equally as or more convenient than the Wesson test. However, for the evaluation of refined oils as outlined above, the Wesson method appears preferable since it gives a better indication of the point at which further alkali refining becomes wasteful. The chromatographic method not only shows losses beyond this point (cf. Table II) but in contrast to the Wesson method seems to produce adsorption of some neutral oil in the absence of free fatty acids and non-glycerides. Thus it has been found by Desnuelle and Micaelli, and confirmed during the present work, that an oil treated according to the Wesson method and reacidified suffers on repeated Wesson treatments only a loss of weight equal to that of the added free fatty acids. The loss of neutral oil is nil or practically nil[3] even in the case of the easily saponifiable coconut oil while a repeated chromatographic treatment results in a further loss (cf. Table III). Conversely, the chromatographic treatment of crude oils, which gives a higher loss than the Wesson method, yields oils with a free acid content of about 0.15% while Wesson treated oils show an acidity of approximately 0.02%, which is more in line with the results obtained on careful refining in the factory.

Summary

Wesson loss determinations carried out on a number of alkali-refined oils have shown them to retain varying amounts of non-glycerides, depending on the type of the oil and on the method of refining. Oils refined according to the official A.O.C.S. method or with sodium carbonate followed by re-refining are practically free of non-glycerides while those refined with a moderate excess of alkali over the theory (10-25%) retain appreciable amounts of non-glycerides. The Wesson method could therefore be used as a quantitative test for the degree of refining.

The chromatographic method is not suitable for this purpose since it shows losses when applied both to carefully refined and to previously chromatographed oils, but in agreement with the Linteris' and Handschumaker's results it has been found applicable to the estimation of loss constituents in crude oils.

[3]King and Wharton (18), on determining the Wesson loss of neutralized and bleached, but not Wesson treated cottonseed oil, to which 2% of free fatty acids had been added, found a loss of neutral oil amounting to 0.2%. However there remains the question whether this loss was actually due to saponification of neutral oil or to the removal of rest amounts of non-glycerides.

Acknowledgment

The authors are indebted to F. B. Shorland, director of the Fats Research Laboratory, D.S.I.R., for permission to publish, and to S. G. Brooker, chief chemist of Abels Ltd., Auckland, N. Z., for valuable suggestions and criticism.

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[Received September 5, 1951]

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

Report of the Literature Review Committee *

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Physiology and Biochemistry

REVIEWS. The reviews concerning the text of this division were on the following subjects: Biochemical and nutritional aspects of fat chemistry (Deuel & Greenberg—*Fortschr. Chem. org. Naturstoffe 6*, 1); fatty acids in nutrition (Holman—*Proc. 3rd Conf. on Research Am. Meat Inst., Chicago, 1951*, 1; *Fette u. Seifen 53*, 332), lipide metabolism (Gurin—*Ann. Rev. Biochem. 20*, 179); oxidation of fat by autoöxidation as well as the biological process (Täufel—*Fette u. Seifen 53*, 558); importance of unsaturated acids in dermatology (Fiedler—*Ibid. 52*, 721); serum cholesterol and phospholipide (Wallis—*Bull. Ayer Clin. Lab. Penna. Hosp. 4*, 79); formation of milk fat (Täufel—*Z. Lebensm.-Untersuch. u. -Forsch. 93*, 140); and antilipotropic activity of cystine (Tyner—*Univ. Microfilms, Ann Arbor, Mich., Pub. No. 2470*, 68 pp.). The papers given in a symposium on studies on arteriosclerosis which concerned biochemistry of lipides were on renal hypertension of dietary origin (Hartroft—*J. Gerontol. 6*, 154), lipfanogens and antilipfanogens (Sims—*Ibid.* 159), lipide metabolism (Kendall—*Ibid.* 162), and metabolism of arterial tissue (Kirk—*Ibid.* 167).

FAT NUTRITION. Of the new reports on nutritive value of butter versus margarine, no difference was found in two studies while one report maintained that summer butter contained some unknown growth stimulating factor. Euler & Euler (*Arkiv. Kemi 3*, 31) recorded no significant differences in growth rate, reproduction, or lactation through four generations of rats on the two fats. Smits (*Voeding 11*, 298), who surveyed pertinent literature on the subject, was of the same opinion. Groot's (*Mededel Lab. Univ. Amsterdam, Inst. Volksvoed 11*, No. 14, 84 pp.) work indicated that the unsaponifiable fraction of summer butter had a growth promoting effect which was not due to known vitamins. He did not obtain a growth effect from vaccenic acid or the phospholipides of the butter.

The nutritive value of mono-, di-, and triglycerides were found to be the same when compared on the basis of growth, appearance, and post mortem examinations of rats fed at 25% level of the pure materials (*Mattson et al.—J. Am. Oil Chemists' Soc. 28*, 386). A comparison of monoglycerides prepared from cottonseed oil versus cottonseed oil also showed no difference as measured by growth response, reproduction ability, and lactation performance (Ames *et al.—Ibid.* 31).

Some studies reflected the need for fat in nutrition. When rats and pigs were fed diets containing two to 20% fat, the best growths and assimilations of protein and carbohydrate were obtained at the highest fat levels (Tangl *et al.—Agartudomany 2*, 365). Gomberg (*Arch. Tiernähr. 2*, 307) recorded the reductions in daily gains of calves after reducing the fat content of the milk on which they were maintained. Later he showed that adding easily digestible carbohydrates to the low-fat maintenance milks could aid in closing the difference. Diets of fat and protein were able to prolong considerably the life

of rats on vitamin B_1-deficient diets; whereas, the presence of as much as nine per cent carbohydrate in the diets resulted in a high fatality rate of the test animals (Yudkin—*Biochem. J. 48*, 608). In such studies, the fats did not cause the noticeable or prolonged rise in the blood pyruvate as was known to occur when carbohydrates were fed to the vitamin B_1-deficient animals (De Caro & Rindi—*Nature 167*, 114). Kaunitz & Slanetz (*Fed. Proc. 10*, 360) found in studies on the vitamin B sparing action of lard that when rancid lard was fed with the diet it was deleterious, whereas fed separately from the vitamins it resulted in normal growth. They also associated the protective action of lard on vitamin B deficiencies with certain molecularly distilled fractions of the fat but they were unable to conclude whether lard contained vitamins or whether other properties were responsible.

Earlier literature that had described a biotin-like action of oleic acid was supplemented by new information. This activity was recently reported in both *cis-* and *trans*-isomers of the acid; but with the latter and with *cis*-isomers the effectiveness lessened as the double bond was moved away from the nine-carbon position (Amber *et al.*—*J. Biol. Chem. 192*, 611). Similarly, oleic acid was found to be a more effective replacement for biotin than vaccenic acid (Sankar & Sarma—*J. Sci. Ind. Res., India 10B*, 3). Although oleic acid did have a sparing action for biotin and folic acid, deficiencies of these did not improve absorption of the acid from the intestines (Woodruff—*Brit. J. Exptl. Path. 31*, 405).

Kaunitz & Slanetz (*Fed. Proc. 10*, 360), who previously discovered that lard contained a vitamin A-like factor, have now recorded that the factor occurs in other fats and oils. The vitamin A-like effect of the materials tested, expressed in units per gram, were lard 4, chicken fat 10, beef fat 10, olive oil 3, sesame oil none, and cottonseed oil none. The factor in lard was concentrated to 60-90 units per gram by molecular distillation. Engel (*Voeding 12*, 310) did not confirm the above results.

The effect of fat and fatty emulsifiers on utilization of vitamin A and carotene by rats was studied. Burns *et al.* (*Arch. Biochem. 30*, 341, 347) found that polyoxyethylene sorbitan monopalmitate (Tween 40) diminished the utilization of vitamin A while it increased slightly that of β-carotene; and that fat increased the utilization of both; whereas, mineral oil was detrimental even when dietary fat was increased. According to Deuel *et al.* (*J. Nutr. 43*, 371) the vitamin A potency of carotene was 30% greater in margarine than in cottonseed oil; whereas the potencies of preformed vitamin in the two oils were the same. Vitamin A esters diluted with mineral oil or ethyl laurate were not utilized by rats—probably because the diluent inhibited hydrolysis *in vivo* (Week & Sevigne—*J. Nutr. 42*, 525). Rats fed trilaurin plus methyl linoleate as the fat source had reproductive failure until the diet was supplemented by 2.5% wheat germ oil. This was interpreted to indicate that natural oils contained an undetermined factor, not identified with vitamins or essential fatty acids, which was necessary to permit the female rat to rear her young (Keane *et al.*—*J. Nutr. 45*, 275). Earlier work showing that coconut oil improved calcium utilization was complicated by reports which indicated that the refined and deodorized product had an adverse effect (De & Karkun—*Science & Culture 15*, 486); furthermore, certain fractions could be crystallized from coconut oil which were superior in inducing calcium and magnesium utilization (Sen & Mukerjee—*Indian J. Physiol. 4*, 137). Kaufmann & Schmidt (*Fette u. Seifen 52*, 528) indicated that phosphatides had growth promoting value for *Lycastis ranauensis*, a type of glow worm.

The new work on essential fatty acids concerned metabolism principally. *Trans*-isomers of the essential fatty acids were biologically inactive (Holman—*Proc. Soc. Exptl. Biol. & Med. 76*, 100). Thus, the detection of polyunsaturated fatty acids in a tissue does not imply that the substance is active. Fat deficient rats receiving ethyl arachidonate as the essential fat deposited 33% of the ingested polyunsaturated acid (Holman & Taylor—*Arch. Biochem 29*, 295). Fat deficiency in rats caused a marked increase in liver cytochrome oxidase activity and a decrease in endogenous respiration; the former being reduced by essential fatty acid (Kunkel & Williams—*J. Biol. Chem. 189*, 755). Low polyunsaturated acid content of tissue of rats was effected by withdrawal of essential fatty acid, pyridoxine, or thiamine from the diet; but with deficiency of the latter two, symptoms of essential fatty deficiency did not occur Medes *et al.*—*Arch. Biochem. Biophys. 32*, 70). It was concluded that pyridoxine had no special supplementary action in production, or conservation of essential fatty acids. Similarly no interrelationship between essential fatty acid and tocopherol requirements could be demonstrated (Anisfeld *et al.*—*J. Nutr. 45*, 599).

