed to the reversion but were not wholly responsible. In another investigation, they (*Ibid.* 360) observed that reduction of oxidation of soybean oil by inert atmosphere did not diminish the tendency to revert, but it produced a reversion that was more persistent in taste than that resulting in air.

**BIOCHEMICAL RANCIDITY.** Hejna & Hedrick (*J. Bact.* 51, 601) rated the capacity of several organisms to produce rancidity reactions. In coconut oil emulsions most free fat acids were formed by *Aspergillus*, least by *Pseudomonas*; greatest peroxide formation was by *Aspergillus*, and least by *Pseudomonas*. In soybean oil emulsions most free fat acids were formed by *Penicillium*, least by *Serratia* (with N.D.G.A.); and least reduction was by *Aspergillus*. According to Greenwood *et al.* (*Proc. Inst. Food Tech.* 1945, 58) the use of calcium propionate to inhibit the rancidification of bacon was unsuccessful, for moldicidal amounts rendered the product inedible. Van Fleet (*Biodynamica* 5, 297) measured the reduction-oxidation potentials of fat emulsions and noticed the occurrence of two types of plant oxidase; one was cyanide-stable and heat-labile, the other cyanide-labile and heat-stable. A change in potential on oxidation was attributed to the removal of oxygen from the medium when fats were oxidized by light.

Loury *et al.* (*Bull. matieres grasses inst. colonial Marseille* 29, 5, 25) recorded studies on the natural lipases. He found that they act dissimilarly on different oils. A large number of inhibitors and activators of these enzymes were evaluated for their effectiveness.

**RADIOCHEMICAL CHANGES IN FATS.** Bombardment of fats with  $\alpha$ -particles caused dehydrogenation, decarboxylation, formation of low molecular weight acids, formation of methane and higher hydrocarbons; and production of carbon monoxide, carbon dioxide, hydrogen, and water (Sheppard *et al.*—*Bull. Am. Assoc. Petroleum Geol.* 30, 32; *J. Am. Chem. Soc.* 68, 1636; Honig—*Science* 104, 27).

### Biochemistry and Physiology

**DESIRABILITY OF FAT IN THE DIET.** Heretofore experiments on the need for fat in the diet have been concerned largely with essential fat acids, fat-soluble vitamins, and energy. Recently, other nutritional

aspects, *i.e.*, growth, physical performance, economy of food utilization, relationship to other nutrients, palatability, and satiety of these food constituents have been investigated. Deuel (*Oil & Soap* 23, 209) has reviewed these aspects.

Forbes *et al.* (*J. Nutr.* 31, 203, 213; 32, 387), in a 70-day metabolism study with growing albino rats, observed that weight gains, digestibility of nitrogen-containing nutrients, and decrease in heat production take place in the order of increasing fat content of isocaloric diets containing from two to 30% fat. In similar experiments with adult rats increasing dietary fat improved nitrogen retention and body gains of fat. A decreasing energy expense in utilizing the higher fat diet was attributed to a decrease of heat necessary for the catabolism of fats as compared to that needed for carbohydrates. Some aspects supporting the desirability of fat were announced by Deuel and coworkers (*Oil & Soap* 23, 211) but have not yet been published in detail. In these, optimum growth of rats occurred when the otherwise adequate diet contained 20% cottonseed oil (33% of caloric intake). Maximum physical performances in rats, as measured by a swimming test, was obtained with a diet containing 40% fat.

Rats maintained at  $-2^{\circ}$  self-selected diets higher in fat (50% of total calories) than they did at room temperature (Dugal *et al.*—*Can. J. Research* 23E, 244). The self-selected high-fat diet at the low temperature also proved superior to the low-fat diets in regard to survival of the rats. Similarly, Mitchell *et al.* (*Am. J. Physiol.* 146, 84) showed that humans on a high-fat diet had a greater tolerance to cold. In these tests only a slight ketonuria, and no hemolysis, was observed.

A determination of the specific dynamic action of diets high in carbohydrates as compared to those high in fat found them practically the same in this respect (Ring—*J. Nutr.* 32, 653). In a study to measure the activity of folic acid preparations in poultry diets, high-fat diets reduced the effectiveness of this nutritive accessory (Luckey *et al.*—*Proc. Soc. Exptl. Biol. Med.* 62, 307). Isocaloric substitution of fat for carbohydrates in cotton rat diets reduced dental caries in proportion to the fat in the diet (Schweigert *et al.*—*J. Nutr.* 31, 439). Rats on a thiamine-free diet containing 1.8% fat lived 25-30 days; this survival period was doubled by isocalorically increasing the fat to 26% at the expense of carbohydrate (DeCaro—*Boll. soc. ital. biol. sper.* 15, 553). Geyer *et al.* (*J. Biol. Chem.* 162, 251) believed that fat was in some way involved in the utilization of galactose because the excretion of galactose by rats fed skim milk was markedly lowered by inclusion of fat in the diet.

The contributions on "essential" fat acids contained two reviews (Hansen & Burr—*J. Am. Med. Assoc.* 132, 855; Desnuelle—*Inds. corps gras* 2, 172). In a search for the biologically active fat acids, Jürgens *et al.* (*Helv. Physiol. Pharmacol. Acta* 3, 41) found that linoleic was active, while the saturated, the monounsaturated, and the following diunsaturated acids, 2,6-phytodienoic, 10,13-nona-decadienoic, 11,14-icosadienoic, and 9,10-octadecadienoic acids, were inactive. Hove & Harris (*J. Nutr.* 31, 699) showed that  $\alpha$ -tocopherol exerted a sparing action on the "essential" fat acids in the rat, presumably through protection of the double bonds, not only in the intestinal tract but also in the tissue. This was also

true in the nutrition of caterpillars of the flour moth where linoleic or linolenic acid was shown to be essential by Fraenkel & Blewett (*Biochem. J.* 40, xxii). The growth of *Lactobacillus casei* was stimulated by oleic acid (Williams & Fieger—*J. Biol. Chem.* 166, 335).

RELATIVE DIETARY VALUES OF FATS. The comparative value of butter and other fats maintained a position of exaggerated interest due to the continued scarcity of butter. The major question was, "Are margarine and other fats nutritionally equivalent to butter?" In two critical reviews on this subject Deuel (*Science* 103, 183; *Oil & Soap* 23, 209) cited experiments which affirm the equality of vitamin-fortified margarine and butter. This author announced in the latter review that a generation test with rats on a skim milk and margarine diet had passed the fifteenth generation without indications of approaching deficiencies. Deuel and coworkers (*J. Nutr.* 31, 737, 747; 32, 69) also added new evidence to reaffirm the interchangeability of butter and other fats without onset of deleterious nutritive effects. No differences were found between butter, margarine, a commercial hydrogenated fat, or corn, cottonseed, peanut, or soybean oils when compared for growth of rats on restricted and adequate caloric intake, also on injection of hormones. In tests using fat-free skimmed milk powder as the basal ration, the growth responses were essentially the same as with the above fats. In their human experiments comparing butter and margarine, the digestibilities were found to be practically identical (97%). The results of Parrish *et al.* (*Ibid.* 31, 321) on the growth value of various fats agreed with those of Deuel *et al.* In attempting to clarify discordant results of others, they suggested that differences may be related to the food intake. According to Euler *et al.* (*Arkiv. Kemi Mineral. Geol.* 17A, No. 19, 15; 22A, No. 8), margarine fats may be superior to butterfat when evaluated on the basis of reproduction and lactation. Spector (*Arch. Biochem.* 11, 167) observed no consistent distinction between the fat acids of butter and of corn oil for the growth and metabolism of seven microorganisms. A better growth response in rats by summer butter as compared to other fats was attributed by Boer *et al.* (*Arch. néerland physiol.* 28, 57) to the presence of a growth factor among the fat acids. In a later paper (Boer *et al.*—*Nature* 158, 201), this growth factor was identified as vaccenic acid. However, this suggestion should be examined in the light of past literature which records the presence of vaccenic acid as common to animal fats.

Some work on relative dietary value of fats was concerned with the butter versus margarine question. Abelin (*Experientia* 2, 105) believed that oils were nutritionally superior to fats because they contained more sterols, which he thought had special value in increasing body weight and protection against infection. Mattil *et al.* (*Science* 104, 255) suggested that plastic shortening made from lard has an advantage over hydrogenated vegetable oil shortening because the major component is unhydrogenated; and hence no appreciable destruction of the naturally occurring, essential, unsaturated fat acids has taken place. Both types of shortening were equally well digested. Crampton & Mills (*Can. J. Research* 24F, 157) compared compound animal-vegetable, hydrogenated vegetable, and lard shortenings when incorporated into

biscuits. Lard was slightly more digestible. The fats were unaffected by the baking temperatures. A slower growth of rats on the biscuits baked at 425° F. as compared to those baked at 375° F. was attributed to the effect of the heat on the protein fraction. Mattil (*Oil & Soap* 23, 344) statistically analyzed the recorded digestibility data on fats and concluded that the amount of C<sub>18</sub> or higher saturated acids is the chief limiting factor. This suggestion accounted for the lesser degree of correlation which existed between digestibility and melting point.

According to Paraf *et al.* (*Compt. rend. soc. biol.* 139, 863), fat acids containing  $\alpha$ -methyl side chains are very toxic. Kraut *et al.* (*Biochem. Z.* 317, 187) and Kabelitz (*Ibid.* 316, 409), with new tests, confirmed past statements to the effect that the German synthetic fats were innocuous. However, unfavorable reports on these fats may have been banned from publication for an investigation of the German fat and oil industry revealed unpublished work disputing such findings (Markley—*U. S. Dept. Com. Off. Tech. Service P. B.* 18302).

**ABSORPTION OF FATS.** The one review on the subject was by Frazer (*Physiol. Rev.* 26, 103). This was of critical nature in support of his partition theory in contrast to the lipolytic hypothesis. Special emphasis was placed on fat acid-bile salt-monoglyceride emulsification to produce negatively charged particles of less than 0.5  $\mu$  diameter which can pass the outer border of the intestinal wall through small canals. This same author (*Nature* 157, 414) also presented histological evidence that choline aids the dispersion and passage of fat through the intestinal mucosa. This finding was also confirmed by Artom & Cornatzer (*J. Biol. Chem.* 165, 393). The mechanism in this case was attributed to the stimulation of choline for phosphorylation of the lipids.

Utilization of fats as influenced by bile was evident in some other observations. Maggioni & Passalacqua (*Boll. soc. ital. biol. sper.* 20, 432) reported that an infant with complete atresia of the bile duct showed a fat absorption of only 67-78%. The finding of bile in the gastric juice of crayfish by Vonk (*Rec. trav. chim.* 64, 320) supported the hypothesis that bile acids were responsible for dissolving fats even in crustacea. Heretofore, it was believed that the occurrence of bile acids was limited to vertebrates. The conditioning of fats for absorption by bile salts was evident in the data of Ruffo & Imparato (*Boll. soc. ital. biol. sper.* 19, 314, 317, 319) who measured the reduction of viscosity of suspensions of oleic acid by additions of sodium glycocholate and sodium taurocholate. Very young calves required that their food fats be finely dispersed, for homogenization of fat in skim milk prevented an alopecia which occurred in animals fed the same ration without homogenization of the constituents (Bate *et al.*—*J. Dairy Sci.* 29, 41).

Cooke *et al.* (*Quart. J. Med.* 15, 141) recorded the anomalies of absorption of fat by 120 humans with various intestinal disturbances. Patients with diarrhea, anemia, and gastro-intestinal disorders (except for those with steatorrhea) absorbed fat almost normally; while abnormal absorption occurred with idiopathic steatorrhea, tropical sprue, pancreatitis, and following certain gastro-intestinal surgery. Wollaeger *et al.* (*Gastroenterology* 6, 93) also reported impaired fat absorption following gastro-intestinal surgery.

An investigation on the fats eliminated by the intestines, with special reference to dietary unsaturated fats, by Wikoff *et al.* (*Am. J. Digestive Diseases* 13, 228) revealed an unexplained anomaly of fat absorption. In test diets of 10 and 20% triolein, dogs eliminated more lipids and soaps in the feces when the lower concentration was fed. The lipids of the feces on the lower olein diet did not contain appreciable quantities of oleic acid.

The *in vitro* hydrolysis of fats by enzymes is reviewed here because the rates of such reactions are often associated with rates of intestinal absorption. A method for determination of lipase activity by Archibald (*J. Biol. Chem.* 165, 443) has taken into account the fact that the action increased as the substrate concentration increased up to 37.5%, that the reaction was reversible, and that equilibrium with oleic acid and pancreatic lipase was reached when 50% of the fat acid was still combined with glycerol. Schonheyder & Volqvartz (*Acta Physiol. Scand.* 10, 62, 349; *Enzymologia* 11, 178) activated enzyme hydrolysis by removing the liberated fats from the media by precipitation with calcium chloride. Further work on ideal conditions for reaction of pancreatic lipase indicated that pH 5.5-5.8 was optimum for C<sub>8</sub> and C<sub>4</sub> acids but this shifted to the alkaline side with increasing number of carbon atoms. *In vivo* experiments showed that only the lower triglycerides were split during a 25-minute test period and that in children cow butterfat was split more rapidly than human milk fat. The results regarding effects of the chain length on hydrolysis were confirmed by Menezes & Banerjee (*J. Indian Inst. Sci.* 27A, Pt. 2, 7). Also in this work the presence of thiamine and ascorbic acid had no influence, rancidification lowered the rate, while addition of unsaponifiable accelerated the rate of hydrolysis. The latter was attributed to the sterols. A reduction of digestibility by refining and deodorization was believed to be due to removal of fat soluble vitamins, sterols, etc. and was said to be counteracted by fortification with these. Ahmad and Sareen [*J. Sci. & Ind. Research (India)* 4, 710] recorded the rates of hydrolysis of various oils with ricinoleic lipase.

