

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

Report of the Literature Review Committee*

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

Physiology and Biochemistry

REVIEWS. The reviews concerning the subject matter of this division of the fat literature had the following texts: Chemistry of lipides (Deuel—*Ann. Rev. Biochem.* 19, 89), lipide metabolism (Deuel—*Calif. Med.* 72, 197), non-caloric functions of fat in the diet (Deuel—*J. Am. Diet. Assoc.* 26, 255), biogenesis of fatty acids or natural fats (Täufel—*Fette u. Seifen* 52, 398), formation of milk fat (Täufel—*Ibid.* 9), end metabolism of fatty acids (Martius—*Ibid.* 206), fat resorption and liver fattening (Lang—*Ibid.* 595), causes of variations in the composition of fish oils (Lovern—*J. Soc. Leather Trade's Chemists'* 34, 7), oxidation of fatty acids in the animal organism (Breusch—*Angew. Chem.* 62, 66), fat soluble vitamins (Moore—*Ann. Rev. Biochem.* 19, 319), insulin and fat metabolism (Breusch—*Fette u. Seifen* 52, 273), and lipoxidases (Franke—*Ibid.* 11).

FAT NUTRITION. Some reports of investigations contained information useful in design of experiments for the nutritional evaluation of fats. Barki *et al.* (*J. Nutr.* 40, 383) recorded that different fats produced their optimum growth at different levels of intake. In this work rats gained most weight on 35% butterfat and on 10% corn oil when equi-caloric diets with 10-35% fat were compared. A comparative evaluation of their nutritive merits based on only one dietary level would therefore be misleading. In tests on the effect of the diet on fat retention by Schulz & Thomas (*Ibid.* 42, 175) the retention of fat on a sucrose diet containing 10% fat was unaffected by *L*-cystine, *DL*-methionine, ascorbic acid, bile salts, sodium fluoride, carotene, and other substances, but retention was decreased by inclusion of 20% charred alfalfa or 10% agar-agar and by substitution of lactose for the sucrose in the diet. Meyer-Brunot *et al.* (*Deut. Arch. klin. Med.* 194, 707) and Wicke (*Ibid.* 196, 445) observed that some of the oil ingested within soybeans was not retained because undigested soybean cells containing oil pass into the stool. Rapid nutritional evaluation of lipides was accomplished by Kaufmann and Schmidt (*Fette u. Seifen* 52, 528) with the use of the glow worm, *Lycastes ranauenses*, as the test species. Growth was measured by the increase in the number of segments.

In work on comparative nutritive value of fats, Deuel *et al.* (*J. Nutr.* 42, 239) have now completed raising rats through 25 generations on rations containing butter fat and rations containing margarine fats. No significant difference in growth, reproduction, or lactation was observed between the two groups. The calcium balance was somewhat more favorably positive for the rats on the margarine fat-containing diet than those on the diet containing butterfat. When De & Karkun (*Indian J. Dairy Sci.* 2, 114) compared butterfat and coconut oil for growth promotion and calcium utilizations, the butterfat was superior especially with regard to calcium utilization. However, Sadasivan (*Current Sci. India* 19, 28) in checking some such reports obtained similar protein,

calcium, and phosphate assimilation with either butterfat or coconut oil as the dietary fat. It might be possible that the age of the test animals might account for the different results, for Kane *et al.* (*J. Gerontol.* 4, 185) recorded that excretion of calcium was proportional to the fat content of the diet only in old animals, and that harder fats had a more pronounced effect.

Other reports on nutritive value of butter concerned the possibility of the presence of a growth factor. Bhalerao & Basu (*Indian J. Dairy Sci.* 2, 154) obtained better growth with the liquid portions of fractionated cow butterfat than with the solid fractions and suggested that a growth promoting factor may be responsible. Jansen & Groot (*12th Internat'l Dairy Congr.* 1949, 191) were unable to obtain special growth acceleration with vaccenic acid and suggested that any growth promotion advantage that butterfat may have might be due to some constituent in the unsaponifiable part.

Kaunitz & Slanetz (*J. Nutr.* 42, 375) concentrated a fraction from lard by molecular distillation which protected animals against signs of vitamin A deficiency. Two percent of distillate provides better pro-

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*LITERATURE REVIEW COMMITTEE

E. W. BLANK E. A. GASTROCK
J. B. BROWN M. M. PISKUR,

Chairman

tection in rats than injections of 7.5 units of vitamin A palmitate. This action could be explained neither by the presence of vitamin A or by an antioxidant in the distillate. This distillate or massive doses of synthetic vitamin A palmitate inhibited the fatal disease in rats caused by vitamin A free diet containing rancid lard (Kaunitz & Slanetz—*Proc. Soc. Exptl. Biol. & Med.* 75, 322). In another investigation on nutrition of spoiled fats it was found that the liver, many tissues, and serum deactivate peroxides of fats (Dubouloz—*Biochem. et Biophys. Acta* 3, 371). Polymerized oils were found to be practically unabsorbed (Lassen *et al.*—*Arch. Biochem.* 23, 1).

In one series of nutrition tests on fat acids containing a methyl side chain, members methylated at the first to fifteenth carbon were investigated (Weitzel *et al.*—*Naturwissenschaften* 37, 68). There were some indications that less digestibility occurred when the methylation was at the eighth or ninth carbon, and dogs that digested 25 g. of methyl stearic acid could not digest 19 g. of α -methylcapric acid.

Some nutrition work contained information on the interrelations of vitamins and fats. Menschik *et al.* (*Ann. N. Y. Acad. Sci.* 52, 94) reported that vitamin E deficient mice lacked subcutaneous, subperitoneal adipose tissue, and the amount of brown adipose tissue was increased. This was interpreted to indicate that vitamin E deficiency affects enzymic reactions during lipide metabolism. Oxidized lard was much more toxic to vitamin E deficient rats than fresh lard (Luttrell & Mason—*Ibid.* 113). Rats fed suboptimal levels of riboflavin excreted less of the riboflavin with 20% corn oil in the ration than when on a fat-free ration (Pearson & Reiser—*Intern. Z. Vitaminforsch* 21, 44). The increases in body weights of rats varied with the fat content of certain diets and were not affected by thiamine intake where the intake was two micrograms or more per day (Krider & Guerrant—*J. Nutr.* 41, 115). Deposition or retention of carcass fat in this work was suppressed by extreme combinations of low-fat and low-thiamine or high-fat and high-thiamine dietaries. Tests on supplementing adequate poultry rations with wheat germ oil by Branion *et al.* (*Sci. Agr.* 27, 545) revealed no beneficial effects of such practice on growth, mortality of young chicks, hatchability of eggs, fertility, or egg production.

The desirability of fat in the diet was evident in various tests. Rats that had been maintained on a fat-poor diet showed extreme hunger for fat (Laubmann—*Österr. Zool. Z.* 2, 2). Supplying assimilatable fat by injection along the spinal column did not reduce their appetite for fat, but when fat was introduced by a gastric fistula, they ate less fat. These mice seemed to select fat according to the fatty acid content rather than by color, taste, or smell. Loss of weight and toxic symptoms in thyroid fed rats were inhibited by feeding high-fat diets (Greenberg & Deuel—*J. Nutr.* 42, 279). Lack of fat in poultry rations decreased fertility and hatchability of the eggs produced (Reiser—*Ibid.* 40, 429). The most highly unsaturated fatty acids tended to disappear from the fat of eggs of hens on fat-free rations. Chicks hatched from such eggs failed to grow at a normal rate (*Ibid.* 42, 319). This was attributed to deficiency of the essential polyunsaturated acids. Decker *et al.* (*Ibid.* 41, 507) reported that mature mice may suffer a chronic essential fatty acid deficiency without showing symptoms in external ap-

pearance. With stresses such as injuries, pregnancy, X-irradiation, etc., symptoms may develop. The growth response obtained on feeding fat-deficient rats linoleic acid was accelerated by injection of a growth hormone whereas without the fatty acid there was no growth response (Deuel *et al.*—*Ibid.* 40, 351). Administration of 10% cottonseed oil to rats receiving linoleate at optimum level resulted in further acceleration of growth. This suggested that oil possessed some effect other than that of linoleic acid which improved growth. The depression in growth of chicks caused by 20% alfalfa meal in the diet was prevented by additions of cholesterol and cottonseed oil in the diet (Peterson—*Ibid.* 42, 597). When the digestible lipides of dairy cattle rations fall below 350 g. per day, production of butterfat may decrease (Leroy—*12th Internat'l Dairy Congr.* 1, 21).

Clinical experience on intravenous infusion of fat emulsions containing 15 and 20% fat into 25 human subjects were recorded by Shafroff (*Proc. Soc. Exptl. Biol. & Med.* 72, 543). Incidences of constitutional reactions amounted to 27%.

Excessively high-fat diets were studied under various conditions. A 4,000 calorie diet containing 295 g. of fat was well tolerated at 0°F. but not at 32°F. (Butson—*Lancet* 258, 993). Introduction of much fat in the diet of dogs at high altitudes increased the acetone bodies in the blood slightly, but no serious disturbance of fat metabolism took place (Ostrogorskaya—*Fiziol. Zhur.* 35, 716). Accelerated skeletal growth and development in male mice and production of articular changes comparable to those occurring in old mice or in degenerative joint disease in man were observed with high fat diets (Silberberg & Silberberg—*Am. J. Path.* 26, 113). Incipient cataracts were observed in rats fed a diet containing 44% fat for 29-84 days (Bertolani—*Boll. soc. ital. biol. sper.* 24, 1152). Ingestion of large amounts of oil or 40% cream reduced the blood coagulation time of human patients (Duncan & Waldron—*Trans. Assoc. Am. Physicians* 62, 179). With 60 cc. of cream the average decrease of clotting time was 280 seconds.

FAT ABSORPTION AND TRANSPORT. The investigations on fat absorption were concerned mostly with the mechanism of the process. The "partition hypothesis" as postulated by Frazer whereby presence of emulsifier, bile acids, emulsification to fine particles, and absorption of hydrolyzed and neutral fat through different pathways are important considerations, received very little support; for most workers recorded results incompatible with the hypothesis. Favarger (*Helv. Physiol. Pharmacol. Acta* 7, 371) suggested that phospholipides have an important role in the absorption of fats; for analyses of intestinal superficial, deep (villi), and mucosa layers after feeding trielaidin showed that more newly formed phospholipides appeared in the epithelial than in the deeper layers. Johnson *et al.* (*Am. J. Dis. Children* 80, 545) in tests on improving fat retention in 13 premature infants and two full term infants with steatorrhea by administration of a commercial emulsifier, "Tween 20," had slight success with only seven of the premature infants. Becker *et al.* (*Gastroenterology* 14, 80), on the other hand, found that "Tween 80" or lipase improved the rate of absorption of fats. They explained this action of the emulsifier on the basis that fat may be absorbed in the unhydrolyzed state, i.e., according to the partition

hypothesis. Berry & Ivy (*Am. J. Physiol.* 162, 80) investigated the partition hypothesis by administering finely emulsified mineral oil to dogs and observing the lymph in the lacteals and thoracic duct for increase in unsaponifiable matter. The results did not support the hypothesis, on the basis that the mineral oil was not absorbed. Tests by Shoshkes *et al.* (*Proc. Soc. Exptl. Biol. & Med.* 75, 680) failed to demonstrate that absorption of fats or oils by the rat could be improved by the presence of emulsifiers such as soybean phosphatides or "Tween 80" even when used in great excess over that needed to make excellent emulsions. In the same work choline also failed to improve absorption of fat. Tidwell (*J. Biol. Chem.* 182, 405) in measuring absorption of fats by systemic lipemia found no improvement of absorption with emulsifying agents; except that material containing choline, i.e., phospholipides, did increase absorption. He also compared absorption of neutral fat and free fatty acids. His data was interpreted to disagree with the partition hypothesis. The fact that choline material increased absorption while other emulsifying agents did not was said to indicate that the choline was involved in a manner other than emulsification. Reiser (*Proc. Soc. Exptl. Biol. & Med.* 74, 666) demonstrated that glycerides were absorbed more rapidly than free fatty acid, but believed that the pathways of absorption were the same. Volk & Popper's (*Gastroenterology* 14, 549) lipide absorption work indicated that fat or vitamin A was absorbed better from aqueous emulsions than as oils, and in excessive doses the lipides were stored in the *lamina propria* of the villi. His tests indicated that choline did not release lipides from the intestines nor increase the absorption of vitamin A.

Bollman *et al.* (*Am. J. Physiol.* 163, 41) studied fat absorption by analyzing the lymph after feeding fat. The lipides in the lymph of the small intestines or thoracic duct increased enormously after fat meals, principally in neutral fat. An increase in phospholipides of the intestinal and thoracic lymph suggested that the small intestine was the source of phospholipides for the plasma during absorption of fat.

Diaz *et al.* (*Bull. Inst. Med. Research Univ. Madrid* 2, 219) reported that fat absorption in dogs during obstruction of the bile duct was normal. The steatorrhea which occurred under this condition was attributed to secretion of fat through the intestinal wall as a result of augmented lipema. Work on action of bile acid in lipide absorption by Schettler (*Z. ges. inn. Med.* 4, 718) indicated that certain bile acids raised the esterified cholesterol levels in liver, spleen, and blood.

Absorption and transport of fat was also studied by means of fats labeled with C^{14} . Coniglio *et al.* (*Proc. Soc. Exptl. Biol. & Med.* 75, 666) fed rats C^{14} fatty acids at intervals of 20 hrs., and dispatched the animals 48 hrs. after the last dose. Absorption was 73-90% and less than one percent of the C^{14} was expired as carbon dioxide. The greatest C^{14} activity was found in the liver and mesenteric fat. In similar work, Bloom *et al.* (*J. Biol. Chem.* 184, 1) recovered practically the same amounts of labelled fat from the thoracic lymph whether the lipide was administered as neutral fat or fatty acids. This finding was not consistent with the partition hypothesis of fat absorption. With the use of labeled phosphorus and carbon in the phosphate and fatty acid portions, respectively,

of the phospholipides, Weinman *et al.* (*Ibid.* 187, 643) demonstrated that the turn-over rate of both portions of the lipide in the blood was the same. This finding rules out the possibility of the phosphorous compounds serving as stable nuclei on which fatty acids were being renewed during transport.

The blood proteins are in some way involved in fat transport for the fat in blood appeared to be in the form of lipoprotein complexes. Much fundamental information on the character of these complexes were recorded in a symposium on the subject. Luck (*Discussions Faraday Soc.* 1949, No. 6, 44) associated the ability of serum albumins for binding fats to the function of positively charged groups and the proximity of these to non-polar side chains. Macheboeuf & Rebeyrotte (*Ibid.* 62) recorded the density, mobility, and solubility of the lipoproteins of horse serum. Gurd *et al.* (*Ibid.* 70) reviewed the concept of α - and β -lipoproteins of human plasma. McFarland (*Ibid.* 74) found that the lipides and serum globulins were unaffected in mobility or concentration by the presence of lecithin, and the finding was interpreted with regard to interaction of lipides and proteins in the plasma. Frazer's (*Ibid.* 81) discussion for the symposium pertained to particle size, effect of pH, and emulsification of the lipoproteins in relation to transport of the lipides. Popjak & McCarthy (*Ibid.* 97) found that serums poor in lipides exerted a lower osmotic pressure than normal serums. Frey-Wyssling (*Ibid.* 130) reviewed the literature on the structural relationship of proteins and lipides in chloroplasts. Booij (*Ibid.* 143) reviewed the theories regarding lipoprotein complexes. Ponder (*Ibid.* 152) considered the structure of lipoproteins as radially oriented lipide and tangentially oriented protein, the components being associated rather than spread out in separate layers.

Swank *et al.* (*Proc. Soc. Exptl. Biol. & Med.* 75, 850) obtained changes in the chromatographic protein patterns of the plasma of dogs by feeding high fat meals or after administration of heparin or "Tween 81." This change was said to result from alterations of the surface active substance present in the blood. Burt's & Rossiter's (*Biochem. J.* 46, 569) records of analyses of the lipides of blood showed that the leucocytes contained 12 times as much fat as did the red cells, the proportion of phospholipides in the two types of cell was about the same, but the red cells contained more cephalin and the polymorphonuclear leucocytes contained more sphingomyelin.

LIVER LIPIDES. The liver functions in the synthesis, transport, and oxidation of fats in some manner which as yet has not been thoroughly elucidated. Present work on how the fat in the liver could be modified should serve as fundamental information on the problem.