High levels of cod-liver oil in the diet of rats and mice induced dystrophy of the skeletal musculature which could be prevented by administration of vitamin E (Tobin—*Arch. Path. 50*, 385; Schwarz—*Z. Physiol. Chem. 283*, 106). This could be the same fatal disease known as steatitis or yellow fat in mink which was also prevented with vitamin E (Lalor—*J. Nutr. 45*, 183; Mason & Hartsough—*Fed. Proc. 10*, 389). In mink the disease was associated with feeding food high in polyunsaturated fat such as fish, horse meat, and linseed oil. The disease could also be produced with cod-liver oil. A histochemical study of yellow-brown pigment found in the adipose tissue under such conditions demonstrated that it was unsaturated fatty acids oxidized beyond the peroxide stage (Grenados & Dam—*Acta Path. Microbiol. Scand. 27*, 591). Hartroft (*Science 113*, 673) suggested that these ceroid-like pigments resulted from the action of those lipid constituents involved and some component of free red blood cells. Dubouloz & Laurent (*Compt. rend. soc. biol. 144*, 1183) discovered that a red-brown substance isolated from beef liver destroyed fatty peroxide. This could be pertinent to this subject, since it was known that tocopherol, an antioxidant, was a preventative.

Because of the appearance of patents and literature on manufacture of edible products from fish and other reverting oils by inhibiting reversion through polymerization, the effect of excessive heating of oils became more pertinent in the study of nutrition of the products. Crampton *et al.* (*J. Nutr. 43*, 431, 533; *44*, 177; *Science 113*, 408) demonstrated that oils polymerized at 275°C. even in the absence of oxygen when fed to rats, depressed growth and efficiency of feed utilization, the severity varying with the degree of original unsaturation of the oils, length of heating each oil, and the level at which the heated oil was incorporated in the diet. The primary cause of the toxic effect was attributed to the presence of dimeric fatty acid radicals which were in some way inimical to the well being of the animals. Oils heated to extremes (340-400°C.) were carcinogenic to mice, although attempts to isolate carcinogens from such oils were unsuccessful (Peacock—*Brit. J. Nutr. 2*, 201).

Even though branched chain acids have not entered human diets since the war when synthetic fats were consumed in Ger-

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many, studies on the physiology of these were still pertinent to the interest of their probable use in animal feeding. Just and Walther (*Chem. Tech., Berlin 2*, No. 11, 332) suggested that the fatty acid produced by the oxidation of mixed paraffins could be fed to and better utilized by livestock in the form of amides of the acids. However, Sartory *et al.* (*Ann. inst. Pasteur 78*, 93, 382) reported that such acids which also contain an ethylenic linkage were highly toxic in any means of administration, and either these or saturated members when injected produced tuberculin reactions in animals. Branched chain fatty acids isolated from tubercle bacilli when fed to rabbits were excreted in urine partly as monocarboxylic and partly as dicarboxylic acids (Asano *et al.*—*J. Pharm. Soc. Japan 64*, No. 8A, 25; No. 9A, 29; *65*, No. 4A, 15; No. 5/6A, 10; *68*, 147; *70*, 477). This indicated that some ω-oxidation had taken place.

FAT ABSORPTION. In a paper on this subject Frazer *et al.* (*Brit. J. Nutr. 3*, 358) described how digestion of fats may be studied by feeding tests in which samples of intestinal contents were removed with the use of a Miller-Abbot intestinal tube. Cook & Thomson (*Quart. J. Exptl. Physiol. 36*, 61) studied absorption of fat and cholesterol by feeding in large amounts so as to outweigh errors due to synthesis or destruction in the intestines. At 16.6% dietary fat rats absorbed 92%, guinea pigs 77%, and rabbits 94%; cholesterol at 1.6% in the diet was absorbed at 34, 47, and 77%, respectively, in these animals. Levels of plasma cholesterol in these animals increased in the same order as absorption. Wollaeger's *et al.* (*Fed. Proc. 10*, 271) studies on absorption showed that in substituting a mixed diet containing fat for a fat-free diet, the excretion of total fats and fatty acids decreased; and when triolein was fed as the fat the fatty acids of the excreted lipides were not all monoenoic. These could cause errors in fat absorption measurements. X-ray irradiation increased fecal fat although absorption of the ingested fat seemed normal (Mead *et al.* —*J. Nutr. 43*, 485).

A study of fats made from peanut oil by blending hydrogenated oil of 60°C. melting point with refined oil as compared to hydrogenating the entire oil to the desired melting point indicated that there was no significant differences in the absorption when fats of the same melting point were compared (Sahasrabudhe & Subrahmanyan—*J. Sci. Ind. Res. India, 10B*, 119). The tests were made on blends and hydrogenated products melting at 38, 45, and 51°C. Tests on six tropical fats (corozo, morro, tambor, sapayulo, cacao volador, and aceituno) indicated 92-97% absorption by rats (Squibb *et al.*—*J. Nutr. 44*, 547).

Some absorption work concerned the mechanism of the process. Favarger & Collet (*Helv. Physiol. Pharmacol. Acta 8*, 15C) estimated that less than one-third of the fat was hydrolyzed during intestinal absorption and was absorbed from the intestines either as the tri- di-, or monoglyceride. Desnuelles' *et al.* (*Compt. rend. soc. biol. 144*, 1182) finding that pancreatic lipase hydrolysis of triolein did not readily yield free glycerol could be considered as supporting this same view. According to Mead *et al.* (*J. Nutr. 43*, 477) emulsified methyl esters of fatty acids were not normally absorbed from intestines unless a normal fat was present. This indicated that glycerides were important factors in the absorption of fatty substances. One of the publications on fat metabolism contained a list of microorganisms which were present in the feces of children and infants and were capable of splitting fats (Goeters—*Z. Kinderheilk 36*, 438).

The importance of emulsification in internal absorption of fats was evident in a number of studies. Mono- and diglycerides facilitate emulsification and absorption (Sager—*Monatschr. Kinderheilk. 99*, 78). Phospholipides increased growth through increased fat utilization in baby pigs on semi-synthetic milk (Sheffy *et al.*—*J. Animal Sci. 10*, 867). Some patients that did not tolerate fat were able to use emulsified fat (Shoshkes *et al.*—*J. Am. Dietetic Assoc. 27*, 197). In similar work on surgical patients the commercial emulsifiers "Tween 80" and animal fat monoglycerides increased fat absorption (Ellison *et al.*—*Surg. Forum, Proc. 36th Clin. Congr. Am. Coll. Surgeons 1950*, 478). However, in work on premature infants and on two full-term infants with steatorrhea, administration of "Tween 20" was no more effective in inducing fat-absorp- than was skim milk alone (Johnson *et al.*—*Am. J. Diseases Children 80*, 545). It was also demonstrated that choline aids absorption of fat (Tidwell & Nagler—*Fed. Proc. 10*, 258).

It seemed quite premature to use the commercial polyoxyalkylene derivatives as emulsifiers for food and internal medical purposes for there still existed some doubt as to their innocuousness. Sorbitan monolaurate and polyoxyethylene sorbitan monolaurate fed at high levels to rats or hamsters caused diarrhea, reduced growth, and increased mortality; and on autopsy distended caeca, oxalate stones in the bladder, degeneration of kidney tubules, incomplete spermatogenesis and other abnormalities were observed (Harris & Sherman—*Arch. Biochem. & Biophysics 34*, 249). "Tween 80" when given to cholesterol fed rabbits caused tremendous foam cell accumulation in the reticulo-endothelial system and lipide infiltration of the renal tubular epithelium (Payne & Duff—*Arch. Path. 51*, 379). In similar studies with "Tween 80" and "Triton 20," increased levels of cholesterol and phospholipides were found in the rabbit's blood, but the incidence of atherosclerosis was much lower in the test group than in controls fed cholesterol without "Tween 80" or "Triton 20" (Kellner *et al.*—*J. Exptl. Med. 93*, 373, 385). A study of the intermediate metabolism of several fatty polyoxyethylene emulsifiers indicated that the polyoxyethylene moiety was completely eliminated in urine and feces (Culver *et al.*—*J. Pharm. & Exptl. Therapeutics 103*, 377). Tests with another emulsifier, "RD11," indicated that it inhibited secretion of the total available pepsin and lysozyme (Labstein & Fogelson—*Am. J. Digestive Dis. 18*, 213).

The pathway of absorption of fats was the concern of some studies. Bloom *et al.* (*Am. J. Physiol. 166*, 451; *J. Biol. Chem. 189*, 261) using C^{14}-labeled fatty acids reported that the long chain acids (C_{14} and longer) passed into the lymph; whereas, with C_{12} and C_{10}, respectively, only 15-55% and 5-19% were recovered from the lymph. This suggested that the major portion of the short chain fatty acids was transported via the blood stream. In this work on palmitic acid, the recovery of 96% from the lymphatics not in the form of phospholipides was interpreted to indicate that phospholipides were not important transport forms of the acid. In comparing the route of absorption of free fatty acids and triglycerides in like manner, Reiser & Bryson (*J. Biol. Chem. 189*, 87) concluded that both were by the same route.

Fundamental information pertinent to intravenous fat feeding was recorded. The fever reaction which occurs in the practice was inhibited by incorporation of the commercial emulsifier "Triton A-20" which also reduced the rate at which the injected fat disappeared from the blood (Lambert & Frost—*Am. J. Physiol. 164*, 490). The excessively high febrile reaction of old emulsions prepared for intravenous nutrition was less thermogenic when reautoclaved (Meng.—*J. Lab. Clin. Med. 37*, 222). In tracing the intravenous injected fat by radioactive means, Ruttenberg *et al.* (*J. Clin. Invest. 28*, 1110) found it in highest concentration in the spleen, lung, and liver, in that order with dogs; and in mice the highest concentration was in the lungs. In similar work Murray & Freeman (*J. Lab. & Clin. Med. 38*, 56) generally agreed with the above in case of artificial emulsions; whereas, the fat of dog chyle injections or fed fat never concentrated in those places. Shafiroff *et al.* (*Exptl. Med. Surg. 9*, 184) demonstrated that fat administered intravenously in human subjects was rapidly oxidized. Deuterolabeled fat was traced in this work. Radioactive means were also used to discover that intravenous administration of carbohydrates with fat reduced the oxidation rate of the latter about one-half; whereas, fasting increased the rate of oxidation of the fat (Geyer & Waddell—*Fed. Proc. 10*, 383). Injection of fat emulsions into the peritoneum resulted in the desaturation of the fatty acids and some decrease in the molecular chain length (Pontremoli & Arrigo—*Boll. soc. ital. biol. sper. 26*, 253).