**MOBILIZATION OF FATS.** Best (*Am. J. Digestive Diseases* 13, 155) summarized the status of knowledge concerning the transport and mobilization of fats under normal, pathological, and experimental conditions.

The work on deposition of fat acids or their conversion in the animal body included both fundamental and applied aspects. Some labeled C<sub>12</sub> or C<sub>14</sub> acids fed to rats were synthesized to and deposited as fat acids of longer chain length (Klem—*Hvalrødets Skrifter, Norske Videnskaps-Akad. Oslo No.* 27, 26 pp.). It was interpreted to indicate that  $\beta$ -oxidation of fat acids is a biologically reversible process. Metabolism of ricinoleic acid by rats was again confirmed (MacQuiddy *et al.*—*J. Lab. Clin. Med.* 31, 763). That the rat can deposit and store hendecanoic (C<sub>11</sub>) acid was just recently recorded (Visscher—*J. Biol. Chem.* 162, 129). In investigations concerning soft pork production, Schulz & Thomas (*Iowa Agr. Expt. Sta. Research Bull.* 336, 18 pp.) observed that dietary adjuncts such as carotene, bile salts, agar-agar, sodium fluoride, and calcium induced deposition, in rats fed soybean oil, of fat with somewhat lower iodine numbers. However, the results were not of practical

significance for solving the oily lard or soft pork problems. Robison (*Ohio Agr. Expt. Sta. Bull.* 664, 26 pp.) recorded that, among hogs fed the same ration, the fat of the rapid gaining hogs was slightly firmer than that of hogs which had gained more slowly.

The discovery that fungicidal C<sub>7</sub>, C<sub>9</sub>, C<sub>11</sub>, and C<sub>13</sub> acid compounds were secreted by the sebaceous glands of the adult human scalp (Rothman *et al.*—*Science* 104, 201; *Proc. Soc. Exptl. Biol. Med.* 60, 394; Shapiro & Rothman—*Arch. Dermat. & Syphilology* 52, 166) is of interest inasmuch as the occurrence of odd chain acids in nature is unusual. The presence of the acids in the adult scalp was said to explain why scalp ring worm is endemic in children but clears up spontaneously with oncoming adolescence.

A project started in 1936 on the analysis of fish lipids has continued to develop data on the deposition, distribution, composition, and metabolism of lipids in fish at various seasons (Black *et al.*—*J. Soc. Chem. Ind.* 64, 326; 65, 13; Roux *et al.*—*Ibid.* 281). In addition to the wealth of physiological information which resulted from this project, it has served as a basis for the origin and development of a fish oil and vitamin concentrate industry in South Africa.

Two investigations were on the fat deposited in milk. A gummy defect of Texas butters was attributed to high melting fat acids, for such butter resulted when the milk fat was obtained from cows which were fed as little as one ounce of either stearic or palmitic acid daily (Arbuckle & Copeland—*Prog. Rept., Tex. Agr. Expt. Sta.* 1031, 4 pp.). According to Anantakrishnan *et al.* (*J. Biol. Chem.* 166, 31; *Biochem. J.* 40, 292), colostrum fat of the cow and buffalo differed in composition from that of normal butterfat. The chief changes were gradual increases in the amount of butyric, myristic, and palmitic acids and a decrease in the amount of stearic and oleic acids.

Reports on the function of the liver in intermediary fat metabolism were concerned mostly with characterizing the effects and mechanism of lipotropic factors. Most of the investigations dealt with the biochemistry of choline. Entenman *et al.* (*J. Biol. Chem.* 162, 111), Fishman & Artom (*Ibid.* 164, 307), and Horning & Eckstein (*Ibid.* 166, 711) observed that the first effect of the choline was to transport phospholipids to the liver and decrease the noncholine phospholipid content of the liver. In continuing the investigation, Entenman *et al.* (*Ibid.* 15) suggested that the liver tissue was concerned not only with the synthesis and supplying of phospholipids to the plasma but also with their removal. The prolonging of the turnover of plasma phospholipids in dogs from 6-10 to 33-160 hours by depriving the animals of their normal liver was submitted as evidence for this statement. These mechanisms were traced by quantitative studies and by the use of radioactive labeled elements. A new technic for this line of work, as suggested by McArthur (*Science* 104, 222), involved replacing the dietary choline with its triethyl homolog. This compound had lipotropic properties, was incorporated in the liver phospholipids, and could be isolated.

In the course of development of the knowledge of choline in lipid metabolism, many varied functions have been attributed to it, and several other compounds were found that provided only part of the functions of choline. In the light of this history,

Almquist (*Science* 103, 722) suggested that choline deficiency was physiologically a multiple deficiency, and compounds with part of the functions of choline should be viewed from the standpoint of exerting a sparing action by releasing residual choline for its other functions. One lipotropic substance, inositol, was the subject of polemic investigations in order to establish whether its specific effect was to reduce the cholesterol content of the high cholesterol "biotin fatty livers." This year's papers on the subject were from Best and his school (*Ibid.* 103, 12; *Biochem. J.* 40, 368, 494) who found that choline was more active than inositol in decreasing glycerides or cholesterol esters of fatty liver. A synergistic lipotropic effect of choline plus inositol was observed but the inositol did not exert a preferential effect on cholesterol. Handler (*J. Nutr.* 31, 621; *J. Biol. Chem.* 162, 77), in work on elucidating the effects of these two factors, found that both have lipotropic activity, but inositol would not cure hemorrhagic kidneys due to choline deficiency. However, inositol plus tocopherol as well as lipocaic preparation decreased the kidney lesions. He therefore considered that tocopherol was synergistic for inositol. Handler & Dubin (*J. Nutr.* 31, 141), further elaborating on liver factors, found that high-fat low-protein diets produced both necrosis and fibrosis in albino rat livers, but when excess nicotinamide was added, fatty infiltration occurred with only minute necrosis. According to Victor & Pappenheimer (*J. Exptl. Med.* 82, 375), either choline or tocopherol cured the ceroid pigment abnormality of rat livers resulting from low protein diets containing excess cystine. The nonidentity of inositol and lipocaic was established by Eilert and Dragstedt (*Am. J. Physiol.* 147, 346) when inositol failed to cure fatty livers produced by low-protein high-fat diets. A newly reported lipotropic substance, manganese, according to Amdur *et al.* (*J. Biol. Chem.* 146, 783) may act through some interaction or relationship with choline.

Certain compounds containing methyl groups might be precursors for choline synthesis in the body. *N'*-methylnicotinamide was one of these, for during metabolism it was demethylated and a lipotropic effect was evident (Najjar & Deal—*J. Biol. Chem.* 162, 741). The transference of methyl groups from betaine, monomethyl- or dimethylaminoethanols to choline during metabolic synthesis was established with tests in which the compounds labeled with deuteriomethyl groups and N<sup>15</sup> were fed as the donors for choline synthesis (du Vigneaud *et al.*—*Ibid.* 164, 603; 165, 639). The compounds could be substituted for choline but were less efficient. In a review on the subject, Lucas *et al.* (*Proc. Soc. Exptl. Biol. Med.* 63, 32) supported the view that there exists many methylatable precursors of choline and that these were present in natural foods.

Attempts were made to apply fundamental knowledge on choline metabolism in the poultry industry. However, according to Lucas *et al.* (*Poultry Sci.* 25, 373) and Clandinin *et al.* (*Ibid.* 509), hens synthesize a substantial amount of choline, for its addition to choline-poor rations or normal rations induced only insignificant improvements. The latter group of investigators also recorded that the common rations are improved significantly with methionine supplements. According to Melass *et al.* (*Proc. Soc. Exptl. Biol. Med.* 62, 174), supplements of choline may be dele-

terious, for amounts between one and four per cent of rations fed to growing chickens reduced the rate of gain by about 12 to 23.8%, depending on the amount fed. Herrmann (*Ibid.* 61, 229, 302) recorded that feeding of choline to old hens will reduce that excess of cholesterol in the blood, aorta, heart, and liver which is common in old age.

In clinical work, Beams (*J. Am. Med. Assoc.* 130, 190) found choline and cystine treatment successful in seven of eight cases for reducing large liver abnormality that sometimes occurs in patients who had ascites. In several cases of pancreatectomy of man, the patients progressed very well on a normal diet with insulin but without lipotropic supplements (Waugh *et al.*—*Proc. Staff Meetings Mayo Clinic* 21, 25; Dixon *et al.*—*Arch. Surg.* 52, 619). Under the same conditions laboratory animals require supplements of choline to exist.

A relationship between the endocrines and fat metabolism in the liver was evident in several investigations. In fasting laboratory animals, the increase in liver fat which occurs in response to administration of anterior pituitary extract was partially inhibited by insulin (Campbell—*Am. J. Physiol.* 147, 742). Fatty livers produced in rabbits by adding cocoa fat to an ordinary diet were inhibited by insulin and favored by adrenalin (Sveinsson—*Skifter Norske Videnskaps-Akad. Oslo Mat.-Naturv. Klasse No. 8*, 138 pp.). Intermediary fat metabolism products, particularly the keto compounds, were believed responsible for partial or complete *in vivo* inactivation of insulin (Nath & Brahmachari—*Nature* 157, 336). Estrone had only a mild lipotropic effect on female rats on diets which produce fatty liver; but, when fed with methionine, it greatly augmented the lipotropic action of the latter (György & Rose—*Arch. Biochem.* 12, 125).

In one series of investigations (Li & Freeman—*Am. J. Physiol.* 145, 646, 660, 667) dogs on fatty-liver-producing diets underwent increases in serum and liver total lipids, but the amount of cholesterol was unaffected. On adding cholesterol to the diet, the increases of fat in serum and liver lipids were accelerated and more cholesterol was found. In connection with this work it was observed that the fat which accumulates in the liver of protein-deficient dogs was largely exogenous in origin. According to Okey (*J. Biol. Chem.* 165, 383) the lipotropic activity of biotin was effected by inhibiting or removing liver cholesterol. The mechanism of utilization and storage of another liver-lipid component, vitamin A, was unaffected by fatty liver producing diets or the presence or absence of choline (Bentley & Morgan—*J. Nutr.* 31, 333).

A critical review on the physiology of the non-fat lipids was prepared by Thannhauser & Schmidt (*Physiol. Revs.* 26, 275). A paper along these lines by Favarger (*Arch. intern. pharmacodynamie* 72, 1) was limited to the effect of cholesterol on fat transport. Foldes & Murphy (*Proc. Soc. Exptl. Biol. Med.* 62, 215, 218) determined cholesterol, cholesterol esters, and phospholipids in the corpuscles and plasma of patients and healthy individuals. All values were higher than normal in the plasma of hypothyroid patients. Similarly, Kountz (*Biol. Symposia* 11, 79) determined blood cholesterol levels of elderly patients. The pathological relationships noticed were that hypercholesterolemia accompanied lowered basal

metabolic rate in women but not in men, and that hypercholesterol was not associated with coronary sclerosis. Similar work by Chaikoff & Entenman (*J. Biol. Chem.* 166, 683) on turtles and turtle egg yolks indicated that the levels of the lipids were related to ovarian activity. Hueper (*Arch. Path.* 41, 139) and Popjak (*Biochem. J.* 40, 608) observed animals excessively fed cholesterol. The former described atheromatous changes that occurred in dogs under that dietary regime; the latter noted that in rabbits the diet induced a lipemia and that the fat reserves of the animals became depleted in three to 12 weeks, in spite of undiminished food intake. Bloch *et al.* (*J. Biol. Chem.* 162, 441) demonstrated with labeled element technic that acetate compounds were used in the synthesis of cholesterol by surviving liver.

In dogs strong doses of phospholipids diminished the respiratory quotient and greatly increased lipid consumption (Capraro & Pasargiklian—*Boll. soc. ital. biol. sper.* 20, 454). Other work on nonfat lipid fraction was of analytical nature: the constituents of egg yolk and calf brain cephalin fractions were determined (Burmester—*J. Biol. Chem.* 165, 565, 577); a micromethod for the partition of phospholipids into monoaminophosphatides and sphingomyelin was developed (Schmidt *et al.*—*Ibid.* 166, 505); the isolation and properties of dipalmityl lecithin from lungs was recorded (Thannhauser *et al.*—*Ibid.* 669).