Several investigators studied the action or efficiency of various materials for preventing fatty infiltration of the liver in animals made predisposed to development of fatty livers. In comparing autoclaved liver, brain, salivary glands, and pancreas, Dragstedt *et al.* (*Proc. Soc. Exptl. Biol. & Med.* 75, 785) reported that the pancreas product contained an effective substance, lipocaic, which was not present in the other tissues. Bruun *et al.* (*Acta Physiol. Scand.* 20, 319) suggested that the lipotropic effect of lipocaic was different from that of choline or proteolytic enzyme

and was not connected with the inositol that it may contain. He reported that this substance was present in pancreas, liver, and muscle. McCormick's & Drill's (*Proc. Soc. Exptl. Biol. & Med.* 74, 626) tests indicated that the lipotropic effect of liver extracts or vitamin B₁₂ concentrates was not due to the small amounts of choline, inositol, and folic acid present in the materials. Crystalline vitamin B₁₂ alone or with small amounts of choline failed to prevent fatty changes in the livers. Best *et al.* (*J. Biol. Chem.* 186, 317) rated the lipotropic effect of various substances in fat-free diets and diets containing fats. Choline exerted a strong and betaine a moderate effect with both diets. Inositol was slightly active in fat-free diets but showed no activity when the diet contained fat. According to Leites & Yakusheva (*Ark. Patol.* 11, No. 4, 44) fatty liver infiltrations caused by carbon tetrachloride were resistant to pancreatic lipotropic factor.

Past work has associated lipotropic action of some substances such as choline to the presence of labile methyl groups; and it had been demonstrated that ethyl homologues of the compounds were also active. The newer workers on ethyl homologues of choline found them much less active than choline, and also found that they were toxic (McArthur & Lucas—*Biochem. J.* 46, 226; Stekol & Weiss—*J. Biol. Chem.* 185, 585; Holton & Ing—*Brit. J. Pharmacol.* 4, 190).

In investigations on lipotropic effects of vitamins, hormones, and enzymes, folic acid (Kelley *et al.*—*J. Biol. Chem.* 187, 529), estrogen (Shipley *et al.*—*Arch. Biochem.* 25, 309), and trypsin (Montgomery *et al.*—*J. Biol. Chem.* 185, 307) acted as anti-fatty liver substances, while growth hormone increased liver lipides. Zinc oxide administration during high fat dietaries reduced deposition of fat in the liver (Sadasivan—*Current Sci.* 19, 10). Under the same conditions cystine in small amounts (0.5%) increased liver fat, but in amounts of about five percent in the diet no increase of the liver lipides was evident (Levy—*Compt. rend. soc. biol.* 143, 1340). The ability of cysteine to augment the liver lipides was inhibited by presence of generous amounts of niacin or tryptophane (Tyner *et al.*—*J. Biol. Chem.* 187, 651).

High fat diets deficient in lipotropic substances did not increase liver cholesterol (Isichenko—*Ark. Patol.* 11, No. 4, 48). In the same work pancreatic factor only at large levels reduced liver cholesterol. Test by Blewett *et al.* (*Nature* 164, 621) on incorporation of radioactive phosphorous in phospholipides after administration of oleic acid and inositol did not support the view that inositol exerted its lipotropic action by stimulating the turnover of phospholipides. Analyses of the large granules mitochondria, from rat livers by Swanson & Artom (*J. Biol. Chem.* 187, 281) showed that the compositions of the lipides were essentially the same as those of the whole unfractionated liver.

BIOLOGICAL OXIDATION OF LIPIDES. Much of the work on biological oxidation of fats was done *in vitro* with liver tissue. Kennedy's & Lehninger's (*J. Biol. Chem.* 185, 275) work indicated that liver mitochondria oxidized normal fatty acids of chain length greater than 12 carbon atoms and C₇ to C₁₇ odd carbon acids chiefly to carbon dioxide, presumably *via* the Krebs tricarboxylic acid cycle. Under identical experimental conditions the shorter normal fatty acids yielded primarily acetoacetate. Weinhouse *et*

al. (*Ibid.* 191) obtained equal amounts of carbon dioxide and acetoacetic acid from higher fatty acids, in contrast to lower fatty acids (up to C₈) which were oxidized mostly to ketones. Oxidation was with rat liver slices. Geyer *et al.* (*Ibid.* 461) did similar work with labeled fatty acids and discussed the results from the standpoint of the influence of the relative number of (CH₃CO-) and (-CH₂CO-) groups on the symmetry of the acetoacetic acid produced. Odd and even numbered acids behaved similarly, suggesting that odd number acids yielded two-carbon fragments equal to those available from the next lower even number acid. The portion of C¹⁴ in the acetoacetic acid derived from certain specifically labeled fatty acids confirmed other reports that the fragments were not formed by alternate oxidation of even numbered acids. In a study of the oxidation of hexanoic acid and derivatives with liver tissue by Witter (*Ibid.* 537) hexanoate, 2-hexanoate, and sorbate oxidized at similar rates; whereas β-hydroxy and β-keto derivatives were slowly oxidized, and α,β-dihydroxy hexanoate and triacetate were not affected. These results were discussed with regard to the possible intermediates of biological oxidation of fat. A finding by Merlini (*J. Physiol. Paris*, 39, 499) that oxygen uptake on oxidation of C₃ to C₇ fatty acids by heart muscle was three times greater with the methyl esters than with the sodium salts was interpreted to constitute evidence for the existence of ω-oxidation of fats.

The rate of oxidation by tissues was measured for studying factors influencing the oxidation. The acceleration of the oxidation by ascorbic acid had already been reported and confirmed in the past. Kuck (*Arch. Biochem.* 26, 351) in a study of other influences found that neither adenosinetriphosphoric acid nor cytochrome added separately had an effect but when used with ascorbic acid they improved oxidation above the values obtained with ascorbic acid alone. Among the organic acids, citric acid was a very effective oxidation inhibitor followed by oxalic and pyruvic acids; malic and fumaric acids caused slight inhibitions; and oxalacetic, lactic, and malonic acids were without effect. The thiobarbituric test was used in this work to estimate the amount of oxidation taking place. Donnan (*J. Biol. Chem.* 182, 415) showed that data obtained with this test paralleled those by the more conventional methods. In Geyer's (*Ibid.* 101) measurements of oxidation of radioactive trilaurin by the amount of carbon dioxide evolved, the reaction was accelerated by the addition of fumarate, malate, oxalacetate, pyruvate, and α-ketoglutarate, whereas malonate decreased activity. Inhibition was also demonstrated with other substances. In tests on oxidation of fatty acid by Johnson & Lardy (*Ibid.* 184, 235) the ratio of phosphate to oxygen uptake was about three in the presence of fluoride as compared to 1.0-1.5 in its absence. It was suggested that inhibition of the reaction by fluorides was at some step prior to the formation of β-keto acids.

Gammeltoft (*Acta Physiol. Scand.* 19, 270, 280) measured the concentration of ketone bodies in arterial and venous blood of human and animal subjects during starvation. In most animals the results were compatible with Lundsgaard's hypothesis of partial oxidation of fatty acids to ketone bodies in the liver and subsequent oxidation in the peripheral tissue. In the starving dog appreciable ketonemia did not de-

velop and, therefore, the ketones in the blood could not represent the entire fat oxidation.

BIOLOGICAL FAT SYNTHESIS. Several compounds were labeled by incorporation of deuterium or radioactive carbon and were used for studying the biological synthesis of fatty acids. Bloek and coworkers (*J. Biol. Chem.* 179, 839; 183, 33, 431, 441; 185, 131) used acetic acids labeled with deuterium, C¹³, and C¹⁴ to trace the synthesis of cholesterol and fat. The half times for synthesis of saturated and unsaturated fatty acids were, respectively, one and two days in the liver, and 16-17 and 19-20 days in the carcass. The half time data for cholesterol synthesis was six days in liver and 31-32 days in the carcass. The observation that neutral fats were regenerated faster than phospholipides was interpreted to suggest that phospholipides were not necessary intermediates for synthesis of triglycerides. In this work methyl groups of acetic acid were identified in cholesterol as sources of carbon atoms 18, 19, 26, 27, and presumably 17; and carbon 25 and probably 10 were furnished by carboxyl groups of acetic acid. Isovaleric acid was a more efficient source of carbon for cholesterol synthesis than acetic acid.

Popjak and workers (*Biochem. J.* 46, 547; 47, 233; *Arch. Biochem.* 23, 508) used acetates labeled both with deuterium and C¹⁴ to demonstrate that cholesterol and fatty acids were synthesized by fetal livers and extrahepatic tissue. Other work on extrahepatic tissue indicated considerable synthesis of fat in intestines and lungs. The volatile fatty acids of milk were synthesized in the mammae. Brady & Gurin (*J. Biol. Chem.* 186, 461) with similar technique showed that fat and cholesterol could be synthesized in liver slices from acetic, pyruvic, butyric, hexanoic, and octanoic acids. It was suggested that fragmentation of the acids to two carbon units first occurred and these were recombined to form the long chain acids. Sakamis' (*Ibid.* 187, 369) work indicated that acetone could be considered as an intermediate in fat synthesis.

Chaikoff's and coworkers' (*J. Biol. Chem.* 184, 727; 185, 845; *Proc. Soc. Exptl. Biol. & Med.* 73, 348) work on conversion of labeled glucose to fat in various tissues, and on metabolism of labeled palmitic acid in normal and liverless dogs indicated that the main site of fat and phosphatide syntheses was in the liver. Conversion of glucose to fat was 10 times as great in liver as in the other tissues. About 20 times as much injected labeled palmitic acid appeared in the phospholipides of the normal dog as in the liverless dog; accordingly the main site of esterification was said to be in the liver.

In biotin deficient rats, the rate of cholesterol synthesis was normal and fatty acid synthesis was slightly increased (Curran—*Proc. Soc. Exptl. Biol. & Med.* 75, 496). The increase of fatty acid synthesis was attributed to the inanition accompanying biotin deficiency and not to the biotin deficiency itself. Rat-liver dehydrogenase converted margaric, palmitic, and myristic acids to the corresponding monethylenic acids in about 10% yields (Fantl & Lincoln—*Australian J. Exptl. Biol. Med. Sci.* 27, 403). The hen was able to synthesize fatty acids with three, four, and five double bonds from linoleic acid (Reiser & Gibson—*J. Nutr.* 40, 429; Reiser—*Ibid.* 42, 325). In similar work by Widmer and Holman (*Arch. Biochem.* 25, 1) the rat synthesized arachidonic acid

from linoleic, and hexanoic from linolenic. In certain conditions the animals synthesized pentaenoic acid from linolenic acid.

An increase in brown adipose tissue and an even higher rise of ascorbic acid content of rats adapted to cold environments suggested that ascorbic acid was intimately linked with some phase of fat metabolism (Page & Babineau—*Can. J. Res.* 28E, 196).

DEPOSITION AND MOBILIZATION OF FATS. The new reports on deposition of fat in the body pertained to the influences of vitamins and hormones on the process. Tocopherol supplementation in swine feeds markedly affected the fatty acid composition of the body fats by increasing the percentage of oleic acid at the expense of the saturated fatty acids (Bratzler *et al.*—*J. Nutr.* 41, 59). When dairy cattle were given 50-75 g. of cod-liver oil the butterfat content of the milk showed a significant decrease (Ferrando—*Bull. soc. chim. biol.* 31, 810). Insulin increased fat deposition in rats and increased the size of the individual fat cells (Marble & Renold—*Trans. Assoc. Am. Physicians* 62, 219). Treatment of rats with adrenocorticotropin caused an increase in the glyceride content of brown adipose tissue (Baker *et al.*—*Proc. Soc. Exptl. Biol. & Med.* 73, 337). When using 2-methyl-4-thiouracil administrations during the fattening of cattle there was a decrease of 0.536 g. of water in the muscles when the amount of fat was increased by one gram.

Several investigators recorded fundamental observations on behavior of body lipides while animals were under conditions forcing mobilization of fat depots. In starving mice cholesterol and phospholipides in the blood and liver rose during the first two days, then dropped until about the sixth day and remained constant thereafter (Schettler—*Arch. ges. Physiol.* 251, 398). In a clinical report on the same subject, undernourishment of patients increased the total fat of the serum, and the cholesterol and phospholipides of the red-blood cells decreased (Horst—*Klin. Wochschr.* 28, 184). In fasted rats there seemed to be a preferential mobilization of the saturated fats for the iodine value of body fats increased (Clement—*Arch. sci. physiol.* 4, 5, 13). When fasting was superimposed with refrigeration the iodine values of the body fats were further increased. In this work denervating certain body regions reduced the fat mobilized from the affected parts.

Mobilization of fat was brought about by administration of certain pituitary or adrenal gland extracts (Levin & Faber—*Proc. Soc. Exptl. Biol. Med.* 74, 758; Payne—*Endocrinology* 45, 305; Köhler & Schneider—*Klin. Wochschr.* 26, 729). In some of this work concentrates of individual hormones were inactive and the investigators postulated that a separate fat-mobilizing factor may exist in the pituitary. In one investigation cortisone and adrenocorticotropic hormone increased serum cholesterol and phospholipides and decreased the neutral fat but the effects of the latter hormone was less pronounced (Adlersberg—*Proc. Soc. Exptl. Biol. Med.* 74, 877).

CHOLESTEROL METABOLISM. Most of the work on cholesterol metabolism involved determining its normal distribution in the blood and how this may be affected by diet, administration of cholesterol, age, etc. Data by Kronerup (*Arch. Internal Med.* 85, 398) showed that women tend to have higher free cholesterol levels in serum than men, and that elderly

men had much higher serum free cholesterol than young men. Sperry & Webb (*J. Biol. Chem.* 187, 107) reported that serum cholesterol concentrations increased with age in some individuals, but they did not consider that the increase was an obligatory concomitant of aging. In work on correlating serum cholesterol and basal metabolic rates Peeler *et al.*'s (*J. Applied Physiology* 3, 197) data showed that the serum cholesterol was a fairly stable characteristic of the individual. In this work the correlation coefficient between the serum cholesterol and the basal metabolic rate was not statistically significant. Twelve persons 31-54 years old showed no deviation from normal blood cholesterol when cholesterol was rigidly removed from the diet (Kabelitz—*Z. ges. exptl. Med.* 113, 515). When cholesterol was fed to rats there occurred an increase in free fatty acids and sterol-like compounds in the feces (Cook *et al.*—*Biochem. J.* 47, 600). These results were interpreted to indicate that there was a balance between the cholesterol intake and synthesis, and the breakdown in the body. Intravenous administration of hypercholesterolemic rabbit plasma resulted in a rapid increase in total serum cholesterol followed by a gradual decrease (Oppenheim & Bruger—*Proc. Exptl. Biol. Med.* 75, 636). Addition of free cholesterol to the dietary fat of the mouse increased absorption of the fatty acids, but large amounts caused a loss in weight (Schettler—*Biochem. Z.* 319, 444). The absorption of the fat was said to improve through esterification with cholesterol and the loss in weight was attributed to increased transport of fatty acids.

Other information on cholesterol metabolism pertained to the process in the diseased state.

LIPIDES IN DISEASES. In studies on vascular diseases many cholesterol analyses were made on body fluids and substances. Ultracentrifugal analyses of blood lipides by Gofman *et al.* (*Circulation* 2, 161; *Science* 111, 166) revealed the presence of a certain class of cholesterol compounds associated with atherosclerosis and associated diseases. Moreton (*J. Lab. & Clin. Med.* 35, 373) associated such diseases with the particle size of blood lipides and the individual's intolerance to fat. Analyses of blood cholesterol and cholesterol ester levels of vascular diseased and normal persons revealed no significant differences (Gertler *et al.*—*Circulation* 2, 380; Morrison—*Am. J. Med. Sci.* 216, 32). Eskimos that lived on a high fat-high cholesterol diet had high serum cholesterol levels, but almost no vascular-renal diseases (Wilber & Levine—*Exptl. Med. Surg.* 8, 422). Data in the preceding few reports was used to reflect doubt on the causative role of serum cholesterol levels in atherosclerosis. A study of diets of normal and essentially hypercholesteremic family groups revealed no relation between the total serum cholesterol and the compositions of the food eaten (Wilkinson—*Arch. Internat'l Med.* 85, 389). Cholesterolemia was reduced with administration of thyroid material (Stamler *et al.*—*J. Lab. & Clin. Med.* 35, 351; *Circulation* 2, 523) and with continued use of vitamin C (Myasnikov—*Klin. Med. U.S.S.R.* 28, No. 2, 3). Choline treatment of patients (Morrison & Gonzales—*Proc. Soc. Exptl. Biol. Med.* 73, 37) and rabbits (Firstbrook—*Ibid.* 74, 741) disposed to hypercholesterolemia and atherosclerosis inhibited both processes. Edema fluid cholesterol averaged 14.6 mg. % in patients with congestive heart failure and 175.2 mg. % in cases of lymphatic obstruction

(White & Sachs—*Science* 112, 18). In human autopsy specimens, the total lipide cholesterol ester and fatty fractions of the adrenal fat were not elevated in hypertension or atherosclerosis when compared to those found after accidental death (Adams & Baxter—*Arch. Path.* 48, 13). The ratios of esterified cholesterol to total cholesterol in the serum of dogs, swine, cows, horses, and geese were recorded (Darraspen *et al.*—*Compt. rend. soc. biol.* 143, 1419). The ratios were below normal in force-fed geese and in cases of hepatic insufficiency.