INTERMEDIATE METABOLISM OF FATS. The communications on intermediate metabolism of fats concerned such aspects of lipides as transport in the body, liver lipides, biological oxidation, biological synthesis, and mobilization. This subject was treated comprehensively by Kartha (*Separate Published by Maharaja's College 1*, 110 pp.; *2*, 111 pp.) in connection with his hypothesis which suggested that there was a natural tendency for fatty acids to be distributed among glycerides in each species at random, except that in some, true randomness was limited by the specificity of the enzymes involved in elaboration of the fats. In vegetable oils, the low content of saturated acids permitted random distribution. In land animals containing large amounts of saturated acids in their fats, the formation of trisaturated glycerides is suppressed to effect the lower melting point which is characteristic of the biological source and within the capacity of the enzyme's system and its specific conditions for action. Thus, after a fat attained the maximum trisaturation possible to remain still fluid in its environment any additional saturated acids could only enter glycerides containing unsaturated acids in order that fluidity was maintained. He suggested that each species elaborates fats primarily as energy stores, to give maximum unit energy possible with fluidity and stability of the environments. Thus aquatic fats contain long chains fluidized by multiple unsaturation; land animal fats contain mostly C_{18} fats with enough

unsaturation for fluidization in each organism; whereas in certain tropical fats fluidization was achieved with the required stability for the environment by elaboration of short chain acids rather than by unsaturation in long chains.

Giordano (*Atti accad. fisiocrit. Siena Studi med senese 18,* 85, 89, 93, 95, 98, 100) in studies on the effect of metabolism of carbohydrates, lipides, and proteins on blood lipides and glycogen found that only small changes occurred. In poultry, according to Walker *et al.* (*Poultry Sci. 30,* 525) slight decreases occurred in blood lipides on low-fat rations, while high-fat rations caused no change. The presence of mixed soybean sterols in diets of cholesterol-fed chicks (Peterson—*Proc. Soc. Exptl. Biol. Med. 78,* 143), and inositol in diets of cholesterol-fed rabbits (Dotti—*Ibid.* 165) inhibited the expected rise of cholesterol levels in the serums. Avidin-rich diets inhibited the capacity of cholesterol-fed rats to store cholesterol (Okey—*J. Nutr. 44,* 83). In the same studies biotin increased the storage of both fatty acids and cholesterol. Studies on fasting rats by Chevallier *et al.* (*Compt. rend. soc. biol. 144,* 1394, 1396) indicated that polyunsaturated fats do not disappear from the body for the first 140 hours or until fat depletion was quite far along. During such depletion, the tocopherol content of the animal remained unchanged.

In metabolism studies with C^{14}-labeled glycerol, most (35-50%) of the C^{14} was expired as carbon dioxide, glycerol moiety of liver lipides accounted for five per cent, and blood and liver glycogen contained about 16% of the labeled carbon (Karnovsky & Gidez—*Fed. Proc. 10,* 205). In similar work Doerschuk (*J. Biol. Chem. 193,* 39) found 80% respired as carbon dioxide, 6% in urine, 1.5% in feces, 2.3% in acetone soluble lipides, .13% in glycerol phosphoric acid, 5.5% in liver and less than 1% in liver lipides. He also found some in the glycerol moiety of lipides.

The work on metabolism of fats in the liver concerned principally agents which modify the amount of fat. Fat infiltration in liver brought about by fat feeding, injection of fat emulsions, starvation, alloxan diabetes, and cholesterol feeding was observed histologically and described (Woerner *et al.*—*Proc. Am. Diabetic Assoc. 10,* 180). In all these cases fat was removed by adding extra choline to the diet. Studies on the relative lipotropic action of choline and inositol on fatty livers, produced under various dietary conditions, indicated that the latter had either no or a slight and variable lipotropic effect (Best *et al.*—*Biochem. J. 48,* 448, 452). Choline appeared to be necessary also for normal bile production, for choline-deficient animals secreted smaller quantities of bile than controls fed choline (Colwell—*Am. J. Physiol. 164,* 274). The unsaturation of fat in fatty livers produced by choline deficiency was similar to that of normal liver fat (Capraro & Marazzi—*Boll. soc. ital. biol. sper. 25,* 51). The lipotropic activity of methionine was confirmed and analyses of the effects presented by Schettler (*Z. ges. exptl. Med. 116,* 444), Burke *et al.* (*J. Biol. Chem. 188,* 723), and Jensen *et al.* (*Ibid. 192,* 395). The latter group of investigators also tested ethionine sulfoxide, S-propylhomocysteine, S-isopropyl-homocysteine, and S-ethylcysteine and found them inactive. György *et al.* (*Am. J. Physiol. 166,* 436, 441) identified a lipotropic fraction from pancreas as being enzyme in nature. It was assumed that the lipotropic activity was based on enzymic liberation of methionine from ingested proteins in the intestines. Schilling's (*Abstracts Commun. 1st Intern. Congr. Biochem. 1949,* 15) studies on lipotropic pancreas fractions indicated that the lipocaic lipotrope was heat stable and did not contain choline, methionine, or a proteolytic enzyme as the active material.

The interrelationships of other dietary materials and their lipotropic effects were studied. The presence of vitamin B_{12} was necessary to obtain a lipotropic effect from choline (Burns & McKibbins—*J. Nutr. 44,* 487). The speed of obtaining abnormal livers in protein deficient animals was increased by a simultaneous deficiency of vitamin E. Excessive cysteine in diets also increased liver fat (Levy—*Arch. sci. physiol. 4,* 211). Chronic alcoholization did not increase the tendency for development of nutritional fatty livers (Forbes & Duncan—*Quart. J. Studies Alc. 11,* 373).

An anterior pituitary preparation administered to rats caused rapid mobilization of lipides from depots with an accumulation in the liver (Campbell & Lucas—*Biochem. J. 48,* 241). In discussing the propylthiouracil, thyroid, and dietary liver injury, Sellers & You (*J. Nutr. 44,* 513) explained the effect of the former as acting through suppression of synthesis of the thyroid hormone. Fatty livers produced by carbon tetrachloride injury were unaffected by supplementary methionine, choline, or cysteine whereas similar damage produced by selenium compounds was inhibited with methionine, but not with choline or cysteine (Sellers *et al.*—*Proc. Soc. Exptl. Biol. Med. 75,* 118).

The fatty acid storage in the lipides of the liver in the horse was studied, because the horse, unlike most of the other animals, readily stores linolenic acid (Bruce & Shorland—*Nature 167,* 236). Like most other animals, the horse stored a greater portion of the linolenic acid present in the glycerides than in the phospholipides; whereas the latter contained at least as much or more linoleic acid as the former.

Biological oxidation of lipides was studied both from the standpoint of the materials that affect the reaction and the mechanism of the reaction. Neoplastic livers (Baker & Meister—*J. Natl. Cancer Inst. 10,* 1191), and livers from rats given cystine (Levy—*Arch. sci. physiol. 4,* 241) were much less efficient for oxidizing fats than normal livers. The fatty oxidation decrease obtained in liver slices by maleic and malonic acids inhibited acetoacetic acid formation to a greater extent than carbon dioxide production (Geyer *et al.*—*J. Biol. Chem. 188,* 185). This work was done with radioactive labeled fatty acids and the analyses of the fragments were presented as evidence in support of the concept that two-carbon fragments only in the form of $-CH_2-CO-$ enter the tricarboxylic cycle. The observation that 2,4-dinitrophenol accelerated the respiration of rat-liver slices metabolizing octanoate and pyruvate, but inhibited phosphorylation of fructose was interpreted to suggest that oxidative phosphorylation was not involved in the oxidation of fatty acids (Fantl *et al.*—*Biochem. J. 48,* 96). However, Kennedy & Lehninger (*J. Biol. Chem. 190,* 361) reported that the oxidation of the fat to acetoacetate was possible only when primed by oxidation of hydrodiphosphopyridine nucleotide; whereas, priming the oxidation by succinate led to the formation of less acetoacetate and complete oxidation of the fatty acid carbon via the Krebs citric acid cycle. Chaikoff *et al.* (*Ibid.* 229) studied liver slice oxidation of tripalmitin in which either the carboxyl, C_5 or C_{11}, had been labeled with C^{14}. The labeled carbons were incorporated about equally into carbon dioxide and acetoacetate. In the latter the labeled carbon was in either the –CO or the –COOH group regardless of whether it came from either of the three labeled positions. These observations were discussed in connection with the mechanism for fatty oxidation. The observation by Bernhard & Gloor (*Helv. Physiol. et Pharmacol. Acta 9,* 17C) that labeled azelaic acid fed to animals was quantitatively excreted in urine was interpreted to prove that no azelaic acid was formed from oxidation of oleyl alcohol or oleic acid *in vivo.*

The mechanism of biological synthesis of lipides was studied by tracing labeled materials into them. Swan (*Arkiv Kemi 3,* 167) administered C^{14}-acetate to mice and found the C^{14} distributed uniformly at alternate carbon atoms in fatty acids in the liver. The C^{14} was found in the carboxyl group only when the mice were sacrificed in less than 30 minutes after injection of the C^{14} acetate. In similar work, Zabin (*J. Biol. Chem. 189,* 355) found more labeled carbon in the carboxyl group than in the remainder of the chain. The finding was interpreted as showing that chain elongation occurred by addition of two carbon particles to the carboxyl end of an acid. Beeckmanns & de Elliott (*Nature 167,* 200) used C^{14}-acetate administration technique to show that the lipide turnover in the liver was quite rapid. According to Brady & Gurin (*J. Biol. Chem. 189,* 371) labeled acetaldehyde was converted to fatty acids and cholesterol more rapidly than was acetate. In tests on utilization of other materials for lipide synthesis, isovalerate was as efficiently included in cholesterol as acetate (Zabin & Bloch—*J. Biol. Chem. 192,* 261, 267); pyruvic, butyric, hexanoic, and octanoic acids furnished carbon atoms for both fatty acids and cholesterol (Brady & Gurin—*Ibid. 186,* 461); ethanol was biologically converted to acetate before utilization in cholesterol synthesis (Curran & Rittenberg—*Ibid. 190,* 17); and tests with vinyl acetic acid, formic acid, crotonaldehyde, aldol, acetoin, and orsellinic acid, showed that none were important precursors of lipides (Brady *et al.*—*Ibid. 193,* 137). Aldol may be an exception for it could be converted into acetoacetate. Fatty acids isolated from yeast grown in the presence of C^{14}-labeled glycine had a high content of C^{14} distributed among the carbons of the chain (Weinhouse *et al.*—*J. Am. Chem. Soc. 73,* 1421). This work indicated that fatty acids were formed from glycine through the steps: serine, pyruvate, acetate, etc. A finding that the deuterium content of liver fat was lower than that of body fat after metabolism of deuterinated lauric acid, and reverse results with oleic acid was suggested to indicate that metabolisms of lauric and oleic acids were different (Jetton *et al.*—*Fed. Proc. 10,* 385).