The fundamental knowledge on physiological oxidation of fats continued to accumulate. Lehninger (*J. Biol. Chem.* 164, 291; 165, 131), from studies using liver enzyme suspensions, developed data which supported the hypothesis that oxidations of fat acids and of pyruvate were similar, for both were converted to two-carbon fragments. The quantitative study of the oxidation showed that all the carbon of the oxidized fat acids was recovered as acetoacetate in the absence of oxalacetate or oxalacetate precursors. In the presence of the latter, part of the carbon was recovered as citric,  $\alpha$ -ketoglutaric, and succinic acids; in such amounts that were accounted for by the Krebs tricarboxylic acid cycle. When using an enzyme suspension from rat heart muscle in concordant tests, acetoacetate did not accumulate. This fact and an accumulation of succinate were interpreted to suggest the Krebs cycle as the major path of oxidation in the heart. A difference in the oxidation mechanism in various tissues was also evident in the work of Breusch & Tulus (*Enzymologia* 11, 169, 352; *Arch. Biochem.* 11, 499), who studied the breakdown of keto fatty acids. The tissues were classified into two groups: (1) liver, kidney, muscle, brain, and pancreas metabolized  $\alpha$ - and  $\beta$ -keto acids; and (2) lung, spleen, placenta, and embryonic muscle were unable to do so. A difference in the oxidative course in different tissues was evident in the work of Medes *et al.* (*J. Biol. Chem.* 162, 1) who reported that ketone bodies were apparent when kidney tissue oxidized acetates, but in heart muscles oxidation occurred without conversion to ketone bodies. Another study by the same group (Weinhouse *et al.*—*Ibid.* 166, 691) confirmed the participation of the tricarboxylic cycle in fat acid metabolism as was also indicated by Lehninger (*op. cit.*). The technic in the last case included the use of easily traced labeled elements. Working along these lines, Tarantino (*Boll. soc. ital. biol. sper.* 21, 130) suggested that various fat acids also behave differently. Guinea pig tissues consumed 90% oxygen

and keto bodies formed in the presence of palmitic acid; with oleic acid no keto acids developed and oxygen consumption was 150%.

In a study of fat acid metabolism in rat muscle, Ehrlich & Waelsch (*J. Biol. Chem.* 163, 195) administered deuterium oxide in drinking water. The rapid appearance of deuterium in the muscle fat acids rather than in the fatty aldehydes was interpreted to suggest that the fatty aldehydes were not involved in synthesis or transport of fat acids. In investigations on rate of oxidation of fat acids by plant juices, Süllmann (*Experientia* 1, 323) suggested that the observations might have relationship to the metabolism of lipids.

THE LIPIDS UNDER PATHOLOGICAL CONDITIONS. Tuft & Tumen (*J. Am. Med. Assoc.* 130, 624) recorded five case reports of patients with gastro-intestinal symptoms in which they suspected some unexplained intolerance of fats.

Man *et al.* (*J. Clin. Investigations* 24, 623) described the effect of various hepatic diseases on the lipids of serum and liver. Hoagland *et al.* (*Am. J. Pub. Health* 36, 1287) demonstrated that in infectious hepatitis a high-fat high-protein diet was superior to elimination of fat or low-fat regimes as was commonly prescribed. Hoffmann *et al.* (*Rev. med. Chile* 73, 392) observed no differences in metabolic rate in rats fed abnormally high fat diets; however, these diets considerably reduced the increase in metabolic rate that was obtainable by thyroxine injections.

Fat metabolism during anoxia was found to be abnormal. Hove *et al.* (*Arch. Biochem.* 8, 395) suggested that the decrease in survival time of rats on high-fat diets under these conditions might have been due to decreasing the animals' respiratory quotient. Under the same conditions, MacLachlan (*Proc. Exptl. Biol. Med.* 63, 147) observed that fat caused an initial acceleration of the emptying of the stomach. Excessive fat in the diets increased the susceptibility of animals to benzene poisoning (Li *et al.*—*Am. J. Physiol.* 145, 158, 166). Past work has shown that dietary fats accelerate carcinogenicity. The most recent work (Kline *et al.*—*Cancer Research* 6, 1. 5) demonstrated a difference among fats in this respect. The incidence of liver tumors in rats fed 0.06% *p*-dimethylaminoazobenzene was 23% when the diet was low in fat, as contrasted to 94, 53, 100, and 87% when the diet contained respectively 5% corn oil, 5 olive oil, 20 corn oil, or 20 Crisco or lard.

Many reports in the past related that cod-liver oil added to a vitamin E deficient rat diet caused abnormal alterations in adipose tissue. Recently a histological description of these abnormalities was published (Mason *et al.*—*Anat. Record* 94, 265). In connection with arterial lesions occurring in dogs fed cod-liver oil, Holman & Swanton (*Proc. Soc. Exptl. Biol. Med.* 63, 87) noticed that the lesions were observed only after renal function was disturbed. They emphasized the role of the kidney in fat metabolism in this connection. According to Reichsman (*Science* 104, 64) the life of animals with chronic hypertension was prolonged by administering oxidized fish oils.

A critical discussion of the endocrine aspects of obesity contained an interesting view of abnormal fat metabolism (Goldzieher—*Am. J. Digestive Diseases* 13, 40).

Acceleration of fattening by use of hormones in commercial poultry production was just recently in-

augurated. New publications described the effects of estrogens on gains in weight, fat deposition, and market quality of cockerels (Sykes—*Poultry Sci.* 24, 542; Sturkie—*Ibid.* 25, 531) and an assay method for determining the fattening potency of orally administered estrogens (Jaap—*Okla. Acad. Sci. Proc.* 25, 37).

FAT SOLUBLE VITAMINS. This year, as in the past, only a small part of the fat soluble vitamin literature will be cited. That is, the scope of the Review is limited to distribution of the vitamins in oils and their analyses.

The major portion of the activities on determination of vitamin A dealt with spectral procedures. Rawlings & Wait (*Oil & Soap* 23, 83) determined the variations in results of the method as affected by different solvents and temperatures, and they suggested that a suitable standard was necessary. The Vitamin Committee of the American Oil Chemists' Society (Embree—*Ibid.* 275) recommended a solution of vitamin A in vegetable oil as a reference standard and outlined a tentative method for the analytical procedure. The data of Caldwell (*J. Biol. Chem.* 166, 565) on the spectral absorption of various carotenoids were of fundamental interest to the determination of vitamin A. A procedure by Awapara *et al.* (*Science* 104, 602) contained a chromatographic step to concentrate the vitamin A for the final spectroscopic determination. This improved accuracy when the amount of vitamin was small. The methods of Brew & Scott (*Ind. Eng. Chem., Anal. Ed.* 18, 46) for mixed feeds and feedstuff samples and Thompson *et al.* (*Ibid.* 113) for egg samples contained the same step. Another procedure for eggs combined spectrophotometric and fluorometric methods, designed to eliminate recourse to saponification and solvent partition (Dutton & Edwards—*Ibid.* 38). Other spectroscopic methods that were published presented details or notes for the determination of the vitamin in fish oils (Morton & Stubbs—*Biochem. J.* 40, lviii, lix) and margarine (Goodwin & Morton—*Analyst* 71, 15; Bowen *et al.*—*Ibid.* 20).

The new methods for determining vitamin A in livers were a micromethod suitable for biochemical study of the vitamin storage (With—*Biochem J.* 40, 249) and a rapid colorimetric control procedure using the Carr-Price reaction (Gallup & Hoefler—*Ind. Eng. Chem., Anal. Ed.* 18, 288). A new reagent for colorimetric determination of vitamin A was prepared by vacuum distillation of glycerol dichlorohydrin with antimony trichloride (Sobel & Werbin—*Ibid.* 570; *Federation Proc.* 5, 155).

A nation-wide survey of the vitamin potency of market butter revealed that 36% of all the creamery butter was "winter" butter of average vitamin A potency of 11,200 international units per pound; the remainder was "summer" butter and it contained an average of 18,000 international units (Maynard *et al.*—*U. S. Dept. Agr. Misc. Pub.* 571, 14 pp.). Similar reports, but of limited scope, were made for the states of Kansas (Parrish *et al.*—*J. Dairy Sci.* 29, 91) and Washington (Ashworth *et al.*—*Wash. Agr. Expt. Sta. Bull.* 466, 8 pp.). A somewhat similar report on the vitamin content of milk in France, contained results as affected by breed of cattle, lactation period, season, and feed (Citeaux—*Ann. agron.* 15, 529).

Jones (*Biochem. J.* 39, 324), in an investigation of the biological assay of vitamin D<sub>3</sub> with chicks, re-



corded that the bone ash criterion was most reliable, but for routine assays weight and tarsal-metatarsal distance were more economical and simpler. In an effort to develop a colorimetric vitamin D method Schaltegger (*Helv. chim. acta* 29, 285) studied the colors produced by 21 aldehydes with ergosterol and vitamin D<sub>2</sub> and chose anisaldehyde, vanillin, *p*-cumin-aldehyde, furfural, and 4-hydroxy-1-naphthaldehyde as giving compounds with the most promising absorption maxima. With a single aldehyde, the possible error in a vitamin D assay was about 20%; this was reduced to about eight per cent by using all five of the above aldehydes. A spectroscopic procedure for estimating vitamin D in oils by DeWitt & Sullivan (*Ind. Eng. Chem., Anal. Ed.* 18, 117) depended on extracting the unsaponifiable, separating vitamin D from vitamin A chromatographically, and measuring the absorption maximum at 500 m $\mu$  of the color produced by reacting the vitamin D with a modified antimony chloride reagent.

In regard to determination of vitamin E in oils, a new modification for both the *a,a'*-dipyridyl-ferric chloride (Tosic & Moore—*Biochem. J.* 39, 498) and the fluorometric (Lieck & Willstaedt—*Svensk Kem. Tid.* 57, 134) methods comprised the removal of interfering material and negligibly active material by adsorption on alumina. Quaife & Harris (*Ind. Eng. Chem., Anal. Ed.* 18, 707) recommended molecular distillation as a means of concentrating vitamin E for its chemical determination.

#### Characteristics and Composition

**METHODS OF ANALYSIS.** Sampling procedures for oleaginous material in tung fruit were corrected so they would be more representative. One hundred samples of the fruit were necessary to obtain values within 0.5% of the true oil content, and 400 were necessary to bring the values within 0.25% (Holmes & Pack—*Proc. Am. Tung Oil Assoc.* 1946, 40). Similar work on drilled samples of fish livers indicated that 100 cores were necessary to give vitamin results reproducible within five per cent when sampling over 2,000 cans of livers (Sanford & Bucher—*Ind. Eng. Chem., Anal. Ed.* 18, 269). A method of properly installing and using bleeders for obtaining representative samples from bulk oil was described (Tomkins—*Oil & Soap* 23, 35).

An investigation of the determination of moisture in cottonseed and its products indicated that more drying should be specified in the American Oil Chemists' Society official method (Hoffpauir & Petty—*Oil & Soap* 23, 285). This work included data showing the time necessary to dry the various products at 101° in both vacuum and forced draft ovens. The results were confirmed by the Society in collaborative work and procedures were modified accordingly (Hopper *et al.*—*Ibid.* 381).

Studies of the fat determination methods for technical control work reflected a desire for rapid procedures. For fat in fish, hydrochloric acid digestion (Voth—*J. Assoc. Official Agr. Chem.* 29, 46) and rapid drying first at 100° for 15 minutes and then to constant weight at 150° (del Val—*Inst. españ. oceanograf., Notas y resúmenes Sér. II, No. 121, 7 pp.*) were used as pretreatments for solvent extraction, with results agreeing well with methods which were standard but more tedious and time consuming. A modified Babcock butterfat test was applied to the

determination of fat in dried eggs (Paley & Rubin—*Food Industries* 18, 1194). An analytical method for the simultaneous determination of fat and sugar depended on alcohol extraction and separation of fat from sugar in the dried extract by the use of ether (Terrier—*Mitt. Lebensm. Hyg.* 36, 398). The Mojonier method was considered accurate for testing 80% cream (Heinemann—*J. Dairy Sci.* 29, 689). The same report recommended the Kohman calculation method for a quick procedure on which to base purchases. The calculations are made from a cup moisture determination and an assumption that the curd is about equivalent to 10% of the moisture. Frampton & Webber (*Oil & Soap* 23, 318) made quantitative studies on various methods for analyzing cottonseeds for fat. The refractive index method did not agree well with other procedures. The composition of petroleum ether extract varied from specimen to specimen, and heating the seed reduced the amount of extract. Absorption spectra of extracts obtained with different solvents showed qualitative differences in the material extracted. Some of this is confirmed in Rewald's (*Ibid.* 19) work on coffee, where different solvents extracted different amounts of phosphatides and nearly all the phosphatides disappeared on roasting. The report of the Soybean Analysis Committee of the American Oil Chemists' Society considered the methods now used for soybeans as satisfactory (Milner—*Ibid.* 288).

Fat extraction methods for biochemical materials contained some innovations. Fowweather & Anderson (*Biochem. J.* 40, 350) mixed feces with plaster of Paris before extraction in order to permit determinations for both split and unsplit fat. The technic minimized hydrolysis. The principle of gravimetrically or volumetrically determining free fat acids after saponification and extraction was recommended for limited samples of biological fluids and tissues (Devis—*Bull. soc. chim. biol.* 26, 232; Schmidt-Nielsen—*Compt. rend. trav. lab. Carlsberg Sér. chim.* 24, 233). The procedure was applicable to detecting small amounts of fats or fat acids on textiles (Hoffpauir & Kettering—*Am. Dyestuff Repr.* 35, 265). A similar procedure for gelatin contained a step in which the free fat acids were adsorbed on alumina and extracted from the adsorbent (Abribat—*Sci. ind. phot.* 14, 1). In flaxseed breeding work, where meager samples are used, the seeds were ground in a small Wiley mill and extracted with petroleum ether (Schricker—*Oil & Soap* 23, 91). A new Soxhlet type laboratory oil extractor had a capacity of 25 to 30 pounds of raw material; thus, large samples of fat are obtainable for research (Salkin & Kaye—*Ind. Eng. Chem., Anal. Ed.* 18, 215).