Abnormalities in lipide metabolism were observed in several diseased states. In diabetic men the lipolytic activity of ultra-articular fat was about 25% of the corresponding activity in the tissues of diabetic females (Renold & Marble—*J. Biol. Chem.* 185, 367). Although liver slices from normal rats and cats readily convert labeled acetate to long chain fatty acids, the livers of alloxanized rats and pancreatectomized cats were relatively incapable of carrying out this process (Brady & Gurin—*Ibid.* 187, 589). Microscopic technic showed that there was considerably more fat in the parenchymal cells as compared to the Kupffer cells in fatty and cirrhotic livers, the fat in the Kupffer cells was increased in diabetics and most patients dying of various malignant tumors, and fat was also noted in the fibrous trabeculae in Laennec's cirrhosis (Volk—*Am. J. Digestive Dis.* 17, 394). Unsaturated fats, especially codliver oil (Hamel & Piller—*Beitr. Klin. Tuberk.* 103, 239), and side chain acids (Weitzel—*Fette u. Seifen* 52, 670) were said to have antituberculosis effects. Development of tuberculosis lesions was accelerated by subcutaneous injections of ethyl oleate (Negre & Bretey—*Compt. rend. soc. biol.* 143, 963). This action was retarded by simultaneous injections of ethylsuccinate. Nineteen of 32 mice of varying leukemic state showed fatty acid oxidase activity of liver homogenates less than 50% of the theoretical (Vestling *et al.*—*Cancer Research* 9, 639). Fat absorption was defective in most patients following vagotomy (Fox & Grimson—*J. Lab. & Clin. Med.* 35, 362). In Wallerian degeneration there was a steady decrease of neutral fat in the peripheral nerve (Johnson *et al.*—*Nature* 164, 108). In the cow serum free fatty acids rise during parturition, ketosis, and milk fever (Craigie—*J. Am. Vet. Med. Assoc.* 117, 107). Chronic intoxication of the dog by hexachlorocyclohexane caused abnormal intracellular deposits of fat, most marked in liver, striated muscle nerve cells, and kidney (Dellemagne *et al.*—*Science* 112, 148).

MICROBIOLOGY OF LIPIDES. The influence of various fatty acids on the growth of some bacteria was investigated. Methyl-3-keto-11-eicosenoate was as active as methyl oleate in replacing biotin in the nutrition of *Lactobacillus arabinosus* (Mitz *et al.*—*J. Am. Chem. Soc.* 72, 1231). Palmitoleic acid supported good lactic acid production of a mutant strain of *Lactobacillus bifidus* at low levels of concentration, and inhibited lactic acid production at high levels, in contrast to oleic acid which supported acid production at both high and low levels of addition (Hassinen *et al.*—*Arch. Biochem.* 25, 91). In tests on a straight rod strain and *Lactobacillus bifidus*, the fatty acids liberated from human and cows milk by pancreatic digestion inhibited growth, lauric and myristic acids being the most toxic to the microorganisms (Tomarelli *et al.*—*J. Biol. Chem.* 187, 197). Bovine

serum albumin, dialyzed human whey, and surface-active compounds afforded protection against the inhibition by the fatty acids. When measuring the oleic acid stimulation of *Lactobacillus casei* different results were often obtained turbidimetrically as compared to titrimetrically (Williams & Andrews—*J. Bact.* 60, 215). A suggested explanation was that oleic acid may interfere with glycolysis so that less acid was formed whereas cell growth was unhampered. Fatty acids also inhibited growth of Rosenbach strain of *Micrococcus pyogenes* (Wynne & Foster—*J. Infectious Dis.* 86, 33).

A *Neurospora* mutant which required acetate could obtain it by degrading fatty acids, except that it could not use palmitic and stearic acids as acetate sources (Lein & Lein—*J. Bact.* 60, 185). It was suggested that acetaldehyde rather than acetyl phosphate was more probably the source of acetate for synthesis of lipides by *Tetrahymena geleii* (Seaman—*J. Biol. Chem.* 186, 97). *Neisseria catarrhalis* oxidized even-carbon straight chain saturated fatty acids more rapidly than odd-carbon acids (Randles—*J. Bact.* 60, 627). This data suggested that the oxidation was in accord with the theory of β -oxidation.

Composition and Characteristics

The communications in which methods of analyses, characteristics, or composition of fats and oils seemed to be the principal contribution are reviewed in this section. Papers cited in other sections of this review contain some such information as related to the main text of the individual papers. For example, some comprehensive treatises on fats and oils cited in the introduction contained analytical data; the section on deterioration contained references to methods for testing for spoilage and stability; papers of the previous section contained methods and analyses incidental to biochemical work, and the analytical methods for soap products were classified in the review on detergents.

For convenience of presentation some analytical data were tabulated.

Only the scope of the text of a small number of papers will be mentioned because the information could not be tabulated or they were reviews, general discussions, or compilations of data. Barker & Hilditch (*J. Sci. Food Agr.* 1, 118) analyzed samples of African sunflower-seed oils to ascertain the influence of soil, geographical region, climate, and variety on the composition. How the same influences affect the characteristics (Buzi—*Ann. sper. agrar., Rome*, 3, 409) and yield (Castorina—*Olearia* 3, 88; Pantanelli & Brandonisio—*Ibid.* 77) of olive oils in Italy were also recorded. The effects of seasonal manufacturing variations on the hardness of New Zealand butters were statistically analyzed for a three-year period (Dolby—*J. Dairy Research* 16, 336). Analyses of the colostrum fat of goat and sheep showed gradual increases in Reichert, Polenske, and saponification values and decreases in iodine values and refractive indexes after parturition (Paul & Anantakrishnan—*Indian J. Dairy Sci.* 3, 7). The analytical constants of seven Spanish fish oils were determined (Aenlle—*Anales fis. y quim., Madrid*, 40, 565). The characteristics of German synthetic fats were determined, and the data were discussed from the standpoint of stability and utility of the fats (Schulte—*Pharm. Zentralhalle* 86, 97).

Some reports on analytical methods were of a comprehensive nature. V. C. Mehlenbacher *et al.* (*J. Am. Oil Chemists' Soc.* 27, 10) and Andrews *et al.* (*Ibid.* 350) summarized reports of subcommittees of the American Oil Chemists' Society dealing with all types of analyses on oleagenous materials. Martinghi (*Olearia* 3, 725, 807, 888) compared the American Oil Chemists' Society methods with those of the comparable Italian society and made several proposals for amending the Italian methods. Reviews on analytical methods employed in fat chemistry were published by Wolff (*Bull. mens. ITERG* 3, 418) and Holmberg (*Svensk Kem. Tid.* 62, 5). General analytical data on vegetable oils suitable for margarine manufacture were given by Grace (*Food in Canada* 9, No. 2, 28). British standards were issued on vegetable oils (*Brit. Standards* 628-32, 650-6), technical cod oil (*Ibid.* 1582), and methods for the analysis of fats and oils (*Ibid.* 684).

ANALYSIS OF FAT SOURCES. The drying of seeds for moisture determination by infrared rays was used by a French concern for many years (Fauve—*Bull. mens. ITERG* 3, 323). Their experience showed that moisture may be determined in about 10 minutes with errors not exceeding 0.6% as compared to the oven method. Determination of moisture in oil seeds by the titration method of Fischer showed good reproducibility and the results were up to 1.9% higher than those by the official oven method (Francois & Sergeant—*Ibid.* 4, 401).

The report of the seed and meal analysis committee of the American Oil Chemists' Society contained specifications of the methods for sampling and analysis of tung fruit (Hopper *et al.*—*J. Am. Oil Chemists' Soc.* 27, 21). Pack *et al.* (*Ibid.* 164) determined the amount of oil that could be extracted from tung fruit according to standard analytical methods as a function of grinding and moisture content. It was shown that plate spacing in the Bauer laboratory mill used for the grinding should be lower than 0.020 inch and moisture was without influence in one method, but in another wet kernels had to be dried to less than 10% moisture.

A newly manufactured modification of the Soxhlet apparatus for determination of fat in seeds was described (Hansen—*Tids. Planteavl* 53, 354). A procedure for fat in seeds by Govantes (*Anales bromatol. Madrid* 1, 353) involved repeated extraction with benzene and separation of the solvent-oil mixtures by centrifuging. The methods described for the determination of the fatty acids in tall oil were based on the determination of the unsaponifiable material, selective esterification of the fatty acids with lower alcohols, and titration of the rosin acids (Hezel—*Fette u. Seifen* 52, 149; Linder & Persson—*Svensk Papperstidn.* 52, 331). Formulas for the calculation of the amount of fatty materials were included with the methods.

Other fat procedures pertained to the determination of the composition of foods. The butyrometric methods for fat content in milk were modified. The Babcock method was standardized by increasing the volume of sample and eliminating the meniscus in reading the fat content (Herreid *et al.*—*J. Dairy Sci.* 33, 685). An equation was designed to adjust fat values obtained by the Gerber method so that they agree more closely with those of the Rose-Gottlieb method (Mulder & Radema—*VII Congr. intern. inds.*

<i>Ocimum litimandacharticum</i> seed ⁸	12.5	1.4852 ²⁵	192.1	192.6				0.4
<i>Ocimum viride</i> seed ⁸	9.7	1.4808 ²⁵	192.2	168.9				1.1
<i>Parnassium laurinum</i> seed kernel ¹	12-	1.5480-	176.8-		2.8-			0.9-
	12.9	1.5627 ⁴⁰	180.8		18.8			1.0
<i>Pistacia vera</i> seed kernel ³²	38.8-	0.9133 ²⁰	194.3	83.7	0.7	68.9		0.8
	40.6							
<i>Pistacia terebinthus</i> seed kernel ³²	39-	0.9146 ²⁰	194.3	86.9		68.6		1.0
	43.8							
<i>Pistacia kdnjuk</i> seed kernel ³²	57.6	0.9157 ²⁰	194.5	98.8	1.7	73.7		0.54
Plum seed ³³		0.917 ²⁰	190.0	110.2	1.0			0.4
Papaver Rhoeas seed (pressure extd.) ³⁴		0.9202 ^{20/4}	189.1	137.6	5.3	78.5		-16-
					(0.9)			-18
Safflower (of Kenya) seed ³⁵	28.6	1.4741 ²⁵	191.2	142.5				0.8
Sea (elephant) blubber ³⁶								
Female								
Male			191.7	152.9	1.1			0.6
			192.1	136.4	1.5			0.6
Sesame (3 American varieties) seed ³⁷	51.2-	0.9188-	188.0-		(0.5-	75.3-		1.6-
	55.6	0.9193 ²⁵	188.3	111.6	1.8)	76.2	(1.1-	0.2-
							4.1)	0.5
Snake fat lobes ³⁸	43.0	0.9268 ^{24/4}	192.6	104.4	(0.5)	77.2		0.07
Mocasin	70.0	0.9323 ^{25/4}	193.5	114.0	(0.2)	84.0		0.13
Prairie Rattler	33.0	0.9252 ^{25/4}	194.7	89.6	(0.3)	70.1		0.13
Bor Constrictor		0.9274 ¹⁵	190.1	143.0			(8.6)	0.6
Spitzklette seeds ³⁹	33.3-	1.4772 ²⁰	190.0-		2.6			0.3
	38.3	1.4709-	191.6		(0.3-			0.3-
<i>Xanthium riparium</i>	24.4-	1.4749 ²⁵	191.6		1.2)			1.3
Sunflower (various variety & origin) seed ⁴⁰	34.4	1.4760-	183.1-	138.3-	1.5-			1.5-
		1.4766 ²⁰	191.3	143.4	9.1			2.9
Tobacco (of Turkey) seed ⁴¹		1.4742-	188.5-	140.3-	(0.8-			1.9-
	30.5-	1.4756 ²⁵	190.3	145.3	2.6)			2.5
Tobacco (various variety & origin) seed ⁴¹	36.0							

FAT ACID COMPOSITION

Oil or Fat Source	Common Saturated Acids			Common Unsaturated Acids			Other Fatty Acids
	C ₁₄ Myristic	C ₁₆ Palmitic	C ₁₈ Stearic	C ₁₈ (-2H) Oleic	C ₁₈ (-4H) Linoleic	C ₁₈ (-6H) Linolenic	
<i>Anemum visnaga</i> ³		5		32.0	13.0		Petroselinic 50
<i>Artemisia scoparia</i> ⁴	5.1	23.5	24.4	10.1	11.3		C ₃₀ 18.0
Bader body ⁵				30.9	8.4	3.9	C ₁₄ (-2H) 1.1, C ₁₆ (-2H) 6.2, C ₃₀ (-5.1H) 14.6
Black sesame seed ⁶	5.7	21.2	8.2	4.8	15.7	66.9	C ₃₀ 3.9
<i>Hyptis spicigera</i>		8.7		9.2	23.3	59.9	
<i>Hyptis spicigera</i>		4.4	3.2	48.5-52.0	20.7-24.1		
<i>Cleophratum inophyllum</i> seed kernel ¹⁰		17.7-18.5	6.1-10.7	24.9	52.1		C ₃₀ 0.6
<i>Chrozophora plicata</i> seeds	0.2	9.0	13.2	29.8	47.9	8.3	Lower than C ₁₉ 0.63
Cornelian Cherry stones ¹²		8.8		38.4	39.6		C ₁₀ 1.6, C ₁₂ 43.0
<i>Ocimum sativum</i> seed kernel ¹³		4.12	16.3	2.6	0.2		C ₂₂₋₂₄ 1.0
Dika (West Africa) seed kernel ¹⁴	46.4	6.2		7.0	71.7	10.2	C ₃₀ 0.4, C ₃₂ 0.3, C ₃₄ (-2H) 1.9, C ₃₆ (-3.5H) 15,
<i>Eurotia biennis</i> seed ¹⁵		8.8	1.3	2.0	12.0	63.3	C ₃₀ (-9.4H) 25.8, C ₃₀ (-9.2H) 10.6
<i>Euphorbia calycina</i> ¹⁶		21.6		6.9	17.2	54.3	C ₁₂ 0.4, C ₃₀ 0.4, C ₃₂ 0.6, C ₃₄ (-2H) 2.8,
<i>Euphorbia erythraea</i> ¹⁷		21.6					C ₁₅ (-3H) 14.1, C ₃₀ (-7.9H) 19.4, C ₃₂ (-5.3H) 20.7
Fish body ¹⁸	6.7	17.4	2.1				C ₃₀ 1.1, C ₃₄ (-2H) 0.4, C ₃₄ (-2H) 2.2,
Pitchard							C ₃₀ and ₃₂ unsatd. 0.4
							C ₁₂ 0.2, C ₃₀ 0.1, C ₃₄ (-2H) 0.6, C ₃₆ (-2H) 8.3,
Maasbanker (or horse mackerel)	7.3	13.1	2.0				C ₃₀ (-4H) 0.7
Hippocampus body ²²	2.3	27.1	22.2	39.3	3.5	1.5	C ₁₄ (-2H) 0.8, C ₁₆ (-2H) 7.8, C ₃₀ (-4H) 1.7
Horses	3.1	25.3	5.1				C ₃₀ 0.7, C ₃₄ (-2H) 0.6, C ₃₆ (-2H) 18.8, C ₃₀ (-4H) 2.4
Bone	1.1	27.4	1.7				
Offal	0.8	17.9	2.5				
Hoof							