Gray *et al.* (*Nature 167,* 954) identified acetic, propionic, butyric, isobutyric, valeric, and caproic acids in the rumen of sheep. When C^{14}-labeled acetic and propionic acids were added to rumen fermentations they were converted to butyric, valeric, caproic, and higher acids. Reiser (*Fed. Proc. 10,* 236) incubated

linolenic acid with rumen contents and found that hydrogenation had taken place. The action was due to the bacteria in rumen, for autoclaved controls underwent no hydrogenation.

Popjak's *et al.* (*Biochem. J. 48,* 411, 612; *49,* 610) studies on synthesis of milk fats indicated that they were formed from acetate in the udder and cholesterol was likewise synthesized in the udder. This was based on tests in which blood containing C^{14}-labeled acetate perfused in udders resulted in appearance of C^{14} containing fatty acids and cholesterol in udder lipides. All of the acids seemed to be synthesized in steps by an elongation of a shorter acid through addition of acetate. However, it was suggested that stearic and oleic acids might possibly come from blood. A study of venous and arterial cord blood of fetus by Ewerbeck & Levens (*Monatrschr. Kinderheilk. 99,* 297) indicated that the placenta did form serum albumin, but was only a permeable barrier to lipides. Thus it seemed that fetal fat came from the maternal blood. With laying hens the kind or amount of fatty acids in the ration did not affect the total lipides, phospholipides, neutral fat, or cholesterol composition of egg yolks (Reiser—*J. Nutr. 44,* 159). In this work hexanoic and pentanoic acid fed with a rigid fat-free diet did not appear in eggs; whereas, linoleic acid did. On fat-free diet oleic acid replaced the polyunsaturated acids in the eggs. The yeast, *Fusarium lycopersici* produced highly unsaturated fatty acids in neutral medium, but at pH of 3.3 the fat formed was more saturated (Kreitman *et al.*—*Nature 166,* 477).

Matsubara (*J. Biochem., Japan, 36,* 17) found normal fats in pigeons maintained on normal diet or one containing olive oil, but found linoleic and linolenic acids in the body lipides when linseed oil was added to the diet. In a short communication on seed fats Hilditch (*Nature 167,* 298) pointed out that each plant elaborates its specific mixture of acids, which could be slightly modified by environmental temperatures. Kartha's (*Separate Pub. by Maharaja's College 1,* 110 pp.; *2,* 111 pp.) communications contained a very comprehensive review on components of seed and animal fats as related to environment.

CHOLESTEROL METABOLISM. Various aspects of cholesterol metabolism were studied. Such aspects as were reported under fat metabolism have been discussed, in the preceding part of this section of the review, and those dealing with diseased conditions will be reviewed in the next part of this section.

A difference between the cholesterol metabolism in the guinea pig and the rabbit was evident because diets high in cholesterol considerably increased the fecal fatty acids in the former, while they increased only to a slight degree such fatty acids in the latter (Cook & Thomson—*Biochem. J. 49,* 72). Analyses of intestinal lymph for cholesterol esters indicated that increases occurred during fasting and during high cholesterol intake (Bollman & Flock—*Am. J. Physiol. 164,* 480). The tests indicated further that large amounts of cholesterol may be esterified by the intestinal mucosa. Since partial hepatectomy inhibited restoration of plasma cholesterol levels after drastic reduction of the plasma cholesterol, it was suggested that its maintenance in the plasma was a normal function of the liver (Friedman *et al.*—*Ibid.* 789). The livers of cholesterol fed, Eck fistula dogs contained 31% fat and 6.1% cholesterol, while those of normal dogs fed cholesterol averaged 8.3% fat and 61% cholesterol (Bailey—*Fed. Proc. 10,* 8). The results were interpreted as indicating that reducing the liver function in dogs decreased the ability to catabolize cholesterol and to synthesize phospholipides.

In healthy humans reduction of dietary fat produced a significant decrease in serum cholesterol concentration and a subsequent diet containing normal quantities of fat, but very little cholesterol resulted in increased serum cholesterol concentrations (Hildreth *et al.*—*Circulation 3,* 641). Weight reduction by rigid caloric restriction in obese individuals decreased plasma cholesterol levels (Walker—*Ibid.* 864).

Cholesterol content of the blood, liver, spleen, and kidney of mice on fat rich diets was considerably increased by feeding sodium or potassium salts of organic acids, or ammonium chloride (Schetiler—*Z. ges. inn. Med. 5,* 736). Induced hypertension resulted in tests in which large amounts (8%) of salt were put in drinking water of chicks, but this did not intensify spontaneous atherosclerosis (Stamler & Katz—*Circulation 3,* 859). Oils such as rape and turnip seed oils, when fed to rats, increased three to five fold the cholesterol content of adrenals (Carroll—*Fed. Proc. 10,* 25). The effect was attributed to the erucic acid in these oils.

LIPIDES UNDER DISEASE CONDITIONS. Earlier, and during last year, Gofman *et al.* (*J. Physical & Colloid Chem. 55,* 80; *Circulation 2,* 161, 205) associated atherosclerosis with the presence of giant molecules of lipides and lipoproteins in the blood. These molecules neither represent any part of the associated acute alimentary lipema nor could be predicted from the analytical total serum cholesterol levels. Other investigators have confirmed the presence of giant molecules in arterial diseases (Gertler *et al.*—*Circulation 2,* 830; Keys—*J. Am. Med. Assoc. 147,* 1514). More striking giant molecules of the type reported by Gofman were centrifuged out of blood of laying hens by Feldman (*Science 113,* 697). Zinn & Griffith (*Am. J. Med. Sci. 220,* 597) attributed the presence of large particles (chylomicrons) in the blood of atherosclerotic and diabetic patients to abnormality in fat transport rather than to absorption, because these were present during fasting. Necheles (*Am. J. Digestive Des. 18,* 229) reported a depression of chylomicrons in aged subjects by using a lipase preparation and he also recorded that "Tween 80" raised them in young subjects. This work was done with the thought that the chylomicrons might cause arteriosclerosis. Considerable reductions of giant particles in blood and increase in the translucence of plasma were obtained with heparin by Block *et al.* (*Circulation 4,* 674), Swank *et al.* (*Am. J. Physiol. 164,* 798), and Graham *et al.* (*Circulation 4,* 666). The latter reduced atherosclerosis in rabbits by administration of heparin; and similarly, in 55 of 59 patients with angina pectoris, a redistribution of lipoproteins of the blood and a reduction of symptoms was found.

In a polemic reply to a record of cholesterol and vitamin E content of foods "published for nutritionists and physicians," Stare & Mann (*J. Am. Oil Chemists' Soc. 28,* 232) reviewed literature showing that vitamin E had no function in human health or disease and cited references indicating no relation between dietary cholesterol and coronary and vascular diseases.

Atherosclerosis and arteriosclerosis were also studied experimentally on animals. No inhibition in the lesions in cholesterol fed rabbits was obtained by the concurrent feeding of inositol, methionine and choline (Capretti & Paglia—*Giorn. clin. med., Parma, 31,* 1120, Marfori *et al.*—*Sperimentale 101,* 132). Pyridoxine supplementation of the diet accelerated production of the arteriosclerosis (Capretti & Magani—*Giorn. clin. med., Parma 31,* 989). Atherosclerotic lesions were induced in rabbits by intravascular injections of cholesterol, colloidal graphite or sodium stearate (Pollak & Wadler—*J. Gerontol. 6,* 217). These results were interpreted to indicate that the lesions were due to the result of precipitation of micelles upon the vascular intima. A comprehensive histological study of the vascular lesions in cholesterol fed rabbits was published by Kelley *et al.* (*Fed. Proc. 10,* 262). Other abnormalities reported found in cholesterol fed rabbits were an increase in lymphocytes and neutrophils (Altschul & Martin—*Arch. Path. 51,* 617), and development of amyloidosis or paramyloidosis (Hoffman *et al.*—*Proc. Soc. Exptl. Biol. & Med. 78,* 37).

The similar nature of a lesion in chickens (cholesterol induced) with arteriosclerosis in man was shown (Stamler & Katz—*Circulation 2,* 705). These workers suggested that the dietary cholesterol may be involved in human atherosclerosis. In a study of age of chicks in relation to susceptibility to atheromatosis Rodbard *et al.* (*Circulation 3,* 867) found that resistance to the vascular lesions disappeared soon after eight weeks of age. According to Horlick & Katz (*Am. Heart J. 38,* 336) atheromatosis developed in chickens in direct relation to the amount of cholesterol in the diet up to about 0.5%, and above this amount no further acceleration, or increase of atherosclerosis was induced. Low fat diets inhibited the development of lesions in the chickens (Horlick *et al.*—*Ibid.* 472), administration of stilbestrol, like cholesterol, induced them (Stamler *et al.*—*Circulation 2,* 722); whereas, lipotropic factors such as choline and inositol failed to exert a prophylactic effect (*Ibid.* 714).

Production of arteriosclerosis in cholesterol fed dogs was accelerated by simultaneous administration of thiouracil (Steiner *et al.*—*Am. Heart J. 38,* 34). As already reported for other species, choline had no effect on an arteriosclerosis-producing regimen in dogs (Davidson *et al.*—*Circulation 3,* 332). The arterial lesions were produced also in dogs by injections of allylamine (McCormick *et al.*—*Feder. Proc. 10,* 363).