A method for the determination of the oil content of a varnish consisting of phthalate resins depended on the fact that phthalic acid is insoluble in benzene while resin and fat acids are soluble (Schrijver—*Verfkroniek* 17, 28). The amount of rosin acids and fat acids in tall oil can be determined by potentiometric titration (Harva—*Finnish Paper Timber J.* 27, 253).

A colorimetric method was found suitable for detecting and quantitatively determining fat acids or their esters in rust preventives, in lubricants, on tin plate, etc. (Hill—*Ind. Eng. Chem., Anal. Ed.* 18, 317). It was based on the formation of hydroxamic acid from the fatty esters by reaction with hydroxylamine hydrochloride in alkaline media; and a stable red-

colored complex of ferric hydroxamate is formed on addition of an acidified solution of alcoholic ferric perchlorate.

Known methods of recording color of oils were compared on oil samples from grayfish livers that have been incubated at 90° F. for different periods of time (Bucher *et al.*—*Oil & Soap* 23, 212). Readings with a colorimeter and with a Beckman spectrophotometer followed closely the order found by visual comparison. For any one oil, three transmission readings at such wave lengths as 500, 600, and 700  $m\mu$  were considered adequate. Ciusa (*Ann. chim. applicata* 35, 128) recommended the microphotometer of Haschek with employment of three color filters for determining both fluorescence and colors of oils. After much collaborative work, an American Oil Chemists' Society Committee recommended grading color of oils by a photoelectric system as a replacement for the Lovibond system (Agee—*Oil & Soap* 23, 292).

Collaborative investigations on the standard refining test resulted in a recommendation that an excess of 0.1 to 0.2% of the 12° Baumé lye refining agent be used (Mitchell *et al.*—*Oil & Soap* 23, 252; Vollertsen *et al.*—*Ibid.* 71). New laboratory technic to replace the present refining test depended on determining the acidity and a new characteristic which was named the dilution value (Desnuelle *et al.*—*Inds. corps gras* 2, 240, 304). This latter was a function of the amount of gums in a crude oil and was measured by determining the amount of sample necessary to change abruptly the surface tension of a definite amount of refined oil under certain standardized conditions. This value for oils of various acidities was found to give results proportional to the refining losses. The procedure is also a useful criterion for evaluating the efficacy of refining methods.

The determination of gas or oxygen in butter is of interest where butter is preserved by dehydrating, degassing, and canning for export to tropical regions. Accordingly, Hills & Conochie (*J. Council Sci. Ind. Research* 18, 366) modified the Rahn & Mohr (*Milchw. Forsch.* 1, 211) apparatus and technic for air in butter so that both free air and gasses in solution are determined. The gas dissolved in the melted butter was released by reducing the pressure to a low value. The solubility of oxygen in fats was also determined at the same laboratory (Morell *et al.*—*J. Council Sci. Ind. Research* 19, 190). Butter saturated with air at 45° dissolved 2.55 milliliters of oxygen per 100 grams and when saturated with oxygen dissolved 12.3 milliliters. Lard saturated with air at 45° dissolved 2.62 milliliters of oxygen.

The new thermic information on fats and oils was developed both for characterization of the products and industrial application. Rapid chilling procedures were submitted to the American Pharmaceutical Association for acceptance as standard technic in the determination of melting points of fats and waxes (Copley—*J. Am. Pharm. Assoc.* 35, 78). The need for such standardization was evident in the discordant results obtained with fats, such as cacao butter chilled in various ways. The new data on specific heats of fats are of particular interest to engineers and processors (Clark *et al.*—*Ind. Eng. Chem.* 38, 350; Ordianz—*Food Industries* 18, 1869). A simple method for determination of the amount of crystallized fat in cream depended on the latent heat required to liquefy the fat (Adriani & Tamsma—*Verslag. Landb. Onder-*

*zoek.* No. 51, (6), G, 79). Bertram (*Chem. Tech.* 1, 101) developed data to show the relationship between heat of combustion and the iodine and saponification values of fats and oils. A thermal analysis on monoacid triglycerides led to the conclusion that these exist in three polymorphic modifications (Ravich *et al.*—*Acta Physicochim. U.R.S.S.* 21, 101, 321). A review on the consistency and plasticity of fats by Ripert & Sisley (*Inds. corps gras* 2, 140) covered the work published in this country during the war. Tabulated data on solubility of fat acids included the solubility of caprylic acid in water at 0 to 90° (Constable & Tegul—*Nature* 157, 735) and the solubility of several saturated fat acids in toluene, *o*-xylene, chlorobenzene, nitrobenzene, 1,4-dioxane, furfural, 1,2-dichloroethane, and nitromethane. The latter information should be applicable to work on liquid-liquid partition of fatty acids.

Trapeznikov (*Compt. rend. acad. sci. U.R.S.S.* 47, 275) measured the degree of effects in hydrated fat acid crystal-electrolyte systems. He elaborated on their polymorphism and the equilibrium of the monolayers, and he also measured the effects of thermal treatment and presence of electrolytes on melting points and on the kinetics of monolayer formation. The data of Douglas & MacKay (*Can. J. Research* 24A, 8) on surface tension of the slightly soluble normal fatty acids,  $C_7$  to  $C_{11}$ , and aqueous solutions of these, support belief in the existence of monolayers of closely packed molecules arranged with long axes perpendicular to the surface.

Filer *et al.* (*J. Am. Chem. Soc.* 68, 167) and Sidhu & Daubert (*Ibid.* 2603) established the x-ray diffraction patterns of several synthetic diacid diglycerides and triglycerides. Crystallization into either  $\beta$ - or  $\beta'$ -phase was found dependent on the rate of crystal growth. The information on mixed glycerides was used in establishing the identity of glycerides of natural origin. For example, it was applied to establish the existence of a symmetrical configuration for the 2-oleyl-distearin isolated from kokum butter. The latter was also confirmed by Lutton (*Ibid.* 676). The natural and synthetic glycerides were found to have three closely agreeing crystalline forms. Lutton (*Oil & Soap* 23, 265) also recorded the different x-ray diffraction patterns of the crystalline forms of oleic acid melting at 13° and at 16°. Sidhu & Daubert (*J. Am. Chem. Soc.* 68, 1975) recorded the x-ray diffraction characteristics of 1-monoarachidin. This completed the data for the homologs from  $C_{10}$  to  $C_{20}$  and showed the relationship of the spacings in this series. The data by Trillat & Brenet (*Compt. rend.* 222, 878) on films of  $\beta$ -chloroethyl esters of the  $C_{12}$  to  $C_{18}$  fat acids were interpreted to indicate that the chains were inclined at the base at an angle of about 72°.

The iodine value of fats from biological material has been determined on 10 to 100  $\mu g.$  samples by a modified Rosenmund-Kuhnenn method (Kretchmer *et al.*—*Arch. Biochem.* 10, 101) and by a modified Kaufmann bromine-combining method (Schmidt-Nielsen—*Compt. rend. trav. lab. Carlsberg Sér. chim.* 25, 87). The excess absorption of iodine by castor oil in rapid methods, that has been attributed to the effect of the hydroxyl groups, was obviated by blocking the hydroxyl groups with the propionyl radical (Skell & Radlove—*Ind. Eng. Chem., Anal. Ed.* 18, 67). The Wijs method was used. Iselin (*Mitt. Lebensm. Hyg.* 36, 377) favored determination of fat acid composition



FAT ACID COMPOSITION

Oil or Fat Source	Common Saturated Acids			Common Unsaturated Acids			Other Fat Acids
	C <sub>14</sub> Myristic	C <sub>16</sub> Palmitic	C <sub>18</sub> Stearic	C <sub>18</sub> (-2H) Oleic	C <sub>18</sub> (-4H) Linoleic	C <sub>18</sub> (-6H) Linolenic	
<i>Amaranthus gangeticus</i> seed <sup>4</sup>			2.2	43.7	27.3		
Bacury seed kernel <sup>3</sup>		20.8	2.2				
<i>Platonia insignis</i>		28	28		4		
Chaulmoogra seeds of Congo <sup>5</sup>		7					Hydrocarpic 6.5, chaulmoogric 38, goric 8
<i>Cadocoba glauca</i>		16					Hydrocarpic 18, chaulmoogric 31, goric 5
<i>Cadocoba wataitichii</i>		13					Hydrocarpic 37, chaulmoogric 6.5, goric 0.1
<i>Elyonocarpus anthelminticus</i>		3					Hydrocarpic 43, chaulmoogric 11.8, goric .6
<i>Elyonocarpus wightianus</i>		11					Hydrocarpic 11, chaulmoogric 51, goric ....
<i>Limbeckia dentata</i>		8.7	9.5	38.45	27.6		C <sub>20</sub> 3.7
<i>Cryptostegia grandiflora</i> seed <sup>1</sup>							
Fissang nut kernel <sup>18</sup>		9.5	1	9.5	26	54(a)	
<i>Ricinodendron africanum</i>		4		20-3.0	15-20		C <sub>22</sub> (-2H) 35-4.5, C <sub>26</sub> (-2H) remainder
Hare's ear mustard seed <sup>14</sup>							
<i>Coringia orientalis</i>		15	9	27	29	20(a)	
Krobanko nut kernel <sup>18</sup>							
<i>Telfairia occidentalis</i>		1.9	1.3	37.6	56.9		C <sub>8</sub> 2.2, C <sub>20</sub> 0.1
Lettuce seed <sup>20</sup>		7.1	17.7	38.4		36.8	
<i>Lactuca scariola</i>		8.7	8.9	31	51	0.4	
<i>Mimosa pudica</i> seed <sup>24</sup>							
Neou nut kernel <sup>18</sup>		12	2	40	15	31(a)	
<i>Parinarium macrophyllum</i>		1.5		29	16	1	C <sub>24</sub> 1.5, C <sub>22</sub> (-2H) 51
Rape seed (French) <sup>27</sup>							Lower sstd. 1.3, higher sstd. 0.5, lower unsstd. 0.4
Saffron thistle seed <sup>28</sup>		7.5	6.6	12.4	70.4	0	
<i>Carthamus lanatus</i>		11.0	17.9	57.8	13.3		
<i>Sideroxylon tomentosum</i> seed <sup>29</sup>		1.8	1.9	49.7	46.6		
<i>Solanum nigrum</i> seed <sup>30</sup>		0.1	3.5	48.6	20.1		
<i>Solanum pseudocapsicum</i> seed <sup>31</sup>							
Sorghum grain (Av. from 14 varieties) <sup>32</sup>		7.8	4.7	39.5	46.5		
<i>Andropogon sorghum</i>							
Sweet (Kharbuza) melon seed kernel <sup>33</sup>	1.1	7.3	0.2	43.1	45.1		C <sub>6</sub> 1.0, C <sub>8</sub> 2.0
<i>Cucumis melo</i>							
Sweet (Sarda) melon seed kernel <sup>33</sup>	2.0	2.0	5.5	32.9	55.5		C <sub>20</sub> 0.9
<i>Cucumis melo</i>		22.4	15.7	28.1	33.0		C <sub>20</sub> 0.8
<i>Terminalia belerica</i> seed <sup>37</sup>							
Tung seed							
<i>Aleurites Fordii</i> (American) <sup>8</sup>		5.5		9	8.5	82(a)	
<i>Aleurites Fordii</i> (Chinese) <sup>8</sup>		4		4	10	77(a)	
<i>Veronica anthelmintica</i> seed <sup>36</sup>	7.4	7	5.9	5.7	9.6		Vernolic 6.4
Watermelon seed (India) <sup>11</sup>							
<i>Citrullus vulgaris</i>	0.2	7.6	6.1	35.3	48.7		C <sub>8</sub> 0.2, C <sub>10</sub> 1.1, C <sub>12</sub> 0.8

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of fats with methods based on iodine and thiocyanogen values, for their results checked those obtained by a selective oxidation technic. Lambou & Dollear (*Oil & Soap* 23, 97) cautioned that the lead thiocyanate reagent must be prepared with care and be of high purity to be stable and give dependable results.

Mareali & Rieman (*Ind. Eng. Chem., Anal. Ed.* 18, 144) have devised a microprocedure for the determination of the saponification values of fats, which is a modification of a recent macromethod (Rieman—*Ibid.* 15, 325) in which titration was done potentiometrically or with use of double indicators. The necessity for a blank determination was eliminated. The saponification technic of the method was further modified by Ketchum (*Ibid.* 18, 273) for semimicro-determinations on samples too difficult to saponify by the above procedure. A new method for the determination of 1-monoglycerides in fats depended on the action of alkali periodates noted by Malaprade. Accordingly, the hydroxyl radicals were oxidized with the formation of two moles of corresponding aldehydes (Ivanoff—*Bull. matières grasses inst. colonial Marseille* 29, 45).