FAT ACID COMPOSITION

Oil or Fat Source	Common Saturated Acids			Common Unsaturated Acids			Other Fatty Acids
	C ₁₄ Myristic	C ₁₆ Palmitic	C ₁₈ Stearic	C ₁₈ (-2H) Oleic	C ₁₈ (-4H) Linoleic	C ₁₈ (-6H) Linolenic	
<i>Luffa cylindrica</i> seed ⁹		9.6	18.9	6.9	64.6		
<i>Macadamia ternifolia</i> seed kernel ²³	1.6	8.0	3.3	59.3	2.2		C ₂₀ 2.2, C ₂₂ 0.8, C ₁₈ (-2H) 20.4, C ₂₀ (-2H) 2.2
Melon (inedible variety) seed ⁹		4.0	22.3	67.5	6.2		
<i>Citrullus vulgaris</i>							
Niger seed ³⁰		7.9	10.6	7.0	72.6	0.9	C ₂₀ 1.0
<i>Guizotia abyssinica</i>							
<i>Ocimum klamaidscharicum</i> seed ⁸		13.9		5.2	16.1	64.8	
<i>Ocimum viride</i> seed ⁸		14.1		14.3	32.5	39.1	
<i>Passiflora laurinum</i> seed kernel ²¹		4.0	1.0	7.5	3.5	84.0	
Safflower (of Kenya) seed ³⁵	Trace	6.4	3.1	13.4	76.9		C ₂₀ 0.2
Seal (elephant) blubber ³⁸	4.4	11.5	2.5		33.0 (-2.4H)		C ₁₂ 0.1, C ₁₄ (-2H) 1.3, C ₁₆ (-2.1H) 13.0, C ₂₀ (-5.9H) 20.6, C ₂₂ (-10.8H) 13.7
Female							C ₂₀ (-5.4H) 18.8, C ₂₂ (-10H) 10.6
Male	5.2	11.4	2.4		35.1 (-2.7H)		
<i>Silllingia tallows</i> (fruit coats) ⁴⁰				27.1	1.6		
<i>Sapium sebiferum</i> (Chinese)	0.5	63.2	7.6	20.4	1.0		C ₁₂ 1.5
<i>Sapium sebiferum</i> (American)	3.4	72.1	1.6	46.4	3.1		
<i>Sapium discolor</i> (Chinese)	1.7	46.8	2.0				
Sugar Cane	2.6	21.1	3.9	8.6	30.5	5.8	
Sunflower (various variety & origin) seed ⁴³		8.7-14.2		14.1-43.1	44.2-75.4		C ₈ 1.7, C ₁₀ 1.0, C ₁₂ 2.8, C ₂₀ 6.4, Non-fatty acids 15.6
Tobacco (various variety & origin) seed ⁴⁴		9.8-13.1		7.8-20.1	68.2-73.8		
Tea seeds ⁴⁵			1.2	86.7	6.8		
<i>Thea sinensis</i>		4.9					

REFERENCES ON THE CHARTS

- N. Ito. *J. Agr. Chem. Soc. Japan* 17, 1005.
- H. J. Deuticke et al. *Naturwissenschaften* 36, 60.
- D. N. Grindley. *J. Sci. Food & Agr.* 1, 53.
- D. B. Parihar & S. Dutt. *Indian Soap J.* 15, 161.
- S. Gupta et al. *J. Chem. Soc.* 1950, 3145.
- T. Yazicioglu. *Fette u. Seifen* 52, 593.
- I. Cornea & A. Rudenco. *Bull. études et recherches tech., Bucharest* 1, 169.
- C. Barker et al. *J. Soc. Chem. Ind.* 69, 71.
- D. N. Grindley. *J. Sci. Food & Agr.* 1, 152.
- O. Hata. *J. Soc. Chem. Ind. Japan* 44, 481.
- P. R. Bhandari et al. *J. Sci. Ind. Research India* 9 B, No. 3, 60.
- G. Rankov & A. Popov. *Annuaire univ. Sofia Faculté phys.-math.* 43, No. 2, 119.
- P. Soni et al. *J. Sci. & Ind. Research India* 8 B, No. 11, 210.
- J. Kobilic. *Chem. Obzor* 24, 118, 129.
- S. Ueno. *J. Nippon Oil Technol. Soc.* 2, No. 5, 10.
- M. L. Meara & C. B. Patel. *J. Sci. Food & Agr.* 1, 48.
- J. P. Riley. *J. Chem. Soc.* 1949, 2728.
- M. M. Black & H. M. Schwartz. *J. Sci. Food & Agr.* 1, 248.
- D. S. Bolley et al. *J. Am. Oil Chemists' Soc.* 27, 571.
- P. Soni & J. S. Aggarwal. *J. Sci. Ind. Research India* 8 B, No. 9, 150.
- T. Yazicioglu. *Fette u. Seifen* 52, 325.
- C. Barker & T. P. Hilditch. *J. Chem. Soc.* 1950, 3141.
- E. G. Brooker & F. B. Shorland. *Biochem. J.* 46, 80.
- N. E. Bühner. *Rev. quim. ind. Rio de Janeiro* 19, No. 216, 21.
- A. S. Neto. *Anais assoc. quim. Brasil* 6, 99.
- R. H. Wiley & A. W. Cagle. *J. Am. Oil Chemists' Soc.* 27, 34.
- K. Täufel & C. Franke. *Fette u. Seifen* 52, 201.
- R. E. Bridge & T. P. Hilditch. *J. Chem. Soc.* 1950, 2396.
- F. von Gizycki & G. Kotitschke. *Fette u. Seifen* 52, 454.
- H. C. Dunn & T. P. Hilditch. *J. Soc. Chem. Ind.* 69, 13.
- J. P. Riley. *J. Chem. Soc.* 1950, 12.
- T. Yazicioglu. *Fette u. Seifen* 52, 6.
- L. Haskó. *Mezgazdasag es Ipar* 2, No. 11, 27.
- W. Awe & G. Kunert. *Fette u. Seifen* 52, 268.
- C. Barker & T. P. Hilditch. *J. Soc. Chem. Ind.* 69, 15.
- G. Winter & W. Nunn. *J. Sci. Food Agr.* 1, 18.
- F. G. T. Menezes et al. *J. Am. Oil Chemists' Soc.* 27, 184.
- C. B. Pollard & J. McLaughlin Jr. *J. Am. Oil Chemists' Soc.* 27, 393.
- J. Fischer & A. Patzenhauer. *Fette u. Seifen* 52, 137.
- S. S. Gupta & M. L. Meara. *J. Chem. Soc.* 1950, 1337.
- S. R. Alpar & S. Esin. *Rev. faculté sci. univ. Istanbul* 14 A, 65.
- D. E. Whyte & B. Hengeveld. *J. Am. Oil Chemists' Soc.* 27, 57.
- C. Barker et al. *J. Soc. Chem. Ind.* 69, 15, 16, *J. Sci. Food & Agr.* 1, 140.
- R. V. Crawford & T. P. Hilditch. *J. Sci. Food & Agr.* 1, 230.
- V. P. Gognadze et al. *Zhur. Anal. Khim.* 5, 308.

agr. Paris 1948, K1). The Schain butyrometric technic, in which fat was released from milk by treatment with isopropyl alcohol and nonionic detergent, in ethyl alcohol, and then treatment with anionic detergent gave good results without the disadvantage of charring as may occur in the Babcock method (Gershenfeld & Ucko—*J. Milk & Food Tech.* 13, 175). The Gerber fat method was modified for use on various foods (Egiazarov—*Gigienna i Sanit.* 1950, No. 5, 45).

In an analytical extraction procedure for fat in butter centrifuging was used to separate the phases and particular precautions to use peroxide-free ether solvent were mentioned (Terrier—*Mitt. Lebensm. Hyg.* 40, 245, 247). The fat in cheese was determined by dispersing in aqueous solution made alkaline with ammonia and extraction with petroleum ether (Peter—*Proc. 12th Intern. Dairy Congr.* 2, 841). Rapid phase separation was obtained by centrifuging. Likewise, the rapidity of a plant control fat determination for cacao products was improved by use of the centrifuges (Kobe—*Anal. Chem.* 22, 700). Where the fat was extracted from dairy products for fat acidity determination, churning in aqueous solution with a Waring Blendor gave good results (Bowen *et al.*—*Sci. Agr.* 29, 551). Fat in skim milk and in nonfat dry milk solids could be determined by extraction and measuring the monolayer area on a prepared surface of a solution of 0.2% acetic acid (Heinemann & Rohr—*J. Dairy Sci.* 33, 703). In another micromethod the fat was spread on 0.3% sulfuric acid (Jones—*Science* 111, 9).

Collaborative work showed that good results for fat in eggs were obtained by the acid hydrolysis method (Mitchell—*J. Assoc. Off. Agr. Chemists'* 33, 699). Acid hydrolysis methods were recommended for determination of fat in feed for the standard methods were too tedious (Dangoumau *et al.*—*Bull. mens. ITERG* 3, 453). The Gerber butyrometric procedure for fat in milk was applied for the rapid estimation of fat in sausage (Talbot—*Analyst* 74, 462), and chicken meat (Moxon *et al.*—*Proc. S. Dakota Acad. Sci.* 26, 34). Bound fat in flours, corn meal, oat flakes, and baked goods was more efficiently extracted when the substance was first treated with the surface active agent, "Zeptherol" (Kiermeier & Patschky—*Z. Lebensm. Untersuch. u. -Forsch.* 90, 98). However, with dried yeast the method was unsuccessful.

Tetrahydrofuran was recommended as the extracting agent for fat determination in blood and serum (Cremer—*Klin. Wochschr.* 27, 755).

QUALITY TESTS. The chromatographic procedure of the International Chemical Union for determining the refining efficiency of crude oils was demonstrated to be reasonably accurate, easy to carry out, and required no special, elaborate, or expensive equipment (Linteris & Handschumaker—*J. Am. Oil Chemists' Soc.* 27, 260). Therefore, it was suggested for use to evaluate crude oils which are traded in commercial channels. The American Oil Chemists' Society Refining Committee advised that a special refining test could be run at a single temperature with good results (James *et al.*—*Ibid.* 236). Desnuelle & Micaelli (*Oleagineux* 5, 161) criticized refining loss evaluation tests, for only the neutral oil lost was considered, but losses of glycerol, lecithin, and other materials should also be important for evaluation of the oils. Sesame

oils from different varieties of sesame seed (Menezes *et al.*—*J. Am. Oil Chemists' Soc.* 27, 184) and solvent-extracted and hydraulic pressed okraseed oils (Hussain & Dollear—*Ibid.* 295) were evaluated by recording refining loss, color, and the characteristics of refined and hydrogenated products derived therefrom.

Since linseed oil is not always alkali refined for use the amount of foots that settles from an acetone solution or on heating to 105-110°C. served for evaluation in place of the laboratory refining test. The rapidity for determining the foots from acetone solution was increased by using centrifugal force to compact the foots layer instead of depending on gravity (Freyer & Shelburne—*J. Am. Oil Chemists' Society* 27, 545). Dooper (*Verfkroniek* 23, 84) recommended drying tests for evaluation of linseed oils, because drying properties do not always parallel the iodine value which has been most used to approximate drying behavior. Barker & Hilditch (*J. Oil & Colour Chemists' Assoc.* 33, 6, 24, 49) compared the characteristics and composition of several oils with their drying characteristics and suggested that oil suitable for drying could be evaluated from the linoleic acid content which should not be less than 66% of the total fatty acids.

According to Wolff (*Bull. mens. ITERG* 4, 165) a mixture of 67% of dioxane and 33% H₂O was an excellent solvent for extraction in the analysis of soap stock for fatty material. Bauschinger (*Fette u. Seifen* 52, 693) discussed the merits of the indicator bromophenol blue in fat testing, particularly for the detection of soap in refined fats and acidity in crude fats.

The annual report of the American Oil Chemists' Society Color Committee indicated that a good correlation of spectrophotometric method with Lovibond red was obtainable from equations involving transmittance readings at 460, 550, 620, and 670 m μ (Agee *et al.*—*J. Am. Oil Chemists' Soc.* 27, 233). Ciusa & Nebbia (*Olearia* 4, 108) discussed the work of this committee and proposed that the spectrophotometric data be converted into the trichromatic expressions recommended by the International Commission on Illumination, for these expressions were easily related to experimentally obtained Lovibond red data. Paltauf (*Fette u. Seifen* 52, 370) suggested that oil color readings could be reported in "iodine color numbers," which was defined as the milligrams of free iodine in 100 cc. of an aqueous potassium iodide solution, the color of which matches the sample.

CHEMICAL CHARACTERISTICS. Benham & Klee (*J. Am. Oil Chemists' Soc.* 27, 127, 130) modified the Rosenmund-Kuhnhehn iodine determination by using mercuric acetate as a reaction catalyst. With ordinary nonconjugated oils only one minute reaction time was necessary, and with conjugated oils good results were afforded with 30-120 minutes' reaction time. Bartl (*Chem. Listy* 37, 98, 109) found that with the Hanus iodine method 400% excess reagent was necessary to obtain suitable iodine values on dehydrated castor oils. A new procedure for iodine determination made use of *N*-chloro-*p*-tholunesulfonamide as the halogenating reagent (Kael & Fink—*Ibid.* 34). Mead & Houton (*Anal. Chem.* 22, 1204) determined unsaturation of oils by hydrogenation in a Barcroft-Warburg apparatus.

In the determination of diene (or conjugated) unsaturation, some conjugated products of linoleic acid do not fully react with the maleic acid reagent. Von

Mikusch (*Z. Anal. Chem.* 130, 412; *Angew. Chem.* 62, 475) obtained the desired reaction by adding one milliliter of 0.1% solution of iodine in acetone to the reagent. He proposed that diene numbers so obtained be called pandiene numbers.

The new literature on the determination of acetyl or hydroxyl value of oils was a review in which slight modifications were suggested for some standard methods (Aenlle & Val—*Anales fis. y quim Madrid*, 40, 414), and a procedure for determination of both hydroxyl value and the amount of glycerol present was designed (Habicht—*Fette u. Seifen* 52, 174). Directions were published for measuring of ketonic fatty acids, i.e., carbonyl value, of fats (Gorbach—*Fette u. Seifen* 52, 405).

Three modified saponification value determinations were proposed. Hezel (*Farbe u. Lack* 56, 10) saponified the oil, titrated with thymolphthaleine indicator to neutralize excess lye and then titrated the soap using bromophenol blue as the indicator. Cherbuliez & Chams (*Festschrift Paul Casparis 1949*, 54) partially saponified dark-colored oils, extracted the soaps, and completed saponification on the residue with excess lye reagent. Täufel & Jacob (*Fette u. Seifen* 52, 10) combined the Reichert-Meissl value determination with that of saponification value by removing alcohol from the saponification value residue and then steam distilling.

PHYSICAL TESTS. Dielectric behavior of fat derivatives obtained by Crowe & Smyth (*J. Am. Chem. Soc.* 72, 1098) was found to confirm thermal observations with regard to polymorphism. The data was also useful as a source of the true melting points of the fatty materials used in the investigation. The same authors (*Ibid.* 5281) recorded similar information for some di- and triglycerides. The data checked X-ray diffraction observations, and the diglycerides were found to show no appreciable amount of dipole orientation. The dielectric data on methyl palmitate that was determined by Cook & Buchanan (*Nature* 165, 358) showed the variations that were caused by different frequencies and at temperatures from -80 to 22°C .

Surface areas calculated for several acids between C_7 to C_{20} on absorption on carbon black were in fair agreement with areas calculated by other methods (Smith & Hurley—*J. Phys. & Colloid Chem.* 53, 1409). The surface tension measurements of purified normal fatty acids from C_{10} to C_{19} , inclusive, revealed an alternation between odd and even acids similar to that in the corresponding melting point curves (Wachs *et al.*—*Kolloid -Z.* 114, 14). The literature on this alternation behavior of the physical constants, i.e., melting point, lattice constant, and surface tension of fatty acid homologs was reviewed (Wachs—*Fette u. Seifen* 52, 456).

Viscosity technic was recommended by Edwards (*J. Soc. Chem. Ind.* 69, 193) to locate the change in phase, gelling point, and similar phenomena. In fat-chemistry the technic could be used to determine breakdown of lubricant soap greases, efficiency of emulsification, effect of pH on emulsifiers, and for other purposes where changes of physical structure take place.

The relative hardness of fats based on the temperature at which a specific degree of crystallization was reached and which was designated as the cloud point could be determined with a newly patented photoelectric apparatus (Clardy—*U. S.* 2,498,941). The

apparatus was designed as a rapid control means in the manufacture of shortening. Another new consistency measurement method was based on the temperature at which a metallic weight falls through a sample of fat that was being gradually heated (Schlenker & Fidaleo—*J. Am. Oil Chemists' Soc.* 27, 80). In a review on consistency measurement of butter, apparatus for determining cutting pressure, bending, and spreadability were illustrated and described (Mohr—*Fette u. Seifen* 52, 342).

The heat contents of hydrogenated and unhydrogenated peanut oils were tabularly and graphically recorded for temperatures from 84° to 350°K . and relation of this data to solid glyceride content and consistency was discussed (Ward & Singleton—*J. Am. Oil Chemists' Soc.* 27, 423).