Abnormal fat metabolism was associated with several diseased states. An increase in neutral fats of the blood, with a moderate increase in cholesterol and a low cholesterol ester: free cholesterol ratios were found in two patients with retinal lipema and colicky abdominal pain (Poulsen—*Acta Med. Scand 138,* 413). In these, other pathology also indicated a disturbance in fat metabolism. Swank (*Am. J. Med. Sci. 220,* 421) found that high fat diets accelerate multiple sclerosis, although they might not be the cause. High fat diets also accelerated the onset of oestoarthritis of aging mice (Silberberg & Silber-

berg—*Arch. Path. 50,* 828). Sixty cubic centimeters of 20% cream fed to each of 100 patients decreased the clotting time of their blood an average of 33.7% at 30 minutes after feeding (Waldron *et al.—Gastroenterology 17,* 360). The abnormal fat deposits in gargoylism contained abnormal ratios of chloroform and chloroform-methanol insoluble fractions (Brante—*Fette u. Seifen 53,* 457). Six patients with steatorrhea and anemia developed improved fat absorption after intravenous administration of iron. Ketosis during fat metabolism was considerably more intense in diabetics than in normal persons (Müller—*Deut. Arch. klin. Med. 196,* 135). In this work the blood sugar curve rose and fell with the ketone curve. By means of radioactive labeled fats, Chaikoff & Weiman (*J. Biol. Chem. 192,* 453) demonstrated that alloxan-diabetic rats converted more of the fat to glucose than normal rats. Blood cholesterol was high in nephrosis and usually low in hyperthyroidism (Schettler & Lukas—*Z. ges. inn. Med. 6,* 14). X-irradiation produced increases in plasma phospholipides in dogs, guinea pigs, rabbits, and mice (Neve & Entenman—*Fed. Proc. 10,* 367). In some young children the presence of starch and dextrin in the diet reduced fat absorption (Scheldon—*Arch. Dis. Childhood 24,* 81; Scheldon & MacMahon—*Ibid.* 245).

LIPIDES IN RELATION TO MICROBIOLOGY AND ENZYMES. Some reports dealt with characterization of the inhibitory action of some fatty acids on microbiological processes. In work on bacteriostatic effect of saturated fatty acids all C_4 through C_{18} acids were effective but the effect decreased with each increase of carbon atoms by twos from C_8 through C_{12} (Hassinen—*Arch. Biochem. & Biophys. 31,* 183). This correlated somewhat with the water solubility of fatty acids. In work on the effect of pH on this action for lauric acid, the maximum bacteriostasis on *Escherichia coli* was at pH 7.6, on *Staphylococcus aureus* at about pH 6, and on *E. typhi* at pH 9 (Dervichian & Mousset—*Ann. inst. Pasteur 77,* 703). The inhibitory action on the growth of *Streptobacillus moniliforms* by sodium oleate was neutralized by addition of bovine serum, while animal lecithin had no effect on the action (Dumoff & Duffy—*Proc. Soc. Exptl. Biol. Med. 77,* 1). The fatty acid fractions obtained from a commercial peptone inhibited the sporulation of aerobic bacilli in a synthetic medium non-inhibitory to growth (Hardwick *et al.—J. Bact. 61,* 145).

Klosa (*Pharmazie 6,* 63) prepared extracts of wheat which contained fatty material that inhibited germination of seeds. One alcohol extract of the wheat inhibited growth in rats.

Other investigators indicated the need of fat for microbiological action. Thus, oleic acid was required for the optimal activity of both formic hydrogenlyase and formic dehydrogenase enzyme systems of *Escherichia coli* (Lichstein & Boyd—*J. Bact. 62,* 415). Different lactobacilli required different amounts of oleate for growth; some grew when either biotin or oleic acid was supplied but the metabolism with regard to behavior of carbon dioxide and aspartic acid in the systems varied with the type of lactobacillus (Broquist & Snell—*J. Biol. Chem. 188,* 431).

Silliker *et al.* (*J. Bact. 61,* 653, 661) developed data on the oxidation of fatty acids by bacteria. All strains of aerobic bacteria tested showed some ability to oxidize the C_9 and C_{10} acids; and glucose-grown cells of *Serratia marcescens* oxidized all saturated acids from C_2 through C_5. The data were consistent with either multiple alternate oxidation or a somewhat modified form of β-oxidation. Oxalic and other acids were formed by *Aspergillus niger* from such fats as olive oil and sunflower oil (Seliber & Golovkina—*Mikrobiologiya 20,* 20).

Rose (*J. Am. Oil Chemists' Soc. 28,* 47) standardized a procedure for preparing active lipase cream from castor beans so that the lipase emulsions had reproducible activity. This lipase was inhibited by soluble synthetic cephalin, egg phosphatide, and salmon egg phosphatide, but not with soybean phosphatide. Nevgi & Ramakrishnan (*J. Indian Chem. Soc. 27,* 255, 260, 263, 264, 265, 331, 333, 334, 337) prepared castor bean lipase in dried form by drying with acetone. The capacities of the preparations to synthesize esters from propionic, butyric, and other acids with several aromatic and aliphatic alcohols were recorded. In addition, hydrolytic action on various esters and the effect of inorganic salts on the reaction were studied. In some cases the lipase preparation from castor beans was compared with lipase concentrates from sesame and safflower. Huang (*J. Chinese Chem. Soc. 18,* 95) hydrolyzed tung oil by mixing with ground castor bean, water, a little acid and manganese sulfate. This mixture was then pyrolyzed at 450°C. to yield motor fuel, gas oil, and lubricant oil. Schoheyder & Volqvartz (*Biochem. & Biophys. Acta 6,* 147) determined the rate at which pancreatic lipase hydrolized acetone-l-caprylylglycerol to *dl*-l-caprylylglycerol. This hydrolysis was explained by an integral equation.

Characteristics and Composition

The literature limited to analyses of the raw materials and the extracted fats is being reviewed in this division. Some other analytic and test information such as that on soap and rancidity seemed more pertinent to other divisions of this communication and is found in other divisions.

A few reports were too comprehensive for classifying in subsections that follow. One by Kaufmann & Baltes (*Fette u. Seifen 53,* 445) contained German standard methods for determining density, flow point, drop point, cold stability, color, refraction, flash point, fire point, sieve analysis, viscosity, surface tension, and pH for the fat and detergent industries. British standards and methods for testing were issued for crude whale oil (*Brit. Standard 836*), cod oil for sulfonation (*Ibid. 868*), and sperm oil (*Ibid. 997*). Micromethods for the fat field were reviewed (Gorbach—*Fette u. Seifen 53,* 3).

ANALYSES OF FAT SOURCES. Some investigators related environment or characteristics of the raw material to the amount or character of the fat. Edward's *et al.* (*J. Sci. Food & Agr. 2,* 429, 431, 472) studies showed that linseed oils grown in New Zealand had higher iodine value and linoleic acid content than those grown in India. A good linolenic acid concentrate could be segregated from the fatty acids of the New Zealand oil by liquid-liquid extraction with pairs of solvents. Their work on Australian sunflower seed oils indicated that those grown in the Northern territory were low in linoleic content and should be exceptionally suitable for edible purposes. In studies on 12 varieties of flax grain those used for textile fibers had less oil and more linamarin (precursor of hydrogen cyanide) content than the flax grain used in producing linseed oil (Andre *et al.—Compt. rend. 231,* 590). The rough rice of eight varieties grown in three United States locations each showed variations in milling yields and lipide contents of bran, true pericarp, and bran fractions which were attributed to the influence of variety and environment of growth (Jurgens & Hoffpauir—*J. Am. Oil Chemists' Soc. 28,* 23). The oil of wheat milling by-products and its refined products were characterized, but quality was not satisfactory for edible purposes (Anselmi—*Rend. est. super. sanita 7,* 466). Hydrogenation of the oil was unsuccessful. Similar work on raw, refined, and hydrogenated safflower seed oils indicated that this oil also was not suitable for edible purposes (Soltoft & Dollear—*J. Am. Oil Chemists' Soc. 28,* 335). Classen *et al.* (*Agron. J. 42,* 478) associated per cent of oil with size of safflower seed, and suggested that visual examination could be used in selection of high oil seed. Cattaneo's *et al's.* (*Anales asoc. quim. argentina 38,* 268) analytical records of ripening olives showed that free fatty acids and unsaponifiable matter decreased on ripening and increased on overripening; during this period oleic acid decreased and linoleic acid increased. The oil content of corn increased in proportion to the size of the germ (Miller & Brimhall—*Agron. J. 43,* 305).

The fat tissues of the intestinal lining, kidneys, and pancreas of pigs were analyzed and chemical, organoleptic, and physical characters recorded (Janicki—*Przemysl Rolny i Spoz. 4,* 242). Brown *et al.* (*J. Animal Sci. 10,* 97) correlated the specific gravity as a measure of the fat content and the yield of primal cuts on 66 pork carcasses. The oil yields and characteristics of the oil were recorded for 30 species of Philippine sharks and rays (Hamm—*Fish & Wildlife Service Res. Dept. No. 23,* 1).