Several papers on physical and chemical tests on fats were of general interest. Baum (*Inds. corps gras* 1, 40) defined and interpreted the significance of the most common tests. Mesnard (*Corps gras savons* 2, 4) reviewed the various reactions involving the double bonds that are used in laboratory testing and industry. Laurent (*Inds. corps gras* 2, 310) described the procedures used for iodine and saponification values at the Hilditch laboratory. Carrière (*Ibid.* 1, 76) recommended procedures for determining iodine, thiocyanogen, and diene values of blown oils. These were slight modifications of known methods. Paquot (*Ibid.* 2, 272) discussed the use of index of refraction for determining oil in seeds, glycerol in water solution, iodine value, and structure of certain acids. The characteristics of ewe and cow butter were discussed from the point of view of analytical differentiation (de Mingo & Calles—*Ion* 6, 299). A similar report on cow butter discussed the significance of the tests and the effects on them of season and fodder (Petersen—*Fette u. Seifen* 51, 25). The characteristics of neat's foot oils and marrow oils were studied with the viewpoint of establishing specifications for the Brazilian products (Borges—*Rev. alimentar* (Rio de Janeiro) 9, 5).

Many new improvements were developed in the technics for determining the composition of fats and oils. Smith & Brown (*Oil & Soap* 23, 9) evaluated the chain lengths of the acids in menhaden oil by distillation of the hydrogenated methyl esters and analyzing the fractions. This work supplemented earlier analyses of the oil by crystallization of the methyl esters, followed by distillation of the fractions (*Ibid.* 22, 321). Hilditch and coworkers (*J. Soc. Chem. Ind.* 65, 8, 74) used this procedure in combination with the spectrographic analyses for polyunsaturated acids and found that none of the eleostearic acid-containing oils that they analyzed contained nonconjugated linolenic acid. Analyses were recorded in this work on linseed, wheat germ, rubber seed, American tung, Chinese tung, essang, neou, and krobanko oils. Also in connection with crystallization separation, Bailey *et al.* (*Oil & Soap* 23, 201) published some preliminary studies on phases of fat-solvent systems. Such basic information should raise

fractional crystallization studies above an empirical basis.

Beadle's (*Oil & Soap* 23, 140) review on applied ultra-violet spectrophotometry contains absorption curves of fats and individual fat acids, before and after isomerization. The simplicity and utility of the technic for studying double bonds and for determining both the composition of natural fats and changes in the double bond system (conjugation) were made evident. Its utility was exemplified in following the progress of the preparation of pure linoleic and linolenic acids (O'Connor *et al.*—*Ibid.* 5); it was used to prove that commercially produced linseed oils do not contain conjugated systems (Nesbitt & Painter—*No. Dakota Agr. Expt. Sta. Bimonthly Bull.* 6, No. 6, 31); and it was instrumental in the identification of *cis,cis*-9,12-linoleic and *cis,cis,cis*-9,12,15-linolenic acids in beef tallow (Knight *et al.* *J. Biol. Chem.* 164, 477). Raman spectra studies by Dupont & Yvernaud (*Bull. soc. chim.* 12, 84) of unsaturated fat acids also suggested that natural oleic, linoleic, and ricinoleic acids were all in the *cis*-form. In this work the C<sub>8</sub> and C<sub>9</sub> fractions from butter and a C<sub>10</sub> acid from castor oil gave Raman lines corresponding to the presence of a triple bond. In similar work, van den Hende (*Bull. soc. chim. Belg.* 54, 88) demonstrated that the double bond of unsaturated acids was indicated clearly and that the technic could be used to detect the lower fat acids. A failure to detect a relationship between absorption coefficients and dielectric constants of fat acids in solution was attributed by Stepanenko & Novikova (*Acta Physicochim. U.R.S.S.* 20, 653) to a molecular association of some sort. All the reports of spectrometric investigations contained data of particular interest as reference material.

A novel procedure for analysis of mixtures of geometric isomers of fat acids was based on the observation that in addition-reactions involving the double bond the *cis*-isomer usually reacted much faster than the *trans*-form (Connor & Wright—*J. Am. Chem. Soc.* 68, 256). When methoxymercuration was applied until one-third complete, followed by analysis with dithizone reagent, the error in composition was only four per cent.

The question of type of fat acid distribution in the glycerides was part of several analytical reports. In lard and tallow, according to Riemenschneider *et al.* (*Oil & Soap* 23, 276) and Luddy & Riemenschneider (*Ibid.* 385), a random distribution of fat acids in the glycerides is found. The percentage of triunsaturated (14.7%) and disaturated (45.9%) glycerides of tallow was much higher than those (1.9 and 25.7%, respectively) of lard. The triunsaturated glycerides (16.0%) in lard were significantly higher than the literature had previously indicated. In this work crystallization procedures for determining trisaturated constituents were favored over oxidation technic because they saved time and results were comparable. The glycerides of Sarda melon seed oils (Ahmad & Dhingra—*J. Indian Chem. Soc.* 22, 337) and *Terminalia belerica* (Kantha *et al.*—*Proc. Indian Acad. Sci.* 23A, 283) approximated the rule of "even" distribution, which is not met in many seed oils. Others for which the distribution of fat acids were recorded were kurchi seed (Irani—*Current Sci.* 15, 161, 191), bacuri seed (Chaves & Pechnik—*Rev. quim. ind.* (Rio de Janeiro) 15, No. 165, 16), and *Solanum nigrum* (Singh & Kumar—*Proc. Indian Acad. Sci.* 22A, 310).

Analyses of the glycerides of milk fat indicated that the acid distribution tended to follow more closely the "even" than the "random" hypothesis (Jack *et al.*—*J. Biol. Chem.* 162, 119). A review by Desnuelle (*Corps gras savons* 1, 100) on determining the glyceride structure of fats, emphasized the work of Hilditch and his school.

Some analytical reports made particular reference to the less common fat acids. Baudart (*Bull. soc. chim.* 11, 174) isolated 11,14-eicosadienoic and 8,11,14-eicosatrienoic acids from fish oils. The technic comprised fractionation by crystallization, bromination of selected fractions, refractionation, and debromination. Baudart (*Compt. rend. trav. faculté sci. Marseille* 1, 79, 86, 127) also isolated and determined the structure of the elupanodonic acid of shark liver, *Carcharodon carcharias*. According to Grindley (*J. Soc. Chim. Ind.* 65, 118), the presence of C<sub>20</sub> to C<sub>24</sub> saturated acids in Sudan Caesalpinioideae seed oils appeared to be characteristic of the entire leguminosae family. A newly reported natural fat acid, 10-hydroxy-8-octadecenoic, was found to be present in *Vermonia anthelmintica* seed oil to the extent of 62.4%; Vidyarthi (*Patna Univ. J.* 1, 51), the discoverer, named it "vernolic" acid. Bergström & Trobeck's (*Svensk Papperstidn.* 49, 330) analysis of tall oil failed to confirm the presence of carnaubic acid as was previously reported. An analysis of human milk fat by Brown & Orians (*Arch. Biochem.* 9, 201) indicated that it was more similar to human body fat than it was to a typical butterfat. Only small traces of acids lower than C<sub>10</sub> were found, the presence of C<sub>10</sub> to C<sub>18</sub> mono-enoic acids was verified, and small amounts of tri- and di-enoic C<sub>18</sub> acids were present although normal linoleic was negligible in quantity. Bergström *et al.* (*Nature* 157, 306) observed that the unsaturated acids occurring normally in the animal body had a strong bactericidal action on *Mycobacterium tuberculosis*.

Much other information on characteristics and fat acid composition of fats and oils has been most conveniently entered in tables appended to this section of the Review.

In regard to the unsaponifiable material in fats, two groups of collaborators (Mehlenbacher *et al.*—*Oil & Soap* 23, 20; Kirsten *et al.*—*J. Assoc. Official Agr. Chem.* 29, 248) reported that the British Society of Public Analysts' method gave excessive values. The improvement in the method suggested by Sylvester *et al.* (*Analyst* 70, 295) involved chromatographic removal of contaminating free fat acids from the extracted matter before the evaporation of the solvent.

The activity on determination of gossypol in cottonseed oil and products centered on the method based on spectral absorption of dianilino derivative of gossypol (Smith—*Ind. Eng. Chem., Anal. Ed.* 18, 41, 43; Smith & Halverson—*Oil & Soap* 23, 361; Boatner—*Ibid.* 235). A newly proposed method depended on the ability of gossypol to reduce the copper of Fehling's solution (Podol'skaya—*J. Applied Chem.* (U.S.S.R.) 17, 657). Velluz & Lederer (*Bull. soc. chim. biol.* 27, 211, 218, 419) applied chromatographic technic to separate and determine the sterol constituents of wool fat. Williams (*Analyst* 71, 259) reviewed the use of the technic in analysis of fats, particularly the unsaponifiable constituents. Cholesterol in plasma has been determined photometrically by a modified

Liebermann-Burchard reaction (Saifer & Kammerer—*J. Biol. Chem.* 164, 657).

Giral *et al.* (*J. Biol. Chem.* 162, 55, 61), in working with insect fats, observed that sulfur compounds appeared in the fats of the family, Catantopidae. According to von Fellenberg (*Mitt. Lebensm. Hyg.* 36, 355), fresh rapeseed oil contains 1.26-3.12 mg. % sulfur, but all of this appears in the residues which settles out after five months' storage. In the case of the seed oils of Cruciferae (André & Kogane-Charles—*Compt. rend.* 218, 850), the sulfur is present as volatile organic sulfur compounds and these are said to be responsible for the odors.

Several communications dealt with detecting adulteration of oils or identifying tests. Chromatography was applied to estimate squalene in oils by a procedure intended for detection of olive oil in oil mixtures (Fitelson—*J. Assoc. Official Agr. Chem.* 28, 247). In another investigation (da Silveira—*Rev. alimentar* (Rio de Janeiro) 9, No. 6, 6, 86) olive oil could not be distinguished from pataúá oil by the Bellier value, *i.e.*, temperature at which the free fat acids begin to crystallize from a solution. The author recorded the Bellier values of many Brazilian edible oils. Mora (*Afinidad* 23, 362) proposed that the term "pure olive oil" be applicable to solvent extracted olive oils; heretofore the title was limited to pressed oils, and application of the name to the solvent extracted product was considered adulteration. Sen (*Indian Med. Gaz.* 81, 126) described several color reactions for detecting adulteration of mustard oil with argemone oil. The most promising were based on colors developed by copper acetate and ferric chloride according to special procedures. Zeidler (*Farben-Ztg.* 47, 294) reviewed several tests and their limitations for detecting tung oil in mixtures. The appearance of brown polyhedral figures on an oil mixture when concentrated sulfuric acid is dropped on it indicated the presence of tung oil. Although the test could not detect less than a minimum of 10% tung oil in mixtures, it was considered quite useful in industry. A procedure for detecting the adulteration of an oil by dyeing with FD&C Yellow Nos. 3 and 4 depended on saponification of the oil, extraction of the dye with ether, and titrating with standard titanium chloride solution (Newburger—*J. Assoc. Official Agr. Chem.* 28, 636).

### Detergents

SOAP MANUFACTURE. The year's developments in soap manufacture were minor improvements in common processes. The balling tendency of granular soaps when added to warm water was reduced by manufacture from fat stock containing increasing amounts of fat acids of the coconut oil group (Byerly—*U. S.* 2,388,632). Of two patented soaps containing a considerable amount of tall oil or rosin, one formula contained 20-30% tetrasodium pyrophosphate as a builder (Van Zile—*U. S.* 2,402,473) and the other contained a mixture of soluble inorganic alkaline salts and bentonite (Garverich—*U. S.* 2,411,090). Schmidt & Edwards (*U. S.* 2,393,421) discovered that sperm oil could be wholly converted to soap by saponification with anhydrous alkali at 275°. Under these conditions the fatty alcohols of the oil were transformed to soap with the release of hydrogen. A war surplus aluminum soap incendiary product, "Napalm," was converted to regular soap by hy-

drolisis, recovery of acids, and saponification (Anon.—*Soap Sanit. Chemicals* 22, No. 12, 49). One Russian soap patent (Molchanovskii—*U.S.S.R.* 64,520) recommended saponifying the whole tea and tung oil seeds. In a comparison of preliminary deglycerination versus direct saponification in soap technic, Bergeron (*Inds. corps gras* 2, 19) favored the latter because it was most economical and gave higher yields of soap and glycerol. The use of precipitating agents such as calcium acetate, ground wood, and clay for carrying down impurities of soaps has been patented (Mandrups Machinfabrik Akt.—*Dan.* 62,868). The novelties in continuous systems of soap making were improved mixing devices for the saponification zones of the Sharples equipment (Sender & Jones—*U. S.* 2,397,161-2; Sender—*U.S.* 2,411,468-9) and a new continuous system in which strong lye was used to saponify the oil dissolved in relatively large amounts of inert solvent (Gunther—*U. S.* 2,401,756).