A review on polymorphism of fats by Lutton (*Ibid.* 276) covered the saturated single acid triglycerides, the mixed C_{16} to C_{18} saturated triglycerides, saturated diglyceride, and saturated monoglycerides. Examples were given of the value of polymorphism in identifying naturally occurring glycerides. Lutton & Jackson (*J. Am. Chem. Soc.* 72, 3254) added to this knowledge by determining the melting point of four forms of 2-oleoyldipalmitin. This data did not agree with those of some others with regard to the existence of a so-called vitreous form of fats. New data of this type was recorded for synthetic unsaturated diacid diglycerides (Benedict *et al.*—*J. Am. Oil Chemists' Soc.* 27, 91). One form to two polymorphic forms of the compounds were revealed. According to Singleton *et al.* (*Ibid.* 143) stearic acid crystallized from benzene exhibited X-ray patterns corresponding to the B-form and crystallized from acetic acid the C-form was developed. The polymorphic forms of C_{18} fatty acids and triglycerides were characterized as to the course of the melting curve, formation of solid solutions, formation of eutectics, and microcrystal structure (Ravich *et al.*—*Izvest. Sektora Fiz-Khim. Anal. Inst. Obschei i Neorg. Khim. Akad. Nauk S.S.S.R.* 15, 47; 16, No. 3, 142).

X-ray diffraction powder patterns were recorded for silver, amide, and anilide derivatives of fatty acids for the purpose of serving for identification (Matthews *et al.*—*Anal. Chem.* 22, 514). For the same purpose, X-ray diffractions and melting point-composition studies were recorded for 9, 10-epoxy- and dihydroxystearic acids and 9, 10-epoxyoctadecanol (Witnauer & Swern—*J. Am. Chem. Soc.* 72, 3364).

COMPOSITION. Many methods and aids have been suggested for the analysis of free fatty acid mixtures from fats and oils. Paquot (*Bull. soc. chim. France* 1950, 400, 402, 404) developed mathematical formulas for determining the composition of mixtures of palmitic, stearic, and oleic acids from iodine and acid number data and he used freezing point diagrams to determine the composition of binary and ternary mixtures of saturated acids. According to Peters & Clark (*J. Am. Oil Chemists' Soc.* 27, 201) the composition of commercial mixtures of stearic and palmitic acids could be approximated from the titer using a prepared curve which showed the relationship of composition to the titer. The melting-point behavior of mixtures of tetra- and hexabromides of linoleic and linolenic acids were determined (Paschke & Wheeler—*Ibid.* 9). The data was considered approximate and would require more study before an empirical method for estimating the fatty acids could be based on these

curves. A microtitration neutralization equivalent method was developed for the analysis of binary mixtures of fatty acids (Tous & Pizarro—*Anales real soc. espan. fis. y quim.* 46B, 105).

Fatty hydroxamic acids were determined by hydrolysis with known excess of ethanol-hydrochloric acid solution and the excess hydrochloric acid was titrated (Roe & Swern—*Anal. Chem.* 22, 1160). The method was designed for determination of degree of conversion in a study of the reaction of fats with hydroxylamine.

A review on the analytical separation of constituents of fats by paper chromatography was prepared by Kaufmann (*Fette u. Seifen* 52, 331). Chromatography methods for determination of the unsaponifiable matter in fats were being investigated by the Association of Official Agricultural Chemists (Kirsten—*J. Assoc. Off. Agr. Chemists'* 33, 748). Howard's & Martin's (*Biochem. J.* 46, 532) chromatographic procedure for separating fatty acid involved use of kieselguhr treated with dichlorodimethylsilane as the stationary phase and aqueous methanol-octane or acetone-medicinal paraffin as solvents. In this system the stationary phase was less polar of the two phases. In a similar system, Boldingh (*Rec. trav. chim.* 69, 247) used natural or synthetic rubber containing absorbed benzene as the stationary phase and strong polar solvents for the mobile phase. Hagdahl & Holman (*J. Am. Chem. Soc.* 72, 701; *J. Biol. Chem.* 182, 421) obtained more sensitive separation by depressing the solubility of the fatty acids. Thus, C₁ through C₆ acids were separated with water as the mobile solvent, C₆ through C₁₂ acids were separated from 50% alcohol, C₁₀ through C₁₄ acid required 65% alcohol, etc. A new oxidation method for determining saturated fatty acids was based on nondisruptive oxidation of the unsaturated acids by performic acid, extraction of the oxidized mixtures with petroleum ether, and finally chromatographic purification of the saturated acids (Fitelson—*J. Am. Oil Chemists' Soc.* 27, 1).

Construction and use of nomographic charts for the calculation of the fatty acid composition of fats from the iodine and thiocyanogen values were described (Hussain & Dollear—*Ibid.* 206).

General informational communications on the application of spectrophotometry for analysis of fats and oils were written by Binkerd & Harwood (*Ibid.* 60), Kaufmann *et al.* (*Fette u. Seifen* 52, 210), and Struber (*Ibid.* 562). Special cell holders were designed for the Beckman spectrophotometer for applying the instrument for analysis of margarine or shortening (McGuine & Moss—*J. Am. Oil Chemists' Soc.* 27, 159). Potter & Kummerow (*Ibid.* 190) cautioned that presence of appreciable nonsaponifiable material in fats could cause appreciable error in the spectrophotometric analyses of the fats. The high unsaponifiable of liver lipides and the yellow pigment of chicken fat were mentioned as particular sources of error in analyses of the respective fats. The infrared spectrophotometric method was more rapid, specific, and accurate than the more common lead salt-alcohol method for the determination of *trans*-octadecenoic acids and esters (Swern *et al.*—*Ibid.* 17). Two papers contained procedures for the spectrophotometric microdetermination of polyunsaturated fat acids with use of alkali isomerization. The method of O'Connell & Daubert (*Arch. Biochem* 25, 444) was designed for determination of unsaturated

fatty acids in blood. In the micromethod of Berk *et al.* (*Anal. Chem.* 22, 718) the ethylene glycol-potassium hydroxide isomerization step was replaced with isomerization with aqueous alkali at high temperature and pressure to improve the absorption readings.

The configuration of the mixed glycerides occurring in cacao butter (Meara—*J. Chem. Soc.* 1949, 2154) and soybean oil (Dutton *et al.*—*J. Am. Oil Chemists' Soc.* 27, 25) were determined, and the deviations of the data from the fundamental concept of "even distribution" of the fatty acids among the glycerides were pointed out. The analyses of the milk fats of the cow and buffalo were determined in work which indicated that milk and depot fats of animals were produced by the same general mechanism (Achaya & Hilditch—*Proc. Roy. Soc., London*, B137, 187).

The periodic method for the determination of monoglycerides and free glycerol in fats was modified to eliminate the danger of secondary reactions due to elevated temperatures (Pohle & Mehlenbacher—*J. Am. Oil Chemists' Society* 27, 54). When chloroform was used as the solvent in the removal of free glycerol, hard fats were dissolved without applying heat. Kummerow & Daubert (*Ibid.* 100) considered this method for determination of monoglycerides nonspecific for they obtained monoglyceride values from saponified fats which did not contain monoglycerides.

The structures of several fatty acids were investigated. Gupta *et al.* (*J. Chem. Soc.* 1950, 3484) reported that the so-called "vaccenic acid" of body and milk fats contained *trans*-10- and *trans*-11-octadecenoic acids. Heretofore all fatty acids recorded in natural fats were invariably *cis*-isomers. In work on differentiating the C₁₈ monenoic acids to establish the structure of natural vaccenic acid, synthetic *trans*-11-octadecenoic acid and natural vaccenic acid gave essentially identical infrared spectra, but the X-ray patterns were distinctly different (Bumpus *et al.*—*J. Am. Chem. Soc.* 72, 2116). The synthetic acid and elaidic acid showed identical X-ray patterns, but by chemical evidence they were not the same substance. Pigulevskii & Naidenova (*Doklady Akad. Nauk S.S. S.R.* 72, 717) interpreted Raman spectra data on linoleic acid to indicate that only *cis*-isomers of the acids occur in nature. The unusual short chain diene acid of stillingia oil was identified as 2, 4-decadienoic acid (Crossley & Hilditch—*J. Chem. Soc.* 1949, 3353). Kaufmann & Keller (*Fette u. Seifen* 52, 389) reviewed the literature written concerning the tetraenoic C₁₈ acid, "parimar acid," in the seed fats of Balsaminaceae. A review of the molecular structure of fatty acids was prepared by Baltes (*Ibid.* 429). Several highly unsaturated fatty acids were isolated from bonito oil (Matsuda—*J. Soc. Chem. Ind. Japan* 45, suppl. 3, 4, 6, 8, 134) and codliver oil (Treibs & Schlegel—*Pharmazie* 5, 303), and their structures were determined.

Studies were made on the methods for the determination of vitamin A in oils. Sanford *et al.* (*Com. Fisheries Rev.* 12, No. 6, 4; No. 11A, 18) suggested that in the extraction of low-oil fish livers for the determination of vitamin A, high ratio of solvents were required for complete extraction, and the use of admixtures of pumice with the liver sample appeared to aid the extraction. The vitamin A committee of the Association of Official Agricultural Chemists' recommend that the physical-chemical method be adopted as standard (Wilke—*J. Assoc. Off. Agr. Chemists'* 33,

613). A micro-method for separating free and esterified vitamin A was based on absorbing the vitamin A alcohol on alumina from a petroleum ether solution of the sample (Eden—*Biochem. J.* 46, 259). The vitamin A potencies of the liver oils of sea cod, flounder (Sanford *et al.*—*Com. Fisheries Rev.* 12, No. 11A, 29), Indian sharks (Bose & Subrahmanyam—*Indian J. Med. Res.* 37, 1), and haddock (Mocoroa—*Anales fis. y quim., Madrid*, 40, 597), and the seasonal variations of the vitamin A of Victorian butterfat (Farrer—*Australian J. Sci. Research B2*, 355) were recorded.

Many other communications contained information on constituents of fats other than the fatty glycerides. The various fractions of shea-butter obtained with ethanol extraction and distillation were investigated in work connected with improving the product (Pansard—*Oleagineux* 5, 234). The presence of resinous alcohols, phytosterols, and certain hydrocarbons made it difficult to use. The cinnamic acid present was dissolved out with boiling water and recrystallized. The absence of enanthol and undecylenic acid in the distillation of the oil from rye ergot was attributed to the pyrolytic processes occurring during the distillation. A method for the determination of various lipides in biological material comprised complete extraction with an acetone-ethanol mixture and fractionation into phospholipides (precipitation with acetone saturated with calcium chloride), free cholesterol (separation with digitonin), and combined cholesterol (saponification followed by digitonin treatment) (Lorenzini—*Giorn. clin. med.* 30, 30). Data on the cholesterol, phytosterol, and tocopherol content of food products and animal tissues were compiled from the literature and tabulated (Lange—*J. Am. Oil Chemists' Soc.* 27, 414). Partial separation of cholesterol and ergosterol mixtures was done chromatographically (Kawahara & Dutton—*Ibid.* 161). Aqueous solutions of cholesterol were prepared using a non-ionic emulsifier (Neuzil *et al.*—*Compt. rend* 229, 600). The Villavecchia test was adapted for the determination of sesanol in sesame oil (Budowski *et al.*—*J. Am. Oil Chemists' Soc.* 27, 307). The tocopherol content of the edible oils sold in the markets in the city of Buenos Aires were recorded (Herraiz & Herrero—*Ann. N. Y. Acad. Sci.* 52, 306). A microdetermination of plasma phospholipides was based on precipitating them from lipide extracts with trichloroacetic acid (Zilversmit & Davis—*J. Lab. Clin. Med.* 35, 155). The phosphatides separated from soybean and corn were compared in regard to content of nitrogen, phosphorus, choline nitrogen, inositol and sugar (Scholfield *et al.*—*J. Am. Oil Chemists' Soc.* 27, 352). The literature was reviewed for the amount of phosphatides in dairy products (Kaufmann *et al.*—*Fette u. Seifen* 52, 600). A review on the various lipids present in the nervous system was prepared from 201 references (Brante—*Acta Physiol. Scand.* 18, Suppl. 63, 189, +26 pp.). A micromethod for detecting enzymatic breakdown of phospholipides was devised with the thought that a measurement of this type breakdown would be useful in food investigations (Rose—*Food Tech.* 4, 230).

Some of the hydrocarbon constituents of rice oil were isolated and their properties were recorded (Kato—*J. Nippon Oil Technology Soc.* 2, No. 4, 34). The phosphorus content of Canadian lards was generally less than 0.001% (Lips—*Can. J. Research* 28F, 21). Some sulfur compounds of the oil of crucifer

seeds were isolated and their properties were determined (Andre—*Ann. nutrition et aliment.* 3, 329; Andre & Delaveau—*Compt. rend.* 229, 555). It was suggested that the odor of some of these oils might be due in part to methyl mercaptans.

DETECTION OF ADULTERATION. Oleomargarine and butter were distinguished by the temperature at which they dissolve in a 2:1 mixture by volume of respectively ethanol and isoamyl alcohol (Felman & Lepper—*J. Assoc. Off. Agr. Chemists'* 33, 492). In this test the critical temperature of solution was 42-53°C. for butter and 66-75°C. for oleomargarine. In an investigation on substitution of vegetable fats for butterfat in ice cream, the Reichert-Meissl, Polenske, and saponification values were used in detecting the foreign fats (Martin *et al.*—*Ice Cream Trade J.* 45, No. 11, 48). In a lecture on the problem of adulteration of ice cream by substituting cheaper fats for the butter fat, Raines (*Assoc. Food & Drug Off. U. S. Quarterly Bull.* 14, 137) told of the analytical problems involved, the evidence necessary to obtain conviction, and of the need for improved regulations to protect the consumers.

The purity of cacao fat could be ascertained from the critical temperature of the start of its crystallization in aniline (van Voorst—*Chem. Weekblad* 46, 150).

In a lecture on Food and Drug Administration, Dunbar (*Assoc. Food & Drug Off. U. S.* 14, 98) described the new margarine laws, told of the use of squalene from shark liver oil to mask adulterated olive oil against detection, and of the organization of a "Food Law Institute" to give instructions and do research on food and drug laws. Fitelson (*Ibid.* 128) explained in more detail how the squalene by-product of fish liver oil vitamin concentration was being used to adjust the squalene content of adulterated olive oil so that adulteration could not be identified by laboratory tests. Anselmi and Macelli (*Rend. est super. sanità* 12, 358) suggested that the squalene test for adulteration of olive oil should be studied to make it more precise. The squalene test was used for characterization of the oils used for imbedding canned fish (Hadorn & Jungkunz—*Mitt. Lebensm Hyg.* 40, 202).

Chatterjee (*Indian Soap J.* 15, 253) discussed the adulteration of peanut oil with special reference to materials which make it hard to hydrogenate. The methods investigated for detection of peanut oil were concerned with the Bellier test (Kirsten—*J. Assoc. Off. Agr. Chemists'* 33, 747; Lacerda—*Rev. soc. brasil quim.* 17, 29) and the Lorentz-Lorenz formula (Nayar *et al.*—*Current Sci. India* 19, 54). The latter test was used in connection with adulteration of mustard oils with peanut oil.

A test for the detection of castor oil depended on the formation of 10,12-octadecadienoic acid after alkali isomerization (von Mikusch—*Australia Dept. Supply. Paint Notes* 5, No. 5, 125; *Farbe u. Lack* 55, 361).

The hexabromide value of the extracted fat was recommended for the detection of horse meat or fat in meat products or animal fats (Dallery—*Analyst* 75, 336; Hynds—*J. Assoc. Off. Agr. Chemists'* 33, 752).

When fatty oils were adulterated with at least 10% mineral oil the saponified sample yielded a milky solution on dilution with water (Mendritzki—*Süddeut. Apoth.-Ztg.* 89, 973).

The iodine value was not useful for characterization of lanolin because results were variable (Gänssle—*Fette u. Seifen* 52, 164). The thiocyanogen value was more reasonably constant.

Detergents

MANUFACTURE. Most of the work on improving soap stock dealt with the rosin-containing material. When rosin was heated with alkali and sulfur or sulfur-containing material, yellowing of the soap made therefrom was reduced (Hampton—*U. S.* 2,497,882). The tall oil from sulfate pulp processes was recovered as the soap and spray dried to reduce odor (Young & Calleton—*U. S.* 2,507,237). Such soap was useful in flotation processes or for cleaning metals. Tall oil soap when diluted with water would precipitate colored bodies and some sodium lignocerate to yield a solution of a better soap product (Hasselstrom—*U. S.* 2,519,903). Better removal of extraction hydrocarbons by distillation was obtained from rosin soap when 10-45% of the soap was converted to the free rosin acids by reaction with mineral acids (Gayer—*U. S.* 2,492,038).