American Oil Chemists' Society collaborative work on seed analyses indicated that sliced peanut kernels for analysis should be mixed with a "Velocity Mixer" (Law *et al.—J. Am. Oil Chemists' Soc. 28,* 380). Curves and tables of weight loss with oven drying at 110° and 130°, and yields of oil with varying extraction time were determined for sesame seed as basic information for analytical procedures (Stark & Hoffpauir—*Ibid.* 516). These showed that combination moisture and volatile matter determinations should be by drying a five gram sample two hours at 130° in a forced draft oven; and that the American Oil Chemists' Society method for oil in cottonseed was suitable for oil in the sesame seed. A special complete moisture determination apparatus which used infrared rays for drying, when standardized for soybean analyses, gave moisture values 0.5-1.0% lower than standard procedure on various mustard seeds and peanuts (Francois & Sergent—*Bull. mens. ITERG 4,* 456). The equilibrium moisture content of tung fruit and its components for different humidities were determined (Holmes *et al.—J. Am. Oil Chemists' Soc. 28,* 218). As most useful figures from these data the writer records that at 75.4% relative humidity the results were: ground outer hulls 19.6, ground inner hulls 14.8, ground shell 12.4, whole tung kernels 6.1, ground press cake 10.3, whole seeds 8.7, and whole fruit 12.4% moisture. Moisture determinations on butter indicated that there was no appreciable differences between hand and mechanical shaking

Oil or Fat Source	% Oil or Fat	Specific Gravity	Refr. Index	Acid No. or (% Free Fatty Acids)	Sapon. No.	Iodine No.	(SCN No.)	Acetyl or (OH No.)	R.-M. No.	Polenski No.	% Unsaponifiable	Solidification Point or (Melting Point)	Diene No.
Acacia cyclops funicle[1]	40.6	0.917^{25}	1.4691^{25}	3.25	188.5	71.9					2.78		
Acacia cyclops seed[1]	9.95	0.922^{25}	1.4746^{25}	2.26	186.3	136.9					0.99		
Broad beans[2] *Vicia faba*	8.06	0.9385^{18}	1.484^{20}	18.2	190.2	118.9		9.6	0.57		1.98	—4	
Buffalo gourd seed[3] *Cucurbita foetidissima*	24.3		1.4735^{25}	(2.0)	191.8	136.1		(9.8)	0	0.3	1.53		
Buckwheat leaf meal[4]	5.8		$1.0823^{25/25}$	21.3	187.8	123.9		(7.6)	7.0	0.4	14.9		
Cephalaria syria[5]		0.9263^{20}	1.4716^{20}	6.0	194.9	85.8	65.0	(20.0)	0.25	0.15	1.32	—15	
Curupira chestnut[7]	63.6	0.9068^{15}	1.4727^{15}	(30.9)	167.5	79.0		18.6			2.04	(35)	
Elaeis melanococca fruit[8]		$0.8978^{40/4}$	1.4583^{45}	(1.2)	195.2	65.2					1.0	41.0	
Elm (wild) seed[9] *Ulmus manshurica*	28.8	$0.9525^{20/4}$	1.4552^{20}		251.4	22.5							
Gokuru seed[11] *Xanthium strimarium*	31.5	$0.9167^{25/25}$			195.5	122.5		2.0	0.5	0.12	0.37		
Lupine seed[13] *Lupinus albus*	9.6	0.9229^{15}	1.4758^{19}	5.9	182.9	107.6		(6.3)			3.9	—18	
Lupinus luteus	5.7	0.9193^{15}	1.4772^{19}	20.0	185.0	115.6		(7.8)			4.0	—16.5	
Lupinus augustifolius	4.5	0.9463^{15}	1.4790^{19}	28.0	183.0	104.0		(9.9)			8.0	—17	
Mussel flesh[14] *Mytilus edulis*	6-7	0.9747^{20}	1.4780^{40}	17.6	156.2	131.6			6.9	0.4	16.7	(—2.4)	29.97
Osageorange fruit[16] *Maclura pomifera*		0.9793^{25}	1.4981^{20}		150.8	100.8	83.8		1.0	0.06		4	
Panicum crusgalli grain[17]	5.0-6.5	0.904^{20}	1.475^{15}	16.9	194.7	122.1		17.0	1.45	0.79			
Papri tree seed kernel[18] *Haloptela integrifolia*	53.2	$0.9001^{30/30}$	1.4580^{40}	1.6	203.7	52.8			0.27	0.25		13.5	
Queen's delight shrub seed[19] *Stillingia sylvatica*	30-33	0.9263^{25}	1.4833^{25}	16.1	189.2	189.9		37.5	1.4	0.11	0.76		
Rohini seed[21] *Mallotus phillipinensis*		$0.9444^{30/30}$		5.2	190.7	166.4					2.3		49.5
Sebastiana lingustrina shrub seed[22]	37	0.9347^{25}	1.4850^{20}	3.6	204.9	191.0		15.4	0.74	0.14	0.76		

FATTY 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	
Acacia cyclops funicle[1]	0.3	21.6	2.7	61.7	3.1	0.3	C_{12} 0.5, C_{20} 0.4, C_{14} (−2H) 0.9, C_{16} (−2H) 7.3, C_{20} (Unsatd.) 1.2
Acacia cyclops seed[1]	0.7	5.6	0.6	10.1	67.7	0.8	C_{12} 0.1, C_{20} 0.8, C_{20-24} 2.9, C_{16} (−2H) 8.6, C_{20} (Unsatd.) 2.1
Broad beans[2] *Vicia faba*		1.8	7.5	41.7	27.3	11.6	C_{20} 1.0, C_{22} 0.1
Buffalo gourd seed[3] *Cucurbita foetidissima*	0.2	6.1	2.2	23.0	65.3		C_{20} and C_{20}+ 0.55, dienes 0.37, trienes 0.66, tetraenes 0.2
Cucumis utilissimus seed[6]	0.5	12.8	7.9	27.5	50.4		C_{12} 0.12
Curupira chestnut[7]		8.0		85.4	0.8	0.6	C_{20} 6.3
Elaeis melanococca fruit[8]	1.0	32.6	4.7	47.5	12.0	0.8	C_{20} 0.5, C_{16} (−2H) 0.9
Elm (wild) seed[9] *Ulmus manshurica*	1-3	12-21		5-8			C_8 2-11, C_{10} 55-59, C_{12} 2-8
Emblica officinalis seed[10]	1.0	3.0	2.2	28.4	44.0	8.8	
Lophira alata seed[12]	0.3	28.8		14.0	11.5		C_{22} 34.3, C_{24} 6.8, C_{22} (−2H) 4.3
Lophira procera seed[12]	0.5	36.6		12.0	26.3		C_{22} 20.2, C_{24} 1.3, C_{22} (−2H) 3.1
Lupine seed[13] *Lupinus albus*	———	10.0	———	60.6	19.9	2.5	C_{22} (−2H) 6.8
Lupinus luteus	———	9.0	———	39.1	45.0	0.9	C_{22} (−2H) 6.0
Lupinus augustifolius	———	10.0	———	47.5	33.7	1.8	C_{22} (−2H) 7.0
Mango kernel[15]	5.1	11.2	31.1	43.8	4.1		C_{10} 0.15, C_{12} 2.7, C_{20} 1.7
Pentaclethra macrophylla tree seed[12]		4.0	2.1	18.7	54.5		C_{20} 3.8, C_{22} 5.9, C_{24} 11.0
Pentaclethra Eetveldeana tree seed[12]		3.7	4.8	53.3	16.1		C_{20} 5.4, C_{22} 13.9, C_{24} 2.8
Queen's delight shrub seed[19] *Stillingia sylvatica*		3.4	1.8	18.5	25.0	48.6	
Rhus succedanea tree seed[20]		25.4		46.8	27.8		
Sebastiana lingustrina shrub seed[22]	———	9.7	———	11.9	38.9	34.9	C_{12} (?) 4.6
Singkamas (Philippine) seed[23] *Pachyrrhizus erosus*	———	38.5	———	32.9	26.4		

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methods in sampling (Weber—*J. Assoc. Off. Agr. Chemists' 33,* 544). A newly described method for determination of moisture in butter comprised centrifuging a 20-gram sample with fat solvent in a special tube in which the moisture was measured in a graduated portion (Pozzi-Escot—*Lait 30,* 225).

The work on determination of fat in materials dealt with development of rapid plant control methods, and ascertaining and improving the accuracy of common procedures. The rapid method in which the material was extracted in a Waring Blendor was adapted to determination of oil in ground tung fruit (Gilbert & Gropp—*J. Am. Oil Chemists' Soc. 28,* 413). A control method for estimating industrial yields from olives comprised homogenizing the olive pulp with salt solution, centrifuging to cause separation, and measuring the oil that separated (Rousseau—*Inds. Agr. et aliment, Paris, 67,* 145).

Several analysts pointed out that to obtain complete extraction of fat from foods, fish, feeds, and the like, the material must first be treated with hydrochloric acid (Stoldt—*Deut. Lebensm. Rundschau 47,* 13; Seeler & Dietrich—*Landw. Forsch 3,* 43; Morgan & Rawling—*Analyst 76,* 161). To increase the rapidity of oil extraction from fish, Stansby (*J. Assoc. Off. Agr. Chem. 34,* 549) tried a pre-refluxing of the material with acetone and with acetone and acid. With acetone alone there was a continual increase of oil extracted for 16 hours, while the presence of one per cent hydrochloric acid considerably reduced extraction time. In tests on the efficiency of various fat extraction methods on eggs, Hadorn & Jungkunz (*Z. Lebensm.-Untersuch. u. -Forsch. 93,* 277) found that petroleum ether and ether quantitatively extracted free fatty acids, glycerides, and cholesterol and 72-78% of the lecithin; extraction with ether, then with alcohol recovered 90% of the lecithin; a 1:1 mixture of alcohol:benzene gave better extraction; but most quantitative extraction was with acid digestion followed by solvent extraction. Digestion with the acid decomposed lecithin into fatty acids, phosphoric acid and choline; affected cholesterol; but did not attack the glycerides. Rapidity could be gained with the American Standard ether extraction method by faster flow of solvent (Hoffmann—*J. Assoc. Off. Agr. Chemists' 34,* 558); however, results were usually slightly low.

New digestion reagents were used in the butyrometer method for determining fat in milk products. A 60% solution of sodium salicylate used with one cubic centimeter of iso-amyl alcohol was said to permit butyrometric fat determinations without the aid of a centrifuge to separate the fat (Gentilini—*Latte 24,* 219). In another method the milk product was first treated with a nonionic detergent and thereafter with an anionic detergent to obtain the fat separation (Gershenfeld & Rosenthal—*J. Milk Food Technol. 14,* 17).

Two fat determinations issued for use in biochemical work were based on extraction, saponification, hydrolysis; and extraction and determination of the fatty acids in the extract by titration (Page & Michaud—*Can. J. Med. Sci. 29,* 239; Müller —*Proc. Koninkl. Nederland. Akad. 54C,* 153).

In a new analytical method of extracting oil from pigment pastes, a mixture of methanol and benzene, or methanol and acetone was used as the solvent so that pigment would not be carried through the filter (Lenz—*Farbe u. Lack 56,* 538). A method for determining oil in olive-oil tank sediment comprised a specific procedure for shaking a sample with carbon tetrachloride and determining the density of the fat solution (Casares-Bescansa—*Anales bromatol., Madrid, 2,* 375). Equations were developed for the calculation of the fat in the sample. The fatty material in refining foots was ascertained by extraction of a dioxane:water mixture, and titration of soaps with hydrochloric acid; and determining total fat on another portion by extraction with petroleum ether (Wolff—*Oleagineux 5,* 646). Stoy (*Deut. Lebensm. Rundschau 46,* 261) cautioned that when analytically extracting fats from water-in-oil emulsions with trichloroethylene, the emulsion should be first boiled with a mixture of hydrochloric acid and alcohol. A special combination beaker-glass and separatory funnel was patented as apparatus for analysis of fat or oil-bearing material (Hustinx—*U. S. 2,539,082*).