Cooling, drying, and molding of the soap were subjects of several patents. Special mixing equipment was designed for aerating and agitating the molten soap in the manufacture of floating products (Bodman—*U. S.* 2,398,776; Ittner—*U. S.* 2,403,925). A light bulky powdered soap was manufactured by obliquely impinging sprayed molten soap against a vibrating, hard surface (Vang—*U. S.* 2,392,072). A new spray dryer made use of compressed air to atomize the aqueous soap solution (Haaland—*Norw.* 68,477). Glossy soap bars were prepared by treating the bars with steam or hot water and drying in a current of air (Penny—*U. S.* 2,391,919; Bodman—*U. S.* 2,392,831). Soap cakes that could be readily partitioned contained planes of material dissimilar to soap and capable of reducing the natural cohesion of the soap (Bodman & Pease—*U. S.* 2,400,871).

Special soaps were improved by various methods. Small amounts of sodium silicate in liquid soaps inhibited the dissolution of glass by the product (Kranich—*U. S.* 2,402,557). Kolle (*Norw.* 68,892) maintained neutrality in soaps by adding two to three per cent alginic acid. Soday (*U. S.* 2,404,003-4) used certain tolyl ethyl alcohols as perfuming agents for soap. Insect infestation of a compounded powdered hand soap containing 50-60% corn meal was prevented by adding four per cent pine oil (Cotton & Frankenfeld—*J. Econ. Entomol.* 39, 419).

Several miscellaneous cleaners contained soap. Two scouring and cleansing devices were pads of vegetable fibers impregnated with soap, resins, and abrasives (Levine—*U. S.* 2,395,054; Rimer—*U. S.* 2,395,068). A new silverware polish comprised abrasive, soap, sodium carbonate, milk, butter, glycerol, and water (Dreyfus—*Can.* 436,697). A newly patented organic solvent cleaner contained a small amount of soap (Ratner—*U. S.* 2,411,938). Chlorinated solvents, glycol compounds, and dibutyl tartrate were suggested as aids for blending soap with organic solvents in industrial products. A dishwashing detergent comprised a mixture of sodium silicate, sodium phosphate, and/or sodium carbonate (Mathieson Alkali Works—*Brit.* 570,171). A detergent for tinware contained similar salts with tin and magnesium salts (Schwartz—*U. S.* 2,391,647).

Soap was used in several nondetergent preparations. A flotation agent for iron ore contained alkali, lignin sulfonate, fat or rosin acids, and soap (Clemmer & Rampack—*U. S.* 2,403,481). A paper size

contained soap, proteinaceous colloids, and resins (Luettgen—*U. S.* 2,399,748). A composition of animal glue and a small amount of soap was prepared for use to agglomerate solids in aqueous suspensions (Tutt *et al.*—*U. S.* 2,403,144). Soaps of C<sub>6</sub> to C<sub>10</sub> fatty acids were used as lubricants for drawing wire (Pape—*Ger.* 745,919 Cl. 23c).

**NONSOAP DETERGENTS.** A new procedure for manufacture of sulfonated cottonseed oil contained a preliminary chlorination step (Kapoor & Mathur—*J. Indian Chem. Soc. Ind. & News Ed.* 8, 94). The products thus prepared had a greater sulfate content, less unreacted fat, and less free fatty acids than a product obtained by treating the original oil with sulfuric acid.

Some investigators prepared new surface active compounds and recorded their properties. Dankova *et al.* (*J. Gen. Chem.* (U.S.S.R.) 15, 189) prepared several benzyl-diethyl fat acid ammonium bromides. These quaternary ammonium salts were water soluble, viscous, oily products with good foaming power. The quaternary compounds tested by Bhatt *et al.* (*J. Sci. & Ind. Research* (India) 4, 771) were fatty acid pyridinium and fatty acid quinolinium halides. The wetting power (sink test) of these increased with molecular weight and the wetting power of the former was greater than of the corresponding latter. Work done along this line by Hart & Niederl (*J. Am. Chem. Soc.* 68, 714) included the preparation of several fatty acid thiomorpholines and the recording of their physical characteristics.

Desseigne (*Inds. corps gras* 1, 136) determined the rates of hydrolysis at boiling of the normal C<sub>2</sub> to C<sub>12</sub> alkyl sulfate sodium salts in weak acid solution. The rate of hydrolysis in normal hydrochloric acid decreased gradually from C<sub>2</sub> to the C<sub>5</sub> product, then increased with each additional carbon atom in the compound. The results with sulfuric acid were similar in trend but lower. The presence of sodium sulfate reduced the rate of hydrolysis. Other data presented in this communication were the effects of temperature and of various concentrations of acids. The practical value of the information was that it could serve as a basis for the selection of raw materials for preparation of products to be used at specific pH's and temperatures.

Harris (*Oil & Soap* 23, 101) developed data for use as a basis for selecting builders for dodecyl sodium benzenesulfonate. Lather was improved and surface and interfacial tensions were decreased with sodium sulfate and magnesium chloride. Greatest improvement of detergency was with alkaline compounds. Soap acted as a synergist for the detergent, but in this case the detergency was decreased in the presence of sodium sulfate.

Some nonsoap detergent developments were on specific products. A patent on a bar form dealt with a method of physically working it and adjusting the moisture (Bodman—*U. S.* 2,407,647). The defatting power and turbidity point of a fatty acid sulfate shampoo product was reduced by increasing the amount of C<sub>5</sub> to C<sub>11</sub> sulfates in the mixture (Flett & Toone—*U. S.* 2,401,726). Ordinary soap was rendered more suitable for use in hard water by addition of fatty acid amides of an alkylolamine (Kroll & Weisberg—*U. S.* 2,404,298), lauryl alcohol and naphthalene sulfonates and starch (Blades—*U. S.* 2,407,130), and monofatty acid derivatives of sodium

phenolsulfonate (Flett—*Can. 433,360*). Three hand cleaners contained nonsoap detergents. A paste product contained heavy petroleum oil, water, sodium petroleum sulfonate, and nonabrasive absorbent material (Showalter—*U. S. 2,392,779*). A liquid product contained mineral oil, stearic acid, water, glycol esters of fatty acids, and glycol (Kleinicke—*U. S. 2,410,168*). The other material was paper associated with sulfate or sulfonate detergent (Cook & Cook—*Brit. 570,852*). The use of small amounts of sulfonated fats in inorganic detergent salt mixtures was patented (Hicks & Saunders—*U. S. 2,404,289*). A patented denture cleaner contained the synthetic detergent, sodium lauryl sulfo-acetate (Snell & Haeseler—*U. S. 2,409,718*). Two patented grease removers comprised organic solvent mixtures and sulfonated detergents (Donlan & Gathman—*U. S. 2,391,087*; Schwartz—*Brit. 560,690*). Reynolds & Rice (*U. S. 2,403,613*) used a mixture of *o*-dichlorobenzene and synthetic detergent to remove grease from concrete, wood, and tile surfaces. An aqueous phosphoric acid solution containing certain tertiary-alkylarylpolyglycols (Douty & Heller—*U. S. 2,396,776*) and a mixture of di-isobutyl phenyl diglycol ether sulfonate and triamylamine (Given—*U. S. 2,413,495*) were prepared for removing rust and grease from metals.

The synthetic surface active agents also were utilized for nondetergent purposes. A paper suggesting that wetting agents could be used as aids in precipitation and saponification reactions illustrated their use in one procedure for determining ethyl acetate in a mixed solvent (Losee—*Can. Chem. Process Inds. 30, No. 9, 90*). Wetting agents rendered plastic peanut butter inadhesive (Rosefield *et al.*—*U. S. 2,397,564*). Amine (Atwood—*U. S. 2,389,605*) and sulfated (Watson—*U. S. 2,403,251*) fatty derivatives were used as dispersing agents in the manufacture of protein plastics. A fluid hectograph composition containing a cation-active detergent could be applied to a backing which even contained greasy material (Bour—*U. S. 2,407,395*). A patented fire-extinguishing foam contained sodium fat acid sulfates, triethanolamine citrate, ethylene glycol, and water (Scheffan & Brown—*Can. 435,733*). The use of a mixture of water insoluble fatty amine and a cation dispersing agent as a pest control composition was patented (Littler—*U. S. 2,387,336*). Two bituminous emulsion formulas contained synthetic detergents as the emulsifying agents (Mikeska—*U. S. 2,389,680*; Buckley & Bly—*U. S. 2,392,457*).

The use of the synthetic detergent, "Tergitol," in road dust-laying oils was described in two patents (Butcher—*U. S. 2,399,464-5*). Dust-laying oils containing detergents also proved useful in medicine. Procedures for oil treating bedclothes to control respiratory infections were issued by the U. S. Navy Medical Research Unit No. 1 (*Science 104, 60*), Puck *et al.* (*Am. J. Hyg. 43, 91*), and Loosli *et al.* (*Ibid. 105*). Bayley & Weatherburn (*Can. J. Research 23F, 402*) discussed and made practical suggestions for applying the oils to woolen blankets. The authors of the papers made suggestions regarding the most efficient detergents for dispersing the oils. Two other medical uses suggested for synthetic detergents were for the stimulation of mucus secretion and inactivation of pepsin in the treatment of stomach ulceration (Shay *et al.*—*Science 103, 50*) and to delay resorption of subcutaneous injection of gonadotropins

(Bischoff—*Am. J. Physiol. 145, 123*). Several commercial synthetic detergents were recommended for use in a procedure to isolate coli phage from sewage (Kalter—*J. Bact. 52, 237*).

A nonsoap laundering method comprised washing in peptonized nonalkaline water and then in alkaline water (Nordfeldt—*Brit. 561,542*). One laundering agent patent dealt with refining saponins (Molchanov—*U.S.S.R. 64,689*). Pectin and mucilaginous substances were precipitated out by boiling with alum, and the saponins were recovered from solution by precipitation with alcohol.

PHYSICO-CHEMICAL STUDIES ON SOAP. Buerger (*Am. Mineral. 30, 551*) recorded x-ray data on crystals of sodium palmitates and sodium stearates, hemihydrates,  $\alpha$ -1:1 and  $\beta$ -1:1 acid:sodium soaps and discussed the layering nature of the crystals. Ryer (*Oil & Soap 23, 310*), on investigating the composition of the crystals that occur in various mixtures of stearic acid and sodium stearate, found individuals of 1:1, 2:3, and 1:2 of soap:acid mixtures, but no evidence of a 2:1 form. Jensen & Lingafelter's (*J. Am. Chem. Soc. 68, 1729*) x-ray data on sodium C<sub>12</sub>, C<sub>14</sub>, C<sub>16</sub>, and C<sub>18</sub> sulfonate hemihydrate crystals indicated that all have the same structure. The arrangement of the crystals was described. Ross & McBain (*Ibid. 547*) demonstrated that two forms of sodium oleate crystals could be distinguished by x-ray powder diagrams. They also recorded x-ray diffractions of hexanolamine oleate.

Walton (*J. Am. Chem. Soc. 68, 1180, 1182*) studied the activity of 1-*n*-dodecansulfonic acid in aqueous solutions and the effect of electrolytes on this activity. A decrease in temperature and increase in ionic strength promoted micelle formation. The activities at 0° did not agree with previously determined freezing point depressions. In the presence of chloride ion (hydrochloric acid) the activity of the compound decreased. Neutral salts increased the effect of hydrochloric acid. These observations were interpreted to indicate a displacement of hydrogen ions from the colloidal micelles. Similar work was carried out by Walton *et al.* (*J. Colloid Sci. 1, 385*) on dodecylbenzylidimethylammonium chloride and sulfamate. The activity in presence of hydrochloric acid suggested that, in the concentration range where micelles are fully established, about 60%, chloride ions are combined in the micelles of the dodecyl salts. Matalon (*Compt. rend. 222, 1213*) and Klevens (*J. Chem. Phys. 14, 567*) plotted the changes occurring in the surface tension and refractive indices of detergents at various concentrations and pointed out the characteristics of the curves which were related to micelle formation, foaming power, or stability. In a discussion of such data, Lachampt (*Bull. soc. chim. 12, 189*) explained the anomalous viscosity of potassium palmitate solutions as compared to potassium oleate on the basis of the Krafft point values.

Harkins and coworkers (*J. Colloid Sci. 1, 105, 469*; *J. Am. Chem. Soc. 68, 220*; *J. Chem. Phys. 13, 534*) studied the structure of soap micelles by means of x-rays. It appeared that the size of micelles of a mixture of soaps in solution was determined by the relative tendencies of the constituents to aggregate and that, if these tendencies of two soaps were dissimilar, the soap of lower aggregation value tended to act as a salt toward the other. In not too dilute solutions the orientation in the interfaces indicated



the existence of double layers of soap molecules with water layers between them, the micelles being four or more double layers thick. Their work on soap micelles in the presence of hydrocarbons showed that the area per soap molecule in the layer of soap was unchanged, whether the dissolved hydrocarbon layer had long molecules (lauryl mercaptan) or flat molecules (benzene); but spherical molecules (tetraethyl lead) had an effect on the area per soap molecule. The communications contained layer spacing data of micelles of several soaps in solutions with various hydrocarbons.