A patented method of bleaching soapstock comprised treating with phosphoric acid or acid phosphates and a compound liberating nascent oxygen (Henderson & Libby—*U. S.* 2,483,414). Many soaps were prepared from soap stocks containing fatty acids in various states of oxidation, and the technic of manufacture, and properties of the products were recorded to serve as basic data for utilizing oxidized acids as soap stock (Allard & Duffaud—*Bull. mens. ITERG* 3, 318; Duffaud—*Ibid.* 4, 251).

Holmberg (*Acta Polytech., Chem. Met. Ser. 1*, No. 10, 23 pp.; No. 11, 61 pp.) reviewed the literature on soapmaking using alkyl esters as intermediates and developed cost information from the experimental tests. The process permitted reproduction and maintenance of a desired fatty acid composition. It was less costly for medium size soap plants than conventional processes. A patent issued to Holmberg (*U. S.* 2,494,127) on this type of process pertained to design of apparatus so that released alcohol distilled off, and the soap solution was directly millable for toilet soap production.

Flaherty (*U. S.* 2,511,575) prepared rosin soap by saponification with sodium carbonate under pressure, the released carbon dioxide being used to maintain the pressure and finally to force the soap through the nozzle of a spray drier. Such a German process was briefly described (Fiala—*Seifen-Öle-Fette-Wachse* 74, 78). The velocity of saponification in soap making processes was accelerated by additions of aromatic hydroxy compounds or low molecular weight alcohols or aldehydes (Rashkovan & Lebedeva—*Zhur. Obshchei Khim.* 20, 253, 258, 261). The presence of 10% 1-hexadecanol and one percent zinc oxide promoted cold saponification by an emulsifying action (Kawakami—*Japan* 174,796).

The innovations in continuous soap manufacture pertained to improvements in the processes developed in the past. Jones (*U. S.* 2,499,388-9) patented controls for proportioning fat and alkali into the system. Owen (*U. S.* 2,525,936) separated nigre and neat soap from molten fitted soap in a continuous process without the use of centrifuges by flowing the soap in a shallow stream and withdrawing the upper (neat)

and the lower (nigre) phases. In a continuous system for manufacturing soap from sperm oil, the apparatus was designed to take advantage of the exothermic heat for heating a new charge (Schmidt & Edwards—*U. S.* 2,492,940). Continuously made soap was freed of glycerol and impurities by continuously washing at 100° with brine solution at a concentration close to the critical concentration below which neat soap was dissolved in the solution (L'Oreal—*Brit.* 636,945). In another patent, washing with weak brine solution was followed by washing with hot water (Societe savons Francois—*Fr.* 941,884). In a continuous system, the glycerol-water mixture was distilled with certain hydrocarbons that were used in the process (Bradford—*U. S.* 2,496,576).

The glycerol distribution in the soap layers during full plant scale operations was determined by Govan (*J. Am. Oil Chemists' Soc.* 27, 44). The glycerol in the curd and lye layers approached a ratio, as the electrolytic strength increased, in which glycerol concentrations in the water of the lye layer was 1.3 times that of the water in the curd layer. The conditions for the most favorable distribution ratio were vigorous boiling on a hard grained curd for a sufficient length of time to assure thorough mixing. Other reports pertaining to glycerol recovery dealt with purifying the glycerol. The purification steps reported were treatment with nascent hydrogen to reduce aldehyde and ketones (Halbedel—*U. S.* 2,505,735), hydrogenation for the same purpose (Vandoni & Lavacchieli—*Ital.* 434,146), ion exchange purification to eliminate distillation (Kahler—*Chem. Eng.* 57, No. 7, 109; Am. Cyanamid Co.—*Brit.* 633,343), and a final distillation step to remove volatile acids (Bhat-tacharya—*J. Proc. Inst. Chemists, India*, 19, 117).

A small amount of information appeared on the non-soap ingredients of soaps. Schindler (*Ital.* 433,392) replaced some of the soap fatty acids with humic acids. Mersolate was considered a better soap ingredient than saponin (Lindner—*Seifensieder-Ztg.* 73, 61). Substitution of glycols for glycerol in toilet soaps was not recommended, because the former are skin irritants (Schneider—*Fette u. Seifen* 52, 423). However, Hussing *et al.* (*Ibid.* 45) recorded test data indicating that 1,3-butylene glycol is not irritating to the skin. The newly patented germicidal ingredients for soaps were 2,2'-dihydroxy halogenated diphenylmethane (Kunz & Gump—*U. S.* 2,535,077), and silver phosphate salts (Mendenhall—*U. S.* 2,510,510). The new optical bleach ingredients for textile soaps were benzimidazole derivatives (Ciba, Ltd.—*Swiss* 235,570, 263,627, 263,490-4). One detergent contained an indicator dye for the purpose of indicating solutions of optimum effectiveness by the tint (Parker—*U. S.* 2,502,881).

The new apparatus for making floating soaps were plodders containing means to incorporate air in the soap being plodded (Marshall—*U. S.* 2,494,891, Schulerud—*U. S.* 2,524,999, 2,525,081). High moisture soap rods were conditioned for pressing into bars with infrared rays and blasting the surface with a strong current of air (Heald—*U. S.* 2,527,062). Soap bars containing decorations were made by laminating inserts into the bar in a continuous process, in which one of each pair of laminated surfaces was moistened to increase adherence (Marshall—*U. S.* 2,528,531). Marks of contrasting color were incorporated in soap bars by combining two soap-extruding machines in

such manner that the letters, symbols, monographs, etc., extended throughout the bar (Hoglin — *U. S. 2,495,005*).

Most of the literature on synthetic detergent manufacture was on newly patented products or manufacturing technics. For convenience of presentation these are listed under the assignee, or the patentee where no assignee was given:

ACME Azienda Chim. Meridionale S.a.R.L.

Sodium proteinate from hair waste (*Ital. 437,788*).

Alix, J.

Sodium α -isopropyl- β -naphthalene-sulfonate (*Fr. 940,070*).

Allied Chem. & Dye Corp.

Alkyl aromatic sulfonate (*U. S. 2,506,417*). Organic nitrosation-sulfitation products (*U. S. 2,510,466*).

Am. Cyanamid Co.

Alkenoxyacetylcarbonylguanidine (*U. S. 2,478,859*). Epichlorohydrin-tetraethylene-pentamine polymer reacted with stearic acid (*U. S. 2,479,480*). Certain pyridine and quinoline derivatives (*Brit. 636,353*).

Arkansas Co., Inc.

Fatty acid-aliphatic amine polyglycol combinations (*U. S. 2,525,771*).

Armour & Co.

1-alkyl-2, 5-dimethylpyrrolidines (*U. S. 2,497,839*).

Bersworth, F. C.

Sodium salts of protein hydrolyzates (*U. S. 2,500,019*).

Busch, G. L.

Neutralizing sulfonated detergents (*U. S. 2,511,043*).

California Research Corp.

Alkyl aryl sulfonates (*U. S. 2,500,024, 2,531,166*).

Chwala, A.

Halogenated glucosides combined with proteins, fatty acids, amines, etc. (*Brit. 625,644*).

Ciba, Ltd.

Fatty acid amides, urethanes, ureas, reacted with bisulfite and aldehyde containing a sulfate group (*Brit. 630,492, U. S. 2,494,966*). N-substituted amino acetals (*Brit. 625,840*). Quaternary pyridinium chloride derivatives (*Swiss 233,347*). Aromatic amino disulfonic acid derivatives (*Swiss 235,195-9, 235,201*). Derivatives of *p*-hydrozene-benzenesulfonic acid (*Swiss 235,200*). Condensation product of fatty acids, an amine and formic acid (*Swiss 236,995*). Reaction product of N-(hydroxymethyl)-amides of fatty acids, phenol, acetic acids and formaldehyde (*Swiss 242,605*). Condensation product of N, N'-bis(chloromethyl)-N, N'-distearoylmethylenediamine and isethionic acid (*Swiss 253,164*). N bis(2,3 dihydroxypropyl) fatty acid amides (*Swiss 255,311-12*). Reaction of stearamide formaldehyde and certain amines (*Swiss 260,278*). Peat or lignin treated with caustic (*Swiss 261,542*). Condensate fatty acids, ethylene oxide and certain amides (*Swiss 263,839-40*).

Clahsen, F. A.

Salting out sulfonated compounds with sulfite solutions (*Dutch 65,460*).

Colgate-Palmolive-Peet Co.

Ammonia sulfamate used to stabilize sulfonates. (*U. S. 2,493,444-5*). Condensation products of olefins with organic acids having α,β -enal group (*U. S. 2,496,358*).

Commercial Solvents Corp.

Fatty acid heated with N-substituted 1, 2 alkane diamines (*U. S. 2,520,102*).

Compagnie produit chim. et electro-metall. Alais, Froges et Camargue.

Reaction products of paraffin or naphthalene sulfonyl chloride and pyridine (*Fr. 940,088*).

Dow Chemical Co.

Alkali salts of sulfosuccinic acid diester of propylene glycol phenyl ethers (*U. S. 2,510,008*).

E. I. DuPont de Nemours & Co.

Alkanesilonic acid esters (*U. S. 2,476,307*). Polyvinylpyridinium alkyl halides (*U. S. 2,487,829*). Copolymerized product of vinyl lactam and ester of fatty acid (*U. S. 2,497,705*). Sulfonation with sulfur dioxide and oxygen with use of azo compounds as catalyst (*U. S. 2,503,253, 2,503,280*).

Fabrizi, F.

Ammonium dodecyl sulfate (*Ital. 442,008*).

J. R. Geigy A.-G.

Sulfonic acid formaldehyde condensed with aromatic hydrocarbons or acids (*U. S. 2,482,942*). Acylbiguanides (*Swiss 232,285, 232,322, 233,345-6*). Organic sulfonic acids treated with quaternary ammonium compounds (*Swiss 255,303-4*).

General Aniline & Film Corp.

Purification of polyethylene oxide ether of diisobutyl phenol (*U. S. 2,496,582*).

Harvel Corp.

Simultaneous condensation and sulfonation of phenols and alkyl ethers of phenols (*Brit. 627,920*).

Heitzmann, P. J.

Fatty acid salts of albuminous material (*Fr. 936,632*).

Kyoritsu Marine Ind. Co.

Oleyl alcohol, paraformaldehyde and hydrochloric acid are condensed and boiled with potassium ethylate (*Japan 154,821*).

Marriott, R. H.

Solution of fatty alcohol sulfates and salt (*Brit. 590,839*).

Monsanto Chem. Co.

Reaction product of ethylene oxide and 2-mercaptobenzothiazole (*U. S. 2,498,617*). Product of ethylene oxide and 5-ethyl nonanol-2 (*U. S. 2,508,035*). Product of ethylene oxide and 2-n-propyl-heptanol (*U. S. 2,508,036*).

J. B. Niederl & Associates, Inc.

Nicotinium alkyl sulfates (*U. S. 2,507,925*).

N. V. Bataafse Petrol. Maatschappij

Use of ammonium salts to inhibit gelation of synthetic detergents (*Dutch 64,014*). Mahogany soap is extracted with polyhydric alcohol and a hydrocarbon (*Dutch 65,718*).

Nostrip, Inc.

Amido-amino-amine soap of fatty acids (*U. S. 2,514,954*).

Onyx Oil & Chem. Co.

Aromatic diquaternary ammonium compounds (*U. S. 2,520,275*).

Petrolite Corp., Ltd.

Polymerized derivatives of pyridine containing fatty and dibasic acid radicals (*U. S. 2,505,948*).

Polasek, J.

Reaction product of urea and sulfonated cyclic hydrocarbons (*Fr. 940,117*).

Procter & Gamble Co.

Process of making monoglyceride sulfonate detergents (*U. S. 2,529,537-9*).

Reilly Tar & Chem. Corp.

Pyridineethanesulfonic acids (*U. S. 2,508,904*).

Rohm & Haas Co.

Quaternary ammonium derivatives of phenyl ethers (*U. S. 2,499,213*). Polyethylene glycol ethers of bis (dialkylhydroxyphenyl) methane (*U. S. 2,504,064*).

Sandoz, Ltd.

Reaction products of fatty acids and β, β' -disubstituted diethyl amine and certain organic sulfates (*Swiss 261,540-1*).

Severoceske Tukove Zavody

Hydrolysis of alkanesulfonyl chlorides (*Brit. 636,650*).

Sharples Chemicals, Inc.

Glycol thioethers (*U. S. 2,494,610*).

Sinclair Refining Co.

Reaction product of chloroacetylated aromatic compounds with sodium sulfate (*U. S. 2,499,997*).

Societe produits chim. Ariege a Lavelanet

Mixture of cyclic sulfonate products and polymers of ethylene oxide (*Ital. 439,173*).

Sonneborn Sons, Inc.

Mahogany soaps are made heat stable by additions of inorganic derivatives of hyposulfurous or sulfoxylic acid (*U. S. 2,486,373*).

Sorge, J.

Paste made by precipitating aluminum hydroxide with hydroxy alkyl amine (*Swiss 266,633*).

Standard Oil Co. of Ohio

Sulfonated thio ethers of polymerized olefines (*U. S. 2,499,377*).

Sugiyama, S.

Spermaceti sulfonate (*Japan 155,985*).

Sun Chem. Corp.

Sulfonated oleonitrile (*U. S. 2,495,105*).

Universal Oil Products Co.

Sulfonated straight-chain alkyl aromatic hydrocarbons (*U. S. 2,479,120*). Diels-Alder condensation products of certain polyolefinic cyclic hydrocarbon fractions (*U. S. 2,514,533*). Polycyclicalkyl arylsulfonic acids (*U. S. 2,524,086*).

Usines de Melle

Higher alcohols sulfonated in inert solvent and neutralized with inorganic alkali ethanol amine (*Fr. 942,075*).

Wyandotte Chemicals Corp.

Mixture of alkyl aryl sulfonates and inorganic salts (*U. S. 2,515,577*).

Deodorized sulfonates were prepared from marine animal oils by polymerizing the oil before sulfonation (Ueno—*J. Nippon Oil Technol. Soc.* 3, No. 1/2, 101). Condensation products of amines such as alanine, serine, and leucine with fatty acids were good detergents (Naudet—*Bull. soc. chim., France* 1950, 358). A method for precipitating unsaponifiable material from mersolate detergent comprised dilution with

water at 90°C. to a 20% concentration at a pH of 7.0 (Ehrbacher & Neu—*Fette u. Seifen* 52, 170).

Several detergent mixtures were patented for special purposes. A detergent for automatic washing machines contained condensation products of tertiary fatty mercaptans with ethylene oxide and a mixture of inorganic salts (Harris—*U. S. 2,522,446-7*). A soap with reduced tendency to form lime soap curd contained sulfonated organic products, an ester of allyl alcohol, and soap (Preston—*U. S. 2,494,580*). Another washing composition contained soap and 1-5% of an alkylene polyamine derivative (Bersworth—*U. S. 2,524,218*). Soap and synthetic detergent were also combined in a bar form (Marshall—*U. S. 2,508,578*). The addition of tylose to synthetic toilet detergent was recommended to inhibit skin irritation (Kuchinka—*Seifen-Öle-Fette-Wachse* 76, 9). A shampoo contained sulfated fatty alcohols and soap of polymerized rosin (Dobbelman, N. V.—*Dutch* 66, 298). Special mixtures of soap and a synthetic detergent were prepared for removal of spray residues from fruit (Larsen & Bresse—*U. S. 2,493,460*).

The detergents for washing household utensils were mixtures of inorganic salts containing small amounts of organic detergent material (duBois—*U. S. 2,519,747*; Little—*U. S. 2,509,440*; Alix—*Fr. 940,069*). New scouring compounds contained novel mixtures of abrasives, inorganic salts and organic detergents (Pape & Santmyers—*U. S. 2,497,057*; Jones—*Brit. 590,264*; Hashimoto—*Japan* 174,118; Munter—*U. S. 2,494,827*; Lietar—*Belg.* 482,363). A paste soap was a mixture of glycerol and sodium silicate (Kobayashi—*Japan* 175,434).

Many communications on detergents provided information on common practices in the industry, described raw material, or known products, or gave other general information. These are most conveniently entered here by tabulation under the subjects treated:

General Papers: Reviews of the detergent field, Chakraborty—*Indian Soap J.* 15, 26; German trade name detergents, Hetzer—*Seifen-Öle-Fette-Wachse* 75, 121; Indian soap industry, Ghosh—*Indian Soap J.* 15, 3; Wahi—*Ibid.* 165; product liability of the soap manufacturer, Gray—*Soap Sanit. Chemicals* 26, No. 11, 37.