CHEMICAL CHARACTERISTICS. A simple glass apparatus was designed for attachment to neutralizers in oil mills, which permitted rapid determination of acidity of the oil during processing (Pepe—*Olearia 4,* 297). A new micromethod for determining acidity of oils in the paint industry was a modification of the Gorbach procedure (Lackner & Flaschka—*Fette u. Seifen 52,* 737). A comparison of acetyl values determined by the British Standard Institute and by the Association of Official Agricultural Chemists' methods showed that both gave similar results (Riley—*Analyst 76,* 40). In a new simple and rapid saponification value determination potassium hydroxide dissolved in a mixture of glycol monoethyl ether and xylene was used as the saponifying agent (Hahn—*Anal. chim. acta 4,* 577). In a microprocedure, the potassium hydroxide was in ethylene glycol as the solvent (Vanetten—*Anal. Chem. 23,* 1677). Saponification value determination experience with lespedeza seed oil showed that exposure to air caused a decrease in the value from 189 to a low of 163 in 45 hours and then an increase to 183 as time of exposure increased (Wiley & Cagle—*J. Am. Oil Chemists' Soc. 28,* 89). In the determination of water-insoluble fatty acids in butter, the addition of 0.5 ml. of normal alkali in excess of that required to neutralize the sample did not significantly affect the results (Hillig—*J. Assoc. Off. Agr. Chemists' 34,* 782).

The methods for characterizing unsaturation of fats and oils were studied principally to increase rapidity of the tests. Rapidity of the Rosenmund-Kuhnhenn iodine value determination was increased by using a smaller sample and decreasing reaction time (Drozdov & Materanskaya—*Myasnaya Ind. S.S.S.R. 22,* No. 4, 31). The Wijs method was accelerated by catalization of the reaction with mercuric acetate and diffused natural light (Neto—*Anais assoc. quim. Brasil 9,* 92). Lopes (*J. Am. Oil Chemists' Soc. 28,* 390) cautioned that the ratios of the halogens in Hanus and Wijs reagents should be unity for most accurate iodine results. He issued analytical methods for determining the ratio of the halogens. Reviews were published on the Woburn method applications to conjugated oils (von Mikusch—*Farbe u. Lack 56,* 341) and on micromethods (Ruziczka—*Mikrochem. ver. Mikrochim. Acta 36/37,* 924). In a direct titration iodine value method the oil was dissolved in an acetic acid solution of sodium acetate and titrated potentiometrically while hot with 0.1 *N* bromine in acetic acid using a bright platinum electrode and calomel reference electrode (Tomicek & Dolezal—*Acta Pharm. Intern. 1,* 31). The results were generally lower by this method than by several standard methods. The Hanus method was adapted to paper chromatography by using a radio active reagent, washing away the unreacted reagent, and measuring radiation (Kaufmann & Budwig—*Fette u. Seifen 53,* 253).

Vandenheuvel (*J. Am. Oil Chemists' Soc. 28,* 512) working with pure fatty material demonstrated that unsaturation as measured by hydrogen was related to refractivity. A straight line related the iodine value and aniline point of fats and oils whose saponification numbers were between 190 and 200 (van Voorst—*Chem. Weekblad 47,* 333). These relationships should be useful in control analytical work.

The analytical methods for determination of hydroxyl groups, dienic content, degree of unsaturation, linoleic acid, and isolinoleic acid in the castor oil dehydration industry were discussed in detail (Desnuelle & Massoni—*Bull. soc. chim. France 1950,* 1180). Methods for determination of hexabromide number were reviewed (Balbi—*Ind. vernice, Milan 4,* 235).

PHYSICAL TESTS. Some physical tests were used to measure qualities useful to the intended application of the oil or fat. New apparatus was designed for determining cutability and pliability of butter (Mohr *et al.*—*Fette u. Seifen 53,* 129). Grummit *et al.* (*J. Am. Oil Chemists' Soc. 28,* 141) standardized a procedure for determining acetone number of polymerized oils, and related this value to the kind of oil, its viscosity, amount of thermal and oxidative processing, and the addition of modifying agents such as maleic anhydride. In comparing red oils employed for oiling of wool, Francois & Juillard (*Bull. mens. ITERG 5,* 293) oiled and deoiled leas and evaluated them with regard to yellowing, oil retention and effect on distribution of dyes. Schlenker (*Fette u. Seifen 53,* 191) recorded data on the relation of the structure of glycerides to melting point and consistency of various fats and how these were altered by interesterification. The data indicated the effect of trisaturated, mixed, and triunsaturated glycerides in mixtures and could serve as fundamental information for blending fats and oils. In an investigation on determining the index of refraction of wool fat considerable variations occurred when samples had different thermal history; i.e., brought up *versus* brought down to temperature (Roussouw—*Onderstepoort J. Vet. Sci. Animal Ind. 24,* 355). A temperature of 50°C. was recommended for the test to avoid the effect of solid components on the results.

A conversion chart was developed to aid in converting spectrophotometer fatty oil color readings to American Oil Chemists' Society photometric color values (Freyer & Shelburne—*J. Am. Oil Chemists' Soc. 28,* 239).

The melting points and microscopic characteristics for the three polymorphic forms of cacao butter (Vaeck—*Rev. intern. chocolaterie 1951,* 12 pp.), tripalmitin (Ravich & Voronova—*Doklady Akad. Nauk S.S.S.R. 77,* 1035, and ricinoleic acid (Hawke—*J. S. African Chem. Inst. 2,* 125) were determined. A study of polymorphism by dilatometric measurements showed

that the melting dilations of stearic acid, monostearin, and tristearin were in direct proportion to the mole percentage of stearyl group in each compound (Singleton & Vicknair—*J. Am. Oil Chemists' Soc. 28,* 342). The polymorphic characteristics of symmetrical mixed saturated triglycerides (Crowe & Smyth—*J. Am. Chem. Soc. 73,* 2040), glycerides in which the 2-position was occupied by a very short chain (Jackson *et al.*—*Ibid.* 4280), and monounsaturated disaturated glycerides (Lutton—*Ibid.* 5595) were investigated, respectively, with regard to dielectric behavior, new forms caused by short acids, and effect of production from solvent and melt. The unit cell and spacings in the α-form of lauric acid and β-form of trilaurin were determined by x-ray diffraction (Vand *et al.*—*Acta Cryst. 4,* 324, 465). The former was monoclinic and the latter triclinic. Huber (*J. Am. Chem. Soc. 73,* 2730) reported that both *cis*- and *trans*-octadecenoic acids could be divided into two groups according to their melting behavior, and a plot of the melting points of either the *cis*- or *trans*-members against position of the double bond resulted in a zigzag line because of alternation of the melting point. Lutton *et al.* (*Ibid.* 2733, 5206) observed similar alternation of physical properties in x-ray diffraction studies. Those with the double bond beginning at an odd-numbered carbon had larger spacings and lower melting points. Both low-melting and high-melting series were observed also in dihydroxystearic acids.

Molecular layers of fatty acids were observed for various purposes. Cathode ray diffractions of unimolecular layers of stearic acid on water indicated that the structure belonged to the rhombic system (Ueda & Takagi—*Science, Japan, 12,* 217). A similar study on the effect of temperature indicated that the films of stearic acid on metals become disoriented at about 120° (Menter—*Research, London, 3,* 381). The sorptions of hexane (Hayes & Dean—*J. Am. Chem. Soc. 73,* 5584) and several longer hydrocarbons (Sobotka *et al.*—*J. Colloid Sci. 5,* 567, 581) by monolayers of stearic acid were measured. In this work, when the data on stearic acid was compared with a branched chain acid, a difference in shape and the character of the films, straight *versus* branched chain, was evident.

The surface tension of saturated C_5-C_{18} acid methyl esters increased linearly with the weight of the esters (Nevin *et al.*—*J. Am. Oil Chemists' Soc. 28,* 325). Ultrasonic velocity was not dispersed in liquid C_{14}-C_{18} saturated fatty acids except near their melting points (Wada *et al.*—*J. Phys. Soc. Japan 5,* 345).

COMPOSITION. Many methods and aids for the determination of composition of fats were investigated. A spinning-band column for the analytical vacuum fractionation of fatty-acid esters was developed (Williamson—*J. Applied Chem. 1,* 33). The Hilditch ester fractionation analysis method with slight modifications such as increasing the size of samples, crystallization of steam non-volatile acids from acetone instead of separation by lead-salt alcohol process, and improvements in the still permitted sufficient accuracy to detect seasonal variations in composition of butterfat (Hansen—*J. Am. Oil Chemists' Soc. 28,* 375).

Pertinent solidification point diagrams of binary mixtures for odd-odd acids C_{11} to C_{25} were constructed (Schuette *et al.*—*Ibid.* 361). Clearly defined inflections which characterize diagrams of straight-chain even numbered carbon acids were not evident with the "odd" acids. The potential application of the diagrams were for the analysis of higher alcohols oxidized to the corresponding acids. Freezing point diagrams of systems of C_{14}-C_{16}-C_{18} saturated acids; palmitic-stearic-oleic acids, and stearic-oleic-linoleic acids were developed (Paquot *et al.*—*Bull. soc. chim. France 1950,* 837; *1951,* 350, 353). The application of these with iodine index were discussed with respect to composition analyses. Based on the fact that urea and thiourea form crystalline addition products of fatty acids, but not with triglycerides and oxidized acids, Martinez-Moreno *et al.* (*Anales real soc. espan. fis. y. quim. 47B,* 229) suggested the following separations were possible by this means: triglycerides from fatty acids, oxidized from nonoxidized fatty acids, chaulmoogric from straight chain acids, and fatty acids from tall oil. Schulte & Kirschner (*Fette u. Seifen 53,* 456) determined the kinetics of the esterification reaction of straight and branch chain C_2-C_{24} acids to investigate esterification as a means of characterizing fatty acids. The side chains reduced the speed of esterification but this characteristic was not suitable for distinguishing various fatty acids. Investigation of the determination of unsaturated component of fats by bromination and fractional crystallization showed that considerable errors occur because of the changing solubility of the bromide of one component in the presence of various others (Mhaskar *et al.*—*J. Univ. Bombay 18,* Pt. 5A, No. 27, 28).