Several groups of investigators recorded characteristics of aqueous detergent-hydrocarbon solutions. McBain & Richards (*Ind. Eng. Chem.* 38, 642) observed that low-molecular-weight hydrocarbons readily solubilized, and the extent of solubility decreased with increasing molecular weights and volumes. Polar compounds dissolved more readily than nonpolar. In general, cationic detergents were more effective than anion-active products. There was a parallelism in behavior of various detergents differing only in degree. However, numerous specificities and structural influences, both of detergents and hydrocarbons, caused some irregularities. Vold & Philipson (*J. Phys. Chem.* 50, 39 and Doscher & Vold (*J. Colloid Sci.* 1, 299) developed phase relations and other physical data on behavior of sodium stearate with cetane and water. Miscibility, emulsion formation, and gelation influences were elucidated in regard to the characteristics of lubricating grease systems.

Physical, thermic, phase, and colloidal data were recorded for solutions of fatty acid derivatives of hexanolamine in benzene, cyclohexane (Gonick—*J. Colloid Sci.* 1, 339), and paraffin oil (Lachamp—*Compt. rend.* 220, 46, 317). The phase systems of aqueous hexanolamine oleate were also determined from data on freezing point, conductivity, index of refraction, transport, and x-ray diffraction (Gonick & McBain—*J. Colloid Sci.* 1, 127; *J. Am. Chem. Soc.* 68, 683; Ross & McBain—*Ibid.* 296).

Palit & McBain (*Ind. Eng. Chem.* 38, 741) demonstrated the utility of soaps in making organic liquids such as glycols miscible with hydrocarbon solvents. The potassium and sodium stearates were most effective for this purpose. The phase behaviors of sodium stearate, palmitate, and oleate in toluene, Nujol, xylene, and pinene were recorded (Hattiangdi—*J. Sci. & Ind. Research (India)* 4, 489; Adarkar & Hattiangdi—*J. Univ. Bombay* 14A, 23). Another paper in this series pointed out that the presence of several hydrocarbons had a synergetic effect on soap gels in pinene (Prasad *et al.*—*Proc. Indian Acad. Sci.* 22A, 320).

Ralston & Hoerr (*J. Am. Chem. Soc.* 68, 851, 2460) recorded the solubilities and electrical behaviors of hexyl- and dodecylammonium chloride in various dilutions of aqueous ethanol. The data were discussed in the light of the present micelle theory.

The mechanism of solubilization of water-insoluble dyes in soap solutions was investigated in several laboratories. McBain & Green (*J. Am. Chem. Soc.* 68, 1731) observed that the amount of hydrocarbon chain in the soap micelle increased slightly from caprylate to myristate, while the solubility increase was disproportionately greater. In all concentrations the addition of alkali or salt increased the solubility of the dye. This was interpreted to indicate increased asso-

ciation of the detergent. Corrin *et al.* (*J. Chem. Phys.* 14, 480) also made use of dye solubilization by detergents to study micelle formation and effect of electrolytes on the systems. The dye, pinacyanol chloride, exhibited an absorption band at 5500 Å which disappeared as the concentration of molecular soap increased. The effects of solubilization, micelle formation, etc., on various absorption bands in the presence of several detergents were discussed. Kolthoff & Johnson (*J. Phys. Chem.* 50, 440) used the amount of dye solubilized at 50° per mole of micellized soap as a means of evaluating the critical concentrations below which micelles were not present.

Several physical measurements were made in regard to the behavior of synthetic detergents with proteins. According to Neurath & Putnam (*J. Biol. Chem.* 160, 397), viscosity and diffusion measurements suggested that denaturation of the proteins occurred as the result of binding of the detergent anions by cationic protein groups with resultant rupture of the protein structure. Pankhurst & Smith's (*Trans. Faraday Soc.* 41, 630) work along these lines dealt with the changes in the isoelectric points of protein-detergent systems and the effects of electrolytes. They believed that the union of cationic detergent and protein was of an electrostatic nature. Bull (*J. Am. Chem. Soc.* 68, 747) calculated the number of moles of sodium dodecyl sulfate bound per  $\beta$ -lactoglobulin mole and plotted these results as a function of the concentration of detergent.

ANALYSES AND COMPARISONS OF DETERGENTS. A new procedure for analysis of pure soap, by Palit (*Oil & Soap* 23, 58) depended on hydrochloric acid titration with the use of double indicators. The solvent used was a mixture of equal volumes of ethylene or propylene glycol and isopropyl alcohol. A method for determining neutral fats in pure soap depended on the acid and saponification values determined in one operation by measuring alkali requirements before and after saponification (Brignoni—*Anales asoc. quim. farm. Uruguay* 47, 148). A method for borax in detergents was based on the fact that soluble silicates, carbonates, and orthophosphates could be precipitated quantitatively by strontium chloride, whereas strontium metaborate was soluble in excess strontium chloride (Blank & Troy—*Oil & Soap* 23, 50). Tergin, a cellulose soap filler, could be determined in the presence of clay and earth fillers by dissolving the cellulose contained in the Tergin in Schweitzer solution from which it could be precipitated with an acid as hydrocellulose (Di Stefano & Davidova—*Ann. chim. applicata* 35, 94). Phenols can be determined in soaps by precipitating the soap with calcium nitrate and determining the phenols in the filtrate by bromine absorption (Cox—*Analyst* 71, 301) or the phenols may be steam distilled and then determined by bromine absorption.

Burton & Robertshaw (*J. Intern. Soc. Leather Trades' Chem.* 30, 279) reviewed and discussed the common methods for determination of unsaponifiable matter (petroleum, sterols, waxes, etc.) in sulfated detergents. In another communication, Burton & Byrne (*Ibid.* 306) listed and suggested tests for 14 types of compounds that may be found in the sulfated oils used in the leather industry. Brooks *et al.*'s (*Ind. Eng. Chem., Anal. Ed.* 18, 544) procedure for analysis of oil-soluble petroleum sulfonates was based on extraction of the sulfonic acids, carboxylic acids, and

mineral oil with chloroform and absorption of the sulfonates and carboxylates on Attapulugus clay. A direct volumetric determination of the organic sulfonates comprised reaction with *p*-toluidine hydrochloride, extraction of the amine-sulfonate salt with chloroform, and titration with alkali (Marron & Schifferli—*Ibid.* 49). Organically combined sulfuric anhydride could be determined by removing inorganic sulfates, decomposing with nitric and perchloric acids, precipitating as barium sulfate, and calculating as sulfuric anhydride (Du Bose & Holland—*Am. Dye-stuff Repr.* 34, 321). A procedure for commercial sodium Mersolate, an alkyl sulfochloride mixture, depended on determination of moisture and various inorganic salts and reporting the remainder as Mersolate (Hintermaier & Kelber—*Fette u. Seifen* 50, 413). Detergent mixtures of mono- and di-alkyl phosphates were analyzed by determining total phosphate, free phosphate, and free fatty alcohol and calculating the composition (McFarlane—*Oil & Soap* 23, 337). Hintermaier (*Fette u. Seifen* 51, 10) described general qualitative tests for identifying various compounds and salts in detergent mixtures. The Soap Analysis Committee of the American Oil Chemists' Society suggested slight modifications for determining anhydrous soap, fatty matter, and starch in their method for analyzing synthetic detergent-soap mixtures (Sheely *et al.*—*Oil & Soap* 23, 80).

In a lecture on comparison of potash soap versus synthetic detergents for toilet use, Kranich (*Soap Sanit. Chemicals* 22, No. 5, 43) objected to the latter because of the higher cost and the defatting action on skin and hair.

The comparative tests on evaluating detergents made use of various criteria. Winsor (*Nature* 157, 660) evaluated the surface interactions and found that sodium alkyl sulfates were readily displaced by soap. The sulfates with the sulfate radical furthest removed from the end of the hydrocarbon chain were most easily displaced. This ease of displacement agreed closely with the detergency value of the products. Krishnappa *et al.* (*Proc. Indian Acad. Sci.* 23A, 1) preferred surface tension measurements to rate detergents and developed a procedure for such comparisons. Harris (*ASTM Bull.* No. 140, 76, No. 141, 49) described the procedures for comparing detergents by the following tests: acid, alkali, and metallic ion stabilities; lime soap dispersions; Draves-Clarkson wetting test; lather value; organic solvent solubility; and washing test. Sixteen commercial products were compared with these tests. Three communications described standardized practical washing tests in which artificially soiled white fabrics were washed and compared for degree of whiteness (Hurwitz—*Am. Dye-stuff Repr.* 35, 83; Bacon—*Ibid.* 34, 556; Kornreich—*Textile Mfr.* 72, 271). Lipets (*Rabota Provedena v Kolloido-electrokhim. Inst. A.N., S.S.S.R. Zavodskaya Lab.* 11, 426) suggested that detergents could be rated according to their wetting capacity for insoluble powders. Whitehead (*Ciba Rev.* 1945, No. 49, 1789), in a review on evaluation of wetting agents, pointed out the limitations of various tests, *i.e.*, the presence of calcium salts affected various products differently; foaming power was not always proportional to scouring properties; etc. He therefore suggested that the laboratory tests should be empirical and should be based on practical conditions. In connection with such suggestions, practical detergency and wetting tests

were developed for products intended for use in the dairy industry (Jensen—*J. Dairy Sci.* 29, 453), for dishwashing (Mann & Ruchhoff—*Pub. Health Repts.* 61, 877), for agricultural sprays (Felber—*J. Agr. Research* 71, 231), for fire-fighting foams (Henschel—*Firemen* 13, No. 3, 7), for wetting of mine dust (Harmon—*U. S. Bur. Mines Circ.* 7351, 6 pp.), and for metal cleaners (Spring *et al.*—*Ind. Eng. Chem., Anal. Ed.* 18, 201).

There was much effort toward developing a test for the germicidal activity of nonsoap detergents because data on these obtained by the standard procedure, phenol coefficient, were not reasonably accurate. The communications of Pressman (*Soap Sanit. Chemicals* 22, No. 4, 137), Bernstein—*Ibid.* No. 9, 131), and Klarmann & Wright (*Ibid.* No. 1, 125; No. 8, 139) described improved bacteriological procedures for the purpose. The new methods gave lower performance value for quaternary ammonium compounds than the standard phenol coefficient method. These authors and DuBois & Dibblee (*Science* 103, 734) recorded the germicidal value of quaternary ammonium compounds on several species of bacteria.

Valko & DuBois (*J. Bact.* 50, 581), Shelton *et al.* (*J. Am. Chem. Soc.* 68, 357, 753, 755), Bosshard (*Helv. Chim. Acta* 27, 1736), and Vogt (*Arch. Pharm.* 282, 27) synthesized many organic quaternary ammonium salts in studies on the effect of structure and chain lengths on the germicidal properties. In general, all the preparations were bactericidal, and the potency was somewhat related to the structure. The number of compounds tested was large, and the interpretations were of such nature as does not permit condensing; hence the readers are referred to the originals for information.

The factors other than structure which may influence the bactericidal potencies of wetting agents were studied. The bactericidal efficiency of Aerosol OT was unaffected by changes in surface tension (Gershenfeld & Shulik—*Am. J. Pharm.* 118, 264). In these tests the surface tensions were adjusted with acetone. Dry bacteria coated with a greasy film were resistant to cationic detergents (Rahn—*Proc. Soc. Exptl. Biol. Med.* 62, 2). Guiteras & Shapiro (*J. Bact.* 52, 635) reported that high alkalinity renders cation-active detergents ineffective germicidally; while Flett (*Am. Perfumer Essent. Oil Rev.* 48, No. 12, 63) found Nacconol NR effective in the pH range from 2.0 to 11.9. Cetyl pyridinium chloride was also found effective under both acid and alkaline conditions (Quisno & Foter—*J. Bact.* 52, 111), and effectiveness *in vivo* correlated well with *in vitro* tests (Kenner *et al.*—*Ibid.* 449). In *in vitro* tests agar partially neutralized the germicide (Quisno—*Ibid.* 51, 602). Pollard (*Science* 103, 758) pointed out that the bactericidal action of Tergitol-7 was selective, and this property could be useful in bacteriological research.

Mueller *et al.* (*J. Dairy Sci.* 29, 751), in discussing the bactericidal properties of detergents, pointed out that only the quaternary ammonium and phosphonium compounds had sufficient germicidal properties and that the latter compounds should be more desirable because they were also non-corrosive. In general articles on the usefulness of cationic germicides, the subjects treated were: the preservation of milk (DuBois & Dibblee—*J. Milk Tech.* 9, 260; Johns & Pritchard—*Can. J. Pub. Health* 37, 500), uses in the food industry (Hucker—*Farm Research* 12, No. 1, 1), and de-

struction of algae (Corley—*U. S.* 2,393,293). Emulsept, a cation-active germicide recommended for use in the food industry, was reported nontoxic after vigorous tests (Vivino & Koppanyi—*J. Am. Pharm. Assoc.* 35, 169).

A miscellaneous group of reports on properties of detergents remained after the above information was classified. One report rated the hemolytic activity of various alkyl sulfates (Ponder—*J. Gen. Physiol.* 30, 15). The amines of the fatty acids of tubercle bacilli were growth inhibitors of the same bacilli (Buu-Hoi—*Nature* 156, 392). In tests on the action of binary soap mixtures on the skin; sodium laurate-sodium caprylate proved highly irritating as did binary mixtures containing sodium ricinoleate (McKinney & Edwards—*Oil & Soap* 23, 198).