Raw Materials: Products for manufacture of synthetic detergents, Widaly—*Seifen-Öle-Fette-Wachse* 76, 157; methyl esters of fatty acids for soap making, Schlenker—*Ibid.* 21; coconut oil for soap making, Iyer—*Soap, India*, 1, No. 3, 11.

Manufacture: Saponification methods, Manneck—*Seifen-Öle-Fette-Wachse* 75, 111; Paleni—*Olearia* 3, 245; cold process soap making, Rao & Ghose—*Indian Soap J.* 15, 188; Ruemele—*Seifen-Öle-Fette-Wachse* 76, 101; continuous saponification, Heinrich—*Ibid.* 295; problems in soap manufacture, Kelber—*Ibid.* 75, 495; mistakes in toilet soap manufacture, Zilske—*Ibid.* 173; limiting concentration of electrolytes in soap solutions, Löffl—*Ibid.* 471; soap milling problems, Reich—*Ibid.* 76, 303; cooling press for soap, Stein—*Ibid.* 75, 388; cooling and drying toilet soap, Zilske—*Ibid.* 76, 89; the problem of formation of colored spots in soap, Heller—*Ibid.* 75, 504; waste disposal in the soap industry, Wittman—*Ibid.* 176.

Special Products Manufacture: Toilet soaps, Vallance—*Soap Sanit. Chemicals* 26, No. 9, 32; No. 10, 47; No. 11, 46; Hempel—*Seifen-Öle-Fette-Wachse* 75, 261; floating soaps, Lesser—*Soap Sanit. Chemicals* 26, No. 3, 41; liquid soaps, Rao—*Indian Soap J.* 15, 247; transparent soaps, Saboor & Dutta—*Ibid.* 83; powdered shampoos, Bergwein—*Seifen-Öle-Fette-Wachse* 75, 373; scrubbing soaps, Trusler—*Soap Sanit. Chemicals* 26, No. 5, 44; hard water bar soap, Lesser—*Ibid.* No. 6, 42; germicidal liquid soap, Bowers—*Ibid.* No. 8, 36; Chase—*Am. Perfumer* 55, 395.

Miscellaneous Ingredients: Perfumery for soap, Schmidt—*Fette u. Seifen* 52, 109; Treffler—*Soap Sanit. Chemicals* 26, No. 8, 33; anon.—*Ibid. Blue Book*, 175; optical bleaches, Stearns *et al.*—*Soap Sanit. Chemicals* 26, No. 4, 48; cellulose derivatives for soap, Lesser—*Ibid.* No. 8, 29. Widaly—*Seifen-Öle-Fette-Wachse* 75, 283; Pollok—*Soap, Perfumery & Cosmetics* 22, 1332; 23, 811; starch as a soap filler, Smith—*Am. Perfumer Essential Oil Rev.* 56, 51; fat-free detergent material, Uhl—*Seifen-Öle-Fette-Wachse* 76, 155; inorganic and organic fillers, Augustin—*Ibid.* 75, 408; phosphates, Trexler—*Soap, Sanit. Chemicals* 26, No. 7, 39; fillers, Shukla—*Indian Soap J.* 15, 149; silicates for soap; Rao & Ghose—*Ibid.* 128; anhydrous phosphates, Lindner—*Seifen-Öle-Fette-Wachse* 76, 133.

Synthetic Detergents: Future possibilities, Fox—*Soap Sanit. Chemicals* 26, No. 6, 37; review Zachert—*J. Southern Research* 1, No. 2, 9; from petroleum, Simcox—*Chemistry & Industry* 1950, 178; German products, Hagge—*Fette u. Seifen* 52, 165; in United Kingdom, Vallance—*Soap Sanit. Chemicals* 26, No. 5, 37; cationic soaps, Palit—*Indian Soap J.* 15, 181; comparisons of soaps versus synthetics, Snell—*Am. Perfumer Essent. Oil Rev.* 55, 221; Lesser—*Soap Sanit. Chemicals* 26, No. 9, 36; McCutcheon—*Ibid.* No. 2, 44; Tum—*Seifen-Öle-Fette-Wachse* 76, 159; Belgundi—*Indian Textile J.* 59, 1081; Seaman—*Soap Perfumery & Cosmetics* 22, 1218; oxyethylene alkylphenol detergents, Linder—*Fette u. Seifen* 52, 613; protein fatty acid condensation products, Ruff—*Ibid.* 352; sulfonation of alkyl aryls, Kircher—*Soap Sanit. Chemicals* 26, No. 12, 48; tetra sodium salt of ethylene diamine tetra acetic acid, Singer & Bersworth—*Ibid.* No. 3, 45; sulfonated oils, Fu & Chen—*J. Chem. Eng. China* 15, 72; Igepon detergents, Kastens & Ayo—*Ind. Eng. Chem.* 42, 1626; classification of detergents, Harper—*Pharm. J.* 164, 265; Schöller—*Textil-Rundschau* 5, 77; Gonzalez—*Industria y quimica* 11, 55.

Mixed Detergents: Soap with fatty alkylolamides, Sussman & Bernstein—*Soap Sanit. Chemicals* 26, No. 4, 37; soap with alkylaryl sulfonates, Flett *et al.*—*Am. Perfumer Essential Oil Rev.* 56, 231; *J. Am. Oil Chemists' Soc.* 27, 304; soap with miscellaneous synthetics, Borghetty & Bergman—*Ibid.* 88.

Properties: Physics and chemistry of detergency, Kling—*Angew. Chem.* 62, 305; detergency, Lindner—*Melliand Textilber.* 29, 203; solubilization by detergents, Klevens—*Chem. Revs.* 47, 1; structure of aqueous solutions of soap, Berry & Bean—*J. Pharm. Pharmacol.* 2, 473; physical

properties, Merrill—*J. Chem. Ed.* 27, 312; soap solutions, Harkins—*Scientific Monthly* 70, 220; Waddams—*Soap, Perfumery & Cosmetics* 23, 915; Colloidal properties, McBain—*Frontiers in Chemistry, colloid*, 8, 113; crystals and micelles of soaps, McBain—*Ibid.* 133; constitution and action, Chwala—*Colloid Chem.* 7, 517; chemical structure and performance, Roebuck—*Ibid.* 529; effect on the skin, Szakall—*Fette u. Seifen* 52, 171.

Uses: Efficient dishwashing, Wilson & Podas—*Modern Sanitation* 2, No. 5, 32; household scouring cleansers, Lesser—*Soap Sanit. Chemicals* 26, No. 5, 40; bottle washing, Ross—*Chem. Inds.* 67, 902; cleaners for removal of oils, Holloway—*Finish* 7, No. 1, 53; skin cleansers, Brechner—*Safety Eng.* 99, No. 6, 15; detergents for the food industry, Sisley—*Rev. fermentations et inds. aliment.* 5, 83; dry cleaning detergents, Morgan—*Soap Sanit. Chemicals* 26, No. 7, 29; linoleum and paint cleaners, Harris *et al.*—*Ibid.* 32; metal cleaners, Tarring—*J. Electrodepositors' Tech. Soc.* 26, No. 13, 8 pp.; detergents for printers' type, Smith—*Soap Sanit. Chemicals* 26, No. 12, 44; detergents for mechanical dishwashing, Tyler—*Ibid.* 37.

Reviews on Methods of Analysis for Soaps: Fatty acid determination, Weber—*Seifen-Öle-Fette-Wachse* 75, 197; orthophosphoric acid determination, Neu—*Fette u. Seifen* 52, 298; control analyses, Schlüter—*Seifen-Öle-Fette-Wachse* 76, 43; testing fat-free detergents, Uhl—*Ibid.* 137; mixed detergents analysis, Hintermaier—*Fette u. Seifen* 52, 689; end-use testing of scrub soaps, du Bois—*Soap Sanit. Chemical Blue Book* 1949, 167; rug cleaner testing, Leonard—*Soap Sanit. Chemicals* 26, No. 1, 42; German standard methods for glycerin analysis, Kaufmann & Neu—*Fette u. Seifen* 52, 682.

CONSTITUENT ANALYSES OF DETERGENTS. In comparing moisture determination of detergents by the air oven, vacuum oven, and Karl Fischer titration, the latter two methods gave good results which agreed very closely (Draper & Milligan—*Texas J. Sci.* 2, 209). Air oven methods of drying at temperatures from 100 to 150° did not give consistent results.

Voituret (*Seifen-Öle-Fette-Wachse* 76, 94) in investigating methods for determination of fatty acids in soap found those in which the fatty acids were released and the volume measured as accurate as more common tedious procedures. A procedure issued by Weber (*Ibid.* 93) comprised releasing fatty acids with inorganic acids, filtering on a moist fluted filter, on which the fatty acids remain and can be washed. After washing, 10 cc. of ethanol was added and the fat was washed through with ether, the solvent distilled, and fat weighed. A similar method was described by Fuchs (*Fette u. Seifen* 52, 23).

The volumetric evolution method for determining carbonates was adapted for analysis of synthetic detergents by using alcohol instead of water solutions of the sample to eliminate foaming (Schuck & Koester—*J. Am. Oil Chem. Soc.* 27, 321). Neu (*Seifen-Öle-Fette-Wachse* 75, 215) recommended the volumetric evolution method and apparatus as used on baking powders for the determination of carbonates in washing powders.

A colorimetric method for determining water-soluble silicates in detergents was based on measuring the color produced after development of silico-molybdic acid (Miller—*J. Am. Oil Chem. Soc.* 27, 348). Orthophosphoric acid was volumetrically determined in cleaning agents by reaction with standard arsenate solution (Neu—*Fette u. Seifen* 52, 298). This author (*Ibid.* 349) also suggested the use of ion exchange technic for removal of alkali cations from synthetic laundering detergents in procedures for their analysis. The most common methods for determining borax in soap could be improved in accuracy by removal of soap, silicates, phosphates, and carbonates by precipitation with strontium chloride and the use of methyl purple instead of methyl red indicator for adjustment of acidity prior to the standard alkali titration (Bernstein & Haftel—*J. Am. Oil Chemists' Soc.* 27, 45). The procedure was not applicable to synthetic detergents which were not precipitated by strontium salts.

Tylose, a commercial methyl cellulose ether, could be qualitatively detected in soap by a red-violet coloration that occurs on contact with concentrated sulfuric acid (Weber—*Fette u. Seifen* 52, 477). One procedure for estimating cellulose ethers and cellulose glycollic acid in soaps was based on hydrolysis and measurement of the reduction of Fehling's solution (Neu—*Seifen-Öle-Fette-Wachse* 76, 65). A method of distinguishing cellulose ethers from cellulose glycolates depended on certain quaternary ammonium compounds precipitating the glycolates (Neu—*Fette u. Seifen* 52, 23). Glycerol was determined in soap products by isolation as a glycerol-copper complex and iodometric determination (Bore—*Bull. mens. ITERG* 4, 168). The American Oil Chemists' Society Glycerine Analysis Committee recommended small modifications in their standard periodate method for determining glycerine (*J. Am. Oil Chemists' Soc.* 27, 412). A method for safrrole in soap comprised steam distillation and ultraviolet absorption measurements on the nonaqueous distillate (Ishler *et al.*—*Anal. Chem.* 22, 458).

Analytical procedures of various design were proposed for synthetic detergents. Bergeron *et al.* (*Bull. mens. ITERG* 4, 118) classified anion-active detergents into three groups depending on the color reactions that they gave with certain acids. The classification was to serve as a basis for qualitative analysis. Neu (*Fette u. Seifen* 52, 349) and Reutenauer (*Bull. mens. ITERG* 4, 61) proposed chromatographic methods to characterize the organic detergents in washing agents. Parisot (*Olearia* 3, 13) suggested analytical schemes depending on selective solubility in water, brine, alcohol, and certain organic solvents. Wurzschmitt (*Chem.-Ztg.* 74, 16) pointed out that analyses for carbon, hydrogen, oxygen, nitrogen, sulfur, halogen, and alkali on the dry detergent together with some simple qualitative tests for groups should permit setting up a balance sheet of the composition. Micaelli & Desnuelle (*Oleagineux* 4, 720) demonstrated the differences in ease of hydrolysis of sulfate, amide, and alkali linkages in detergents that could be useful in analytical schemes. Some sulfonated or sulfated detergents with the dye rosaniline hydrochloride (Karush & Sonenberg—*Anal. Chem.* 22, 175), and with basic fuchsin (Wallin—*Ibid.* 616) gave colored complexes which could be extracted and read in a colorimeter to estimate the amount of such detergents in solutions. A publication on analysis of

sulfonated alkyl aryls contained methods for determination of moisture, unsulfonated hydrocarbons, and sulfonated matter (Reutenauer—*Bull. mens. ITERG* 4, 197).

PHYSICAL PROPERTIES. The surface tensions of solutions of many commercial detergents were estimated from the size of the meniscus formed in test tubes (Scholberg—*J. Phys. & Colloid Chem.* 54, 107). When this surface tension was plotted against concentration, a discontinuity occurred which appeared related to the beginning of micelle formation. Lindner (*Melliand Textilber.* 29, 203) recorded the surface and interfacial (paraffin oil-water) tensions of aqueous solutions of 27 detergents and discussed the data in relation to washing characteristics. Kaminski & McBain (*Proc. Roy. Soc., London A198*, 447) observed that when liquid hydrocarbons were placed quietly upon solutions of certain detergents, spontaneous and in some cases violent emulsification may occur. This phenomenon was attributed to energy released as energies of adsorption and solubilization, and to the turbulence caused by the temporary inequalities of interfacial tension. A measure of this phenomenon was proposed by Bartholome & Schäfer (*Melliand Textilber.* 31, 487) as a useful characteristic of wetting agents. They found that a measure of the time required for a wetting agent to diffuse from the interior of a solution to the surface yielded a theoretical formula which was in good agreement with practical measurements of surface tension and sinking time of fabric squares.

Apparatus was designed to characterize detergent solutions according to the time required for films to drain from a standard surface (Miles *et al.*—*J. Am. Oil Chemists' Soc.* 27, 268). This film drainage rate appeared to be a function of the detergent and the added material. Pure detergent solutions gave fast-drainage rates; this rate was slowed by certain polar organic additives whose polar groups were at the end of a carbon chain, whereas highly branched chain, cyclic structure, and cis-forms of unsaturated organic additives did not slow drainage rates. Ternovskaya & Belopol'skii (*Zhur. Fiz. Khim.* 24, 43) confirmed theoretical concepts with experimental data regarding the nonparallelism of surface tension of detergent solutions and absorption of gases.

Burek (*J. Colloid Sci.* 5, 421) studied the foaming of detergent solutions as it may be related to the rate of surface tension lowering, concentration, and viscosity. The conditions for high foam stability were low surface tension relative to that of the pure solvent, moderate rate of surface tension lowering, and a high bulk or surface viscosity. A new foam test devised by Kaufmann & Budwig (*Fette u. Seifen* 52, 555) comprised placing 0.2 mg. of the material on a filter paper impregnated with copper acetate and adding one drop of a mixture of concentrated ammonium hydroxide and 30% hydrogen peroxide. With fatty acids stable foam production began with C₁₀ acids and was especially good with oleic acid. Simple modifications were proposed for shaking and for gas methods of studying foam formation in detergent solutions (Sinsheimer—*Soap Sanit. Chemicals* 26, No. 8, 38; Gotte—*Melliand Textilber.* 29, 65). Reutenauer & Sicard (*Bull. mens. ITERG* 4, 99) determined the foam formation characteristics of many commercial products recording the minimum concentration at which foam was formed, and foam volume as related

to the concentration, Kraft point of detergent, and surface tension.

A conductometric method for estimating solubility of quaternary ammonium salts of fatty acids was devised and the critical concentrations of several were recorded (Eggenberger *et al.*—*J. Am. Chem. Soc.* 72, 4135). Some evidence was also presented to indicate that fatty acid methyl morpholinium salts are not compatible with soap. Rao & Palit (*J. Indian Chem. Soc.* 26, 577) continued to demonstrate that soaps were more soluble in mixtures of polar and nonpolar solvents than in single solvents, and have recorded data on solubilities of soaps in lower alcohols or glycols mixed with certain chlorinated hydrocarbons. In comparisons of solubility curves, the soaps in hydroxyl organic solvent gave ideal curves whereas with hydrocarbons there was a high degree of nonideality of the solutions (Bondi—*J. Colloid Sci.* 5, 458). In this work sodium stearate dissolved or disintegrated but did not swell in hydroxyl solvents at temperatures above 80°C., swelled slightly in aliphatic hydrocarbons, and swelled considerably in aromatic solvents. In solubility data of dodecylammonium chloride and its methyl derivative, the former existed in two enantiotropic forms in hexane, benzene, and ethanol whereas the secondary, tertiary, and quaternary salts were monotropic in ethanol (Broome & Harwood—*J. Am. Chem. Soc.* 72, 3257).

Several aspects of micelle formation in detergent solutions were investigated. Lange (*Kolloid-Z.* 117, 48) found that application of the law of mass action to micelle formation agreed with experimental data. Corrin & Harkins had previously suggested that equations for this relationship were empirical. Exner (*Naturwissenschaften* 35, 344) made use of increased conductance of solutions, the so-called "Wien effect," in a method for the determination of the critical concentration for micelle formation. For the same purpose Ambler (*Proc. Intern. Congr. Pure & Applied Chem.* 11, 311) applied Tiselius' electrophoretic technique. He identified those products formed by condensation of naphthalene sulfonic acids with formaldehyde which form micellar species.

X-ray pattern studies of micelles were reviewed, new data were included, and the information was discussed in regard to the concept that solubilization occurs in the form of layers of micelles (Davies & Philippoff—*Nature* 164, 1087; Philippoff—*J. Colloid Sci.* 5, 169). In X-ray observations on addition of water to aerosol OT it was shown that water appears to enter the expanded lamellar lattice (Philippoff & McBain—*Nature* 164, 885).

Klevens (*J. Phys. & Colloid Chem.* 54, 1012; *J. Am. Chem. Soc.* 72, 3581, 3780) observed the effects of polar and nonpolar organic compounds on the critical micelle concentrations of detergent solutions. He interpreted the data to indicate the presence of two loci of solubilization in the micelles; one was in the hydrocarbon center of the micelle and the other in the palisade region occupied by the soap molecules. The effects of inorganic electrolytes on these types of solubilizations were discussed. Hattiangdi (*Indian Acad. Sci.* 30A, 320) determined the solubility curves of several sodium soaps in pinene, and interpreted a kink in each curve as indicating micelle formation below those respective temperatures and solution formation above the temperature. In van der Waarden's (*J. Colloid Sci.* 5, 448) investigations, volume of mi-

celles was calculated from the viscosity of sulfonates in hydrocarbon solutions. Volume increased with concentration to some critical concentration and then decreased. Micelle volumes were determined for different cations in the sulfonate. These results were said to agree with the supposition that the sulfonates were present in hydrocarbons as platelike micelles consisting of a sulfonate double layer with the polar groups at the inside. In work by Herzfeld *et al.* (*J. Phys. and Colloid Chem.* 54, 271) the critical micelle concentration of dodecylammonium chloride in presence of alcohols was a linear function of the chain length of the alcohols of 3 to 10 carbon atoms and the effect of salts on the systems was additive.

Granath (*Acta Chem. Scand.* 4, 103), Hoyer & Mysels (*J. Phys. and Colloid Chem.* 54, 966, and Nichols & Kindt (*Anal. Chem.* 22, 785) indicated how dynamic and kinetic data about the micelles formed was obtainable by a technique in which the micelles were "tagged" with minute amounts of solubilized dye material. Various data on micelle formation were included in the reports.

Micelle structure and stability of soap emulsions were also studied by determining the effect of inorganic electrolytes and polar and nonpolar organic compounds on coacervation in the systems. The effect of salts in the soap system was comparable to the known effects of salt in gelatin-water systems (Derwichian—*Bull. soc. chim. France* 1950, 532). The influence of polar and nonpolar organic compounds was explained on the basis of assuming two different actions which resulted in the different substances going to different places in the micelle (Booij *et al.*—*Proc. Koninkl. Nederland. Akad. Wetenschap.* 53, 407). The actions of aliphatic, aromatic, and mixed hydrocarbons were discussed as related to the above mentioned assumption (Booij—*Ibid.* 52, 1100; 53, 59, 299, 882). It was also observed that systems containing phosphatides showed unusual morphology at different pHs (de Jong & de Heer—*Ibid.* 52, 784). The action of inorganic salts, and polar and nonpolar organic solvents on the elastic behavior of oleate solutions was also used in studying the structure of the micelles (de Jong *et al.*—*Ibid.* 53, 7, 233, 743, 759).

The simple aryl sulfonates were found to form water-insoluble complexes with gelatin somewhat similar to the complexes formed by some detergents and acid dyes with gelatin (Smith—*Nature* 164, 447).

Data on the structure of sodium palmitate (Kohlhaas—*Chem. Ber.* 82, 487) and sodium dodecyl sulfate (Rawlings & Lingafelter—*J. Am. Chem. Soc.* 72, 1852) were obtained in X-ray diffraction studies. Stosiek (*J. Chem. Phys.* 18, 1035) attributed disordering of structure of soap, in observations of this type, to a stacking disorder of micellar layers and to random orientation of long chains within each layer.

PERFORMANCE TESTS. The performance of laundry detergents is estimated by standard wash tests using standardized washing machines and standardized soiled fabrics, and measuring light reflectance of the washed fabric. These tests have been rigidly investigated to improve reproducibility and agreement with actual practice. New procedures for this type of test using carbon black as the soil (Vaughn & Suter—*J. Am. Oil Chemist's Soc.* 27, 249) and heavily soiled cotton as the test cloths (Griesinger & Nevison—*Ibid.* 96) were devised. Harris & Brown (*Ibid.* 135, 564) in reviewing the standardization of soil or soiled fab-

ric for the test reported that there was no agreement concerning the type of soil and that commercially available soiled fabrics were not sufficiently standardized to give correlation with practice. Their investigations on five laboratory wash tests and one practical wash test showed good correlation which was better in hard than in soft water. The order of effectiveness of detergents tested was soap, built nonionic and built alkyl aryl sulfonate, and lauralkyl sodium sulfate. In Barker & Kern's (*Ibid.* 113) comparison of two detergency estimation methods, use of light reflectance of soiled and washed fabrics gave results comparable to those involving light transmission of wash liquors for nonionic and anionic detergents, whereas with soap the results were not comparable.

Snell *et al.* (*Ibid.* 62, 73, 289) in a discussion on the nature of soil to be deterged mentioned that soil was retained on fabric by mechanical forces, chemical forces, electrostatic attraction, and bonding. Their work on performance of tall oil soaps showed that it was equivalent to soaps from various unsaturated vegetable oils, but not as good as sodium oleate.

In some performance tests the suspending power aspect of detergency was studied. The data of Weatherburn *et al.* (*Can. J. Research* 28F, 213, 363, *Textile Research J.* 20, 510) showed that ability to suspend carbon increased to a maximum with a certain concentration of the soap and then remained constant. Curves for C₁₄, C₁₆, and C₁₈ saturated soaps were similar, that for C₁₂ lower, and the curve for the oleate was higher. Increase in temperature caused a decrease in suspending power of the lower saturated soaps, and increased that of the longer chain soaps, whereas the suspending power of oleate was unchanged between 25 and 80°C. Addition of builder to soap solutions decreased the suspending ability of the soap. Part of this loss due to builders could be compensated by addition of carboxymethylcellulose. Merrill & Getty (*J. Phys. & Colloid Chem.* 54, 489; *Ind. Eng. Chem.* 42, 856) determined the iron oxide, ilmenite black, and raw umber suspending power of various soap builders and combination of these with an alkyl aryl sulfonate detergent. Dodecyl benzene sodium sulfonate suspended more iron oxide than the builders, less ilmenite than the silicates, but usually more than sodium carbonate or hydroxide, and less umber than almost any builder. Optimum suspending ability for variety of soils was obtained with synthetic detergent-alkaline builder mixtures. In testing the hypothesis that suspendability is due to the detergent increasing the ζ -potential of both soil and fiber, Doscher (*J. Colloid Sci.* 5, 100) found that in many systems the ζ -potential did not correspond to the suspendability.

Some detergency investigations were limited to studies on the individual fibers. Lambert (*Ind. Eng. Chem.* 42, 1394) emphasized the importance of the acidic characteristics of cotton fiber, and pointed out how radioisotope tracers could be used in measuring the adsorption and exchange of calcium on cotton as it occurs in laboratory wash tests simulating hard-water laundering. The same investigator and Sanders (*Ibid.* 1388; *J. Am. Oil Chemists' Soc.* 27, 153) recorded data from various test methods and practical household operations on various detergents and built detergents. The order of decreasing efficiency of the products was: built ionic, built nonionic, built soap, built anionic, nonionic, and anionic.

Nutling (*Am. Dyestuff Repr.* 39, No. 8, P 260) devised a detergency test to evaluate detergents used in finishing processes employed in wool fabric manufacture under conditions of high concentration and low bath ratio. Stadler (*Melliand Textilber.* 31, 556) suggested use of nonionic detergents for wool because anionic and cationic detergents have a strong affinity for wool fiber and require large amounts to maintain the necessary concentration in solution. LaFleur (*Am. Dyestuff Repr.* 39, No. 12, 385) compared detergencies of 28 products on the basis of concentration and cost on woolen cloth soiled with the oil used in wool manufacture. In comparison tests by Furry & McLendon (*Ibid.* 209), 27 synthetic detergents were evaluated for removing soil from cotton and wool fabric. In a study of sorption of soap by various textile fibers the order of increasing sorption was cotton, nylon, acetate, bright viscose, dull viscose, and wool. Cotton and viscose rayon preferentially sorbed alkali, while acetate, rayon, nylon, and wool sorbed fatty acid with lower molecular weight soap and preferentially sorbed alkali with the higher soaps,

In a study of the behavior of silicates, persalts, and soda for laundering artificial wool, Neu (*Fette u. Seifen* 52, 610) recorded interesting observations on destruction of the fiber. Persalts were quite destructive. Washing 50 times in nitrogen atmosphere caused no significant destruction, and boiling for 24 hours in the presence of alkali had less effect than 48 half-hour boilings interspersed with holding periods. The presence of copper or iron in the wash water accelerated the decomposition of the fiber.

Reutenauer *et al.* (*Bull. mens. ITERG* 3, 448; 4, 257; *Teintex* 14, 361) evaluated many commercial detergents and mixtures of these with soap with respect to their sudsing wetting, and emulsifying abilities. The presence of synthetics did not modify the properties of soap solutions, but poor emulsifying properties in synthetics were in certain cases improved by addition of soap.

Skin compatibility tests for raw materials and laundering compounds were devised by Peukert (*Fette u. Seifen* 52, 415), Killian & Marsh (*Soap Sanit. Chemicals* 25, No. 6, 44; No. 11, 31). The former found that skin reactions were higher for all synthetic detergents tested than for soap.

In a detergency test for metal cleaning Harris *et al.* (*A.S.T.M. Bull.* 1950, 308) suggested incorporating "radioactive-tagged" material in the soil and using residual radioactivity as the criterion. A detergency test for dishwashing compounds was based on removal of a soil containing peanut butter, coagulated egg white, and cooked oatmeal from glass slides (Spangler *et al.*—*Am. J. Public Health* 40, 1402).

Cleaning materials were also evaluated in regard to their germicidal abilities. Tests in use for this purpose and their interpretation were compared by Stuart *et al.* (*Soap Sanit. Chemicals* 26, No. 1, 121; No. 2, 127). In comparison of quaternary ammonium compounds and hypochlorites for disinfection of glasses it was shown that the former were adversely affected by hard water and additional amounts were required with such waters (Mallmann & Harley—*Ibid.* No. 3, 126; Dvorkovitz & Crocker—*Ibid.* No. 9, 114). In one investigation on the sterilizing effect of quaternary compounds the efficiencies of 10 products were evaluated at various temperatures and alkalini-

ties (Curran & Evans—*J. Dairy Sci.* 33, 1). The use of detergent sanitizers in dairy utensil cleaning considerably lowered the bacterial count of milk produced (Puhle—*Soap Sanit. Chemicals* 26, No. 12, 133; Lindquist—*Proc. 12th Intern. Dairy Congr.* 3, 294). In a test of 142 surface active agents for bacterial action against tubercle bacilli the materials were not considered good as a group (Smith *et al.*—*Pub. Health Repts.* 65, 1588).

Certain chlorinated diaromatic methanes when added to soaps induced powerful bacteriostatic activity (Price-Bonnett—*Surgery* 24, 542; Bean & Berry—*J. Pharm. Pharmacol.* 2, 484). Alexander *et al.* (*Research, Surface Chemistry Suppl.* 1949, 299, 309, 317, 325) studied bacterial action of soap and detergents and mixtures of these with electrolytes and phenols, and discussed the data from such colloid aspects as particle surface, absorption on particles, effect of micelle, etc. Bactericidal activity of surface-

active agents was comparable to their hemolytic action (Haldenwanger—*Zentr. Bakt. Parasitenk. Abt. 1*, 153, 263) and protein precipitation (Haurowitz—*Bull. faculte med. Istanbul* 12, No. 3, 183). It was suggested that some sort of combination takes place with micelles.

In one investigation the relation between structure of invert soaps and their antimycotia power were recorded (Jerchel & Kimmig—*Chem. Ber.* 83, 277). Mold growth on eggs in storage was inhibited or completely prevented by dipping the eggs in solutions of certain cationic detergents.

The effect of general use of synthetic detergents on bacterial sewage processing was investigated (Wadams—*Surveyor* 109, 39; Elton—*Inst. Sewage Purification J. and Proc.* 1949, 351). Primary sedimentation, bacterial activity, sludge digestion, or methane production were not affected when tested in concentrations likely to be encountered in sewage treatment.

Floor Cleaner Evaluation

M. MARTIN MAGLIO, Vestal Laboratories Inc., St. Louis, Missouri

CONSIDERABLE research has been devoted in the past 15 years to the evaluation of soaps and surfactants¹ as detergents for textiles, particularly cotton and wool. Relatively little has been reported however in the scientific evaluation of such detergents as floor and all-purpose cleaners, despite their volume usage. A notable exception is Harris and his coworkers, who recently reported (6) the evaluation of synthetics, built and unbuilt, for this purpose. Another contribution has been that of Du-Bois, who extended (3) the detergency test method of Gilcreas and O'Brien (5) to include linoleum cleansing, but this method does not simulate actual practice and was rejected for this reason.

In their paper, Harris *et al.* stressed a satisfactory test method and the necessity for following prescribed precautions to obtain duplicable results and presented a statistical treatment of the data. No attempt was made however to compare the detergent properties of a soap against those of another or of a soap against those of a surfactant.

Our object in presenting this paper is three-fold: a) to describe a test method which yields replicable results; b) to record the benefit of our experience with liquid soaps vs. synthetics as floor cleaners; and c) to present a statistical treatment of the data obtained with the hope that in the future most, if not all, such experimental data will be weighed statistically to substantiate further any conclusions derived.

It is an accepted fact that a satisfactory test method should simulate practical conditions as closely as possible and should yield results which can be duplicated under practical or "use" conditions. Accordingly a detergency test method for floor cleaners should utilize the surfaces and the soils as well as the cleaning equipment and methods of cleaning encountered in actual practice. Where necessary or advis-

able, modifications in technique may be made so long as they do not affect the results appreciably.

In our work we employed light-colored linoleum as the surface but had to resort to a synthetic soil (4) as the test soil, due to the complexity and variability of natural soils depending on their environments. Preliminary results on natural soil collected from our office, laboratory, and plant floors over a period of months revealed the advisability of adopting a synthetic soil for our detergency studies so as to permit duplication of results and provide a more difficult measure of detergency.

The preparation of test panels and their soiling were accomplished as described below.

Test Panels. The whitest plain linoleum available was used in this investigation. It was Armstrong's Heavy Grade Battleship Linoleum with Safety Back Standard Gauge. Actually its color was yellowish-white. Panels measuring 5¾ in. by 5¾ in. were cut from a large section of the linoleum, and these were scrubbed individually by means of a good grade of scouring powder and a bristle brush to remove the factory finish. The panels were rinsed thoroughly with plain water and were allowed to dry overnight before soiling.

Standard Soil and Soiling. The standard soil used was essentially that described in Federal Specification for Cleaner; for Painted Surfaces, Containing Synthetic Detergent, P-C-431 (4).

The freshly prepared soil suspension was well mixed and was then applied by means of a three-quarter-inch camel's hair brush to an area 2½ in.

Substance	Grams
Metallic Brown (C. K. Williams and Co., B-3881) ²	20
Hydrogenated Vegetable Oil (Crisco).....	1
Petrolatum, light yellow, U.S.P.....	1
Lubricating Oil (SAE-80).....	1
Kerosene.....	20
Carbon Tetrachloride.....	20

² Consists of 80-85% Fe₂O₃ and 2% maximum calcium as CaCO₃; remainder is silica and silicate minerals.

¹ Coined by General Aniline and Film Corporation to connote surface active agents, including penetrants, emulsifiers, wetting agents, foaming agents, and detergents.