The analytical degradation of fatty acids by oxidation was used to confirm the structure of *n*-octadecenoic acids prepared for x-ray and melting point characterization (Stewart *et al.*—*J. Am. Chem. Soc. 73,* 5903), and for ascertaining the structure and course of oxidation of linoleic and linolenic methyl esters (Toyamo & Matsymoto—*J. Chem. Soc., Japan, Ind. Sec., 54,* 383, 523). Kartha *et al.* (*Current Sci., India, 18,* 8; Separate, Maharaja College *1*) considered the permanganate oxidation method of Hilditch and the Stainsby modification of this for the determination of linoleic acid in glycerides inaccurate. He believed the inaccuracy was due to hydrolysis during oxidation or alkali titration and modified the methods in a manner so that oxidation was done at environments of lower acid concentration.

Much fat analysis investigation was done with chromatographic technique. The application of this technique was reviewed by Devauchelle (*Bull. mens. ITERG 5,* 99) and Williams (Discussions *Faraday Soc. 1949, No. 7,* 264). In combination oxidation-chromotographic analyses methods of unsaturated acids the material was oxidized with permanganate according to Hilditch and the fragments separated and determined chromatographically (Schuette & Nogare—*J. Am. Oil Chemists' Soc. 28,* 229; Begemann *et al.*—*Rec. trav. chim. 69,* 439). Liberman's *et al.* (*Fed. Proc. 10,* 216) modification of Hiscox chromatographic procedure for intermediate fatty acids made use of paper impregnated with paraffin as the stationary phase; employed C_5-C_{10} alcohols as the liquid phase; and substituted methyl amine as the volatile base used with the fatty acids. Fairbairn & Harpur (*Nature 166,* 789) in separations of C_2-C_8 fatty acids used two columns impregnated with different dyes which would indicate the bands. These bands could be removed and titrated. Similarly, Masuyama (*J. Chem. Soc. Japan. Chem. Sect. 71,* 402) converted the fatty acids to colored derivatives by reaction with 4-phenylazophenacyl bromide so that their bands in chromatographs would be visible. Yukawa & Inoue (*J. Agr. Chem. Soc., Japan, 18,* 415, 875, *1950,* 294) obtained improved chromatographic and crystallization separation of mixed fatty acids by separating as the hydroxamic acid derivatives. Herb *et al.* (*J. Am. Oil Chemists' Soc. 28,* 505) combined chromatographic and distillation techniques to isolate arachidonic and docosapentaenic acids from beef adrenal lipides.

Much fundamental data was developed for displacement separation of fatty acids by Holman *et al.* (*J. Am. Chem. Soc. 73,* 1261, 3337, 5285). "Darco carbon" was used as the adsorbent or carrier, and alkyl bromide, alcohol and ethyl esters were useful displacers. In this work, with methyl palmitate-methyl myristate chromatograph systems, palmitic acid appeared between the esters, stearic acid appeared between esters of palmitic and stearic acids, etc. The experiences and data obtained were presented for saturated, unsaturated, and conjugated unsaturated acids.

Kaufmann and coworkers (*Fette u. Seifen 52,* 713; *53,* 69, 285, 390, 405, 408, 555) demonstrated how paper chromatography could be applied to the identification of fats and fatty acids. Fatty acids or their soaps placed on copper acetate impregnated paper could be distinguished by the foam produced. In paper chromatographs with certain dyes, oleic and linoleic acids could be distinguished by fluorescence under ultraviolet light. Various colors were produced by different fatty acids on paper after treatment with osmium tetraoxide, potassium permanganate, or conversion to soaps of heavy metals. Detection and separation work on C_3-C_{20} saturated and unsaturated acids with use of several dyes are given and behavior as salts of phenylhydrazine, hydrazine, copper, thorium, cobalt, nickel, bismuth, manganese, iron, lead, silver, zinc, magnesium, mercury and aluminum described. Oleic acid was determined by chromatographing as the radio active soap and measuring the radiation. Other applications described pertained to the analyses of soap, lacquer raw material, lipides in the living human skin, etc.

The writer also calls attention to work by Hirst & Lancaster (*Trans. Faraday Soc. 47,* 315) in which they determined the effect of water on interactions between stearic acid with powdered oxides and carbides of metals, because this information should be fundamentally pertinent to fat chromatography.

Experience with a 54-unit apparatus indicated that C_5-C_{18} fatty acids could be separated by liquid-liquid countercurrent distribution (Barry *et al.*—*J. Biol. Chem. 188,* 299).

Spectroscopic determination of unsaturated acids with isolated double bonds after alkali isomerization was reviewed (Strüber—*Fette u. Seifen 52,* 562). An American Oil Chemist Society committee report on spectroscopy analysis of oils suggested 12 minor changes in the details of the procedure (Stillman *et al.*—*J. Am. Oil Chemists' Soc. 28,* 331). Privett & Lundberg (*Ibid.* 313) cautioned that errors were caused in this procedure by oxidation products of polyunsaturated acids; and

that such oxidized material must first be removed by countercurrent extraction with aqueous alcohol solutions. Hilditch's *et al's.* (*Analyst 76,* 81) corrections in the procedures pertained to correction of E values used for linoleic acid; because the standard values of the old procedure derived from brominated segregated linolenic acid were different from that segregated by physical means. Kaufmann *et al.* (*Fette u. Seifen 52,* 210) characterized the spectral behavior of α-, β- and allo-elaeostearic, α- and β-licanic, and α- and β-parinic acids; and developed equations so that oils containing these could be analyzed spectrophotometrically.

Infrared spectra technique was also applied to the analysis of fatty material. Infrared data for the saturated C_6-C_{18} acids and their methyl and ethyl esters were presented in plots of percentage transmission against the wavelength in microns as fundamental information for identification of each (O'Connor *et al.*—*J. Am. Oil Chemists' Soc. 28,* 154). Similar data were recorded by Lecomte (*Oleagineux 5,* 685; *6,* 72) and Shreve *et al.* (*Anal. Chem. 22,* 1498). The latter's data were on monounsaturated acids, and methods of distinguishing *cis-* and *trans-*compounds were pointed out. According to Jackson & Callen (*J. Am. Oil Chemists' Soc. 28,* 61) the infrared method for determination of *trans*-isooleic acids in hydrogenated oils was superior to the Twitchell method. Volbert (*Fette u. Seifen 53,* 559) reviewed infrared spectrography applications in the fat field.

Some investigations on composition of fats were limited to specific fatty acids. Hilditch (*Chemistry & Industry 1951,* 846) suggested that trade specifications for fatty oils should be based on their unsaturated fatty acid composition. Shorland *et al.* (*J. New Zealand Inst. Chem. 14,* 142; *Nature 166,* 745; *Chemistry & Industry 1951,* 839; *Biochem. J. 50,* 207) isolated two C_{17} methyl branched chain fatty acids from butterfat. According to analyses of Schaffer and Holm (*J. Dairy Sci. 33,* 865) summer butters contained 2.62-2.71% linoleic acid and 0.81-1.17 linolenic acid, and for winter butters the figures were 2.11-2.17 and 1.05-1.29, respectively. A hexadecatrienoic acid isolated from rape oil had double bonds at 7, 10, and 13 positions (Heyes & Shorland—*Biochem. J. 49,* 503). An acid with four double bonds was isolated from tall oil (Kajanne—*Acta Chem. Scand. 4,* 1147). Arachidonic acid was isolated from beef suprarenal glands (Herb *et al.*—*J. Am. Oil Chemists' Soc. 28,* 55). Lespedeza seed oils contained 22-28% triunsaturated and 26-42% diunsaturated nonconjugated fatty acids (Wiley *et al.*—*Ibid.* 459). Analysis of the seed fats of Balsaminaceen species gave parinic acid contents as follows: *Impatiens Roylei Walpers* 42, *I. parviflora* 46, *I. noli me tangere* 32, *I. fulva* 51, *I. Holstii nana amabilis* 13, *I. Sultanii* 27, and *I. hortensis* 27% (Kaufmann & Keller—*Fette u. Seifen 52,* 389). Other unusual fatty acids isolated were a new hydroxy acid from isano seed oil having one hydroxy radical at the eight carbon (Riley—*J. Chem. Soc. 1951,* 1346, Kaufmann *et al.*—*Fette u. Seifen 53,* 537), 14-hydroxypalmitic acid from bees wax (Toyama & Hirai —*Fette u. Seifen 53,* 556), 9,14-dihydroxy-10,12-octadecadienoic acid from tung oil (Davis *et al.*—*J. Am. Chem. Soc. 72,* 124) ellagic acid from drupes of *Rhus succedanea* (Chen—*J. Toiwan Pharm. Assoc. 2,* 17), α-, β-unsaturated C_{28}–C_{30} acids from tubercle bacilli lipides (Chanley & Polgar—*Nature 166,* 693), a C_{19} acid containing a cyclopropane ring from *Lactobacillus arabinosus* (Hofmann & Lucas—*J. Am. Chem. Soc. 72,* 4328), and a C_{20} and several C_{22} fatty acids from bonito oil (Matsuda —*J. Soc. Chem. Ind., Japan 45B,* 49, 88, 158, 159).

Some analyses of fatty acids were made to show a relationship to source, species, environment or other factors. The component acids of perinephric and interscapulac fats of tame rabbits were very similar, but both were appreciably different than respective fats from wild rabbits (Clement & Meara—*Biochem. J. 49,* 561). The mixed acids of bone fat of cattle contained myristic 1-5, palmitic 19-32, C_{14}-enoic 1-2, C_{16}-enoic 2.6-10, linoleic 1.5-3.2, linolenic 0.7-1.1 mole % with oleic and traces of other acids; thus it was quite different from other fats (Holmberg & Rosenquist—*Svensk Kem. Tid. 63,* 12). Youngs' *et al.* (*Can. J. Chem. 29,* 871) analyses of Argentine and Western Canada rape oil showed 6-10% more eicosenoic acid and 7-10% less erucic acid than previously reported for