A new method for the determination of glycerol was based on spectrophotometrically evaluating the blue color of the sodium cupric-glycerol complex (Whyte—*Oil & Soap* 23, 323). A mixture of glycerol, ethylene glycol, and propylene glycol could be analyzed by oxidation with sodium iodate, and calculating from the amounts of formic acid, acetaldehyde, and formaldehyde formed (Desnuelle & Naudet—*Inds. corps gras* 1, 113). With controlled oxidation glycerol yielded one mole each of formaldehyde and formic acid; ethylene glycol yielded one mole of formaldehyde; and propylene glycol yielded one mole each of acetaldehyde and formaldehyde. The determination of trimethylene glycol, polyglycerols, and glycerol mixtures by the same investigators (*Ibid.* 2, 16) was based on the same principles. Pohle & Mehlenbacher (*Oil & Soap* 23, 48) reported that the average of their 17 glycerol analyses by acetylation on 95% glycerol was 94.97%, and the standard deviation was  $\pm 3\%$ . In a microdetermination, the glycerol was extracted by passing superheated vapors of ethanol over the dried samples and then determined in the distillate by the dichromate procedure (Raveux—*Ann. chim. anal.* 25, 70).

Many communications on soaps and other detergents provided information on common practices in the industry, described some of the raw materials and their advantages, or described certain products. These are most conveniently entered in this Review with slight classification of the subject treated.

Raw materials: fatty acid selection, McCutcheon—*Soap Sanit. Chemicals* 22, No. 12, 43; rosins, Lombard—*Corps gras savons* 2, 132; *Inds. corps gras* 1, 72; Borglin—*Soap Sanit. Chemicals* 22, No. 6, 43; rosin soap in synthetic rubber manufacture, Cuthbertson *et al.*—*Ind. Eng. Chem.* 38, 975; caustic potash, Ferris—*Soap Sanit. Chemicals* 22, No. 3, 41; sodium silicate filler, Rozhdestvenskii—*Pishcheraya Prom.* 1945, No. 1, 27; phosphates in liquid soap, Bachrach—*Soap Sanit. Chemicals* 22, No. 8, 49; builders, Raymore—*Ibid.* No. 10, 48; lecithin, Lesser—*Ibid.* No. 5, 37; peroxy compounds, Richmond—*Soap, Perfumery Cosmetics* 18, 642; thiosulfate, Hetzer—*Seifensieder-Ztg.* 1944, 65; perfumes, Borderas—*Ion* 5, 648.

Technics: toilet soap, McCutcheon—*Soap Sanit. Chemicals* 22, No. 4, 41; soap making in Brazil, Anon.—*Ibid.* No. 10, 46; soap making in the Pacific, Wessock—*Ibid.* No. 3, 38; speeding up the process, Downie—*Mfg. Chemist* 15, 64;

soap washing, Govan—*Oil & Soap* 23, 229; spray process for soaps, Lee—*Soap Sanit. Chemicals* 22, No. 1, 29; drying, Glücklich—*Seifensieder-Ztg.* 1944, 65; water treatment in the soap industry, Anon.—*Ibid.* 66; packaging liquid soap, Peterson—*Soap Sanit. Chemicals* 22, No. 4, 45.

Products: Marseille soap, Rey—*Inds. corps gras* 1, 45; fat poor or fat free shaving agents, Giloy—*Fette u. Seifen* 50, 327; shampoos, Beach—*Soap Sanit. Chemicals* 22, No. 8, 46; Thomssen—*Ibid.* No. 3, 37; No. 4, 47; control of cosmetics, deNavarre—*Soap, Perfumery Cosmetics* 19, 56; aluminum cleaners, Lesser—*Soap Sanit. Chemicals* 22, No. 9, 44; dry cleaning soaps, Fabry—*Ibid.* No. 7, 48; fat free washing agents, Hanegraaf & Eijzinga—*Chem. Weekblad* 41, 15.

Miscellaneous papers on soap: current soap problems, Thomssen—*Soap, Perfumery Cosmetics* 18, 634; notes on current literature, Wigner—*Ibid.* 19, 40; report on visits to factories in U. S., Latour—*Inds. corps gras* 2, 208; army soap and detergent problems, Simpson—*Oil & Soap* 23, 192; phases of soap, Lachampt—*Inds. corps gras* 1, 100; laundry industry of the U. S., Ripert & Sisley—*Ibid.* 2, 72; *Soap in Industry*, Leffingwell & Lesser—a book published by Chemical Publishing Co., 1946.

Comprehensive reviews on nonsoap detergents giving information on economics, properties, products, uses, etc.: Flett—*Colloid Chemistry* 6, 243; Shedlovsky—*Ann. New York Acad. Sci.* 46, 427; Ralston—*Ibid.* 351; Fischer & Gans—*Ibid.* 406; Price—*Ibid.* 349; Ripert & Sisley—*Inds. corps gras* 1, 171; Ripert—*Corps gras savons* 1, 39, 143; Vallance—*Soap, Perfumery Cosmetics* 19, 827; Wigner—*Ibid.* 40; Carroll—*Mfg. Chemist* 17, 192, 239; *Pharm. J.* 155, 272.

Technic of sulfation of oils. Kroch & Tomlinson—*J. Intern. Soc. Leather Trades' Chemists* 30, 316.

Products: sulfated fatty oils, Glicker—*Petroleum* (London) 8, 32, 130, 232; classification of products, McCutcheon—*Soap Sanit. Chemicals* 22, No. 9, 37; sulfonated fatty amides, Vosganiantz—*Inds. corps gras* 2, 249; alkyl aryl sulfonates, Flett—*Soap Sanit. Chemicals* 22, No. 12, 46; nonionic detergents, Ripert—*Soap, Perfumery Cosmetics* 19, 552.

Uses: in industry, Ackley—*Ann. New York Acad. Sci.* 46, 511; in textile operations, Mosher—*Am. Dyestuff Repr.* 35, 168; in the tannery, Smith—*Soap Sanit. Chemicals* 22, No. 5, 46; No. 11, 43; Alaima—*Rev. Columbiana quim.* 2, No. 2, 23; for sanitation, Snell—*Food Packer* 26, No. 13, 46; in biology and medicine, Valko—*Ann. New York Acad. Sci.* 46, 451; Ripert & Sisley—*Soap, Perfumery Cosmetics* 19, 834; in canneries, Burkard—*Canner* 102, No. 9, 20; for flotation of ores, Rogers *et al.*—*Am. Inst. Mining Met. Engrs., Mining Technol.* 10, No. 4, *Tech. Pub. No.* 2022, 30 pp.; in the baking industry—Lehn & Vignolo—*Bakers Digest* 20, 80; in histological fixatives, Chermock & Muller—*Science* 103, 731; in bubble solutions, Stetson—*Soap Sanit. Chemicals* 22, No. 1, 36.

Comparing detergents: comparison of synthetic detergents with soap, Anon.—*Chem. Industries* 58, 946; detergent power of various prod-

ucts, Sisley—*Corps gras savons* 1, 66; Götte—*Die Chemie* 57, 67; Kling—*Tek. Tik.* 74, 565; chemical analysis of quaternary ammoniums, DuBois—*Soap Sanit. Chemicals* 22, No. 11, 125.

The newly patented nonsoap organic detergents are derivatives of fats or by-products of the coal and petroleum industries. These and their methods of manufacture are not discussed in detail in this Review but are listed with only partial classification.

Those references to the patents on hydrocarbons containing sulfur, such as sulfonates and sulfates are: Allied Chemical & Dye Corp.—*U. S.* 2,364,767, 2,387,572, 2,393,526, 2,394,851, 2,397,133, 2,401,726.

American Cyanamid Co.—*U. S.* 2,385,314.

Atlantic Refining Co.—*U. S.* 2,406,763.

Chem. Fabrik Grünau A.-G.—*Ger.* 748,564 Cl. 30h.

Colgate-Palmolive-Peet Co.—*Can.* 434,340; *U. S.* 2,394,320-1.

E. I. duPont de Nemours & Co.—*U. S.* 2,398,426, 2,402,587.

Fife—*Brit.* 561,624.

Gordon & Likhman—*U.S.S.R.* 65,102.

Harvel Corp.—*U. S.* 2,412,264.

Industrial Patents Corp.—*U. S.* 2,394,834.

Jäger & Jäger—*Ger.* 745,637 Cl. 30h.

Kalle & Co. A.-G.—*Ger.* 742,194 Cl. 8i.

Monsanto Chemical Co.—*U. S.* 2,402,823 2,403,038.

National Oil Products Co.—*Brit.* 559,265; *Can.* 432,690; *U. S.* 2,392,945, 2,397,692.

Procter & Gamble Co.—*U. S.* 2,396,278.

Society of Chemical Industry in Basle—*U. S.* 2,399,434.

Solvay Process Co.—*U. S.* 2,381,658, 2,404,289.

L. Sonneborn Sons, Inc.—*U. S.* 2,396,673, 2,409,671.

Union Oil Co. of California—*U. S.* 2,411,819.

Victor Wolf Ltd.—*Brit.* 562,321, 563,482.

In some cases sulfur-containing detergents were prepared by sulfonating nitrogen derivatives, such as amines, amides, etc.:

Aktieselskabet Grindstedvaerket—*Dan.* 63,458.

Allied Chemical & Dye Corp.—*U. S.* 2,388,962.

American Cyanamid Co.—*U. S.* 2,390,476, 2,391,830.

Courtaulds Ltd.—*Brit.* 565,675.

E. I. duPont de Nemours & Co.—*U. S.* 2,390,253.

Everett *et al.*—*Brit.* 564,359.

J. R. Geigy Co.—*Brit.* 560,628.

Huppert—*U. S.* 2,384,837.

I. G. Farbenind. A.-G.—*Ger.* 745,555 Cl. 8i, 745,909 Cl. 8i.

Petrolite Corp.—*U. S.* 2,386,445.

Society of Chemical Industry in Basle—*U. S.* 2,398,990.

Soc. pour l'ind. chim. a Bale—*U. S.* 2,376,911.

Some special amines, amides, and quaternary ammonium compounds were patented as detergents:

Alien Property Custodian—*U. S.* 2,394,306-7.

Alrose Chemical Co.—*U. S.* 2,404,297-8.

Arnold, Hoffman & Co., Inc.—*U. S.* 2,410,788-9.

Commercial Solvents Corp.—*U. S.* 2,389,875, 2,402,791.

E. I. duPont de Nemours & Co.—*U. S.* 2,395,265, 2,395,971.

Emulsol Corp.—*U. S.* 2,388,154, 2,411,434.

General Aniline & Film Corp.—*U. S.* 2,382,185.

Harris—*U. S.* 2,409,275.

Heberlein Patent Corp.—*U. S.* 2,370,405.

Henkel & Cie. G.m.b.H.—*Ger.* 741,305 Cl. 12o.

Hunsdiecker & Vogt—*Ger.* 745,221 Cl. 8o.

Imperial Chemical Industries Ltd.—*U. S.* 2,386,142.

Industrial Patents Corp.—*U. S.* 2,412,113.

Montclair Research Corp.—*U. S.* 2,398,317.

Onyx Oil & Chemical Co.—*U. S.* 2,407,703.

Orelup—*U. S.* 2,388,281.

Parke, Davis & Co.—*U. S.* 2,406,902

Pennsylvania Salt Manufacturing Co.—*U. S.* 2,374,113.

Petrolite Corp.—*U. S.* 2,394,432.

Pure Oil Co.—*U. S.* 2,394,678.

Richards Chemical Works—*U. S.* 2,374,354.

Röhm & Haas Co.—*U. S.* 2,395,336, 2,395,989.

Shell Development Co.—*U. S.* 2,376,105, 2,402,495.

Victor Chemical Works—*U. S.* 2,406,423.

A number of new detergents or their intermediates could not be classified above. This miscellaneous group includes special esters, ethers, and organic phosphates:

E. I. duPont de Nemours & Co.—*U. S.* 2,382,874, 2,396,786, 2,397,602, 2,402,137.

Eastman Kodak Co.—*U. S.* 2,386,250.

Monsanto Chemical Co.—*U. S.* 2,380,699, 2,385,713.

Onyx Oil & Chemical Co.—*U. S.* 2,405,784.

Petrolite Corp.—*U. S.* 2,386,446.

Resinous Products & Chemical Co.—*U. S.* 2,385,790.

Virginia Smelting Co., Inc.—*U. S.* 2,404,913.

## Abstracts

### Oils and Fats

Edited by

M. M. PISKUR and SARAH HICKS

EDIBLE OIL INDUSTRY IN GERMANY. W. H. Goss (Northern Reg. Res. Lab., Peoria, Ill.). *Food Industries*, 19, 320-3 (1947). German oil refining is batchwise and not continuous like ours. Soybean oil reversion, a common problem, is combatted by the removal of lecithin or treatment with live steam. Fatty Et esters were added to margarine.

RENDERING TECHNOLOGY. M. Maillet (Etablissements Olida). *Inds. corps gras* 3, 38-46 (1947). This

review contains legal definitions of the products, and descriptions of raw material, 5 rendering processes, cooling, and packaging. The water bath, kettle, dry autoclave, vapor autoclave, and Danish Titan methods of rendering and equipment were illustrated and described.

CHARACTERISTICS OF CANADIAN LARD. H. J. Lips and G. A. Grant (Natl. Res. Labs. Ottawa) *Can. J. Research* 25F, 63-65 (1947). Mean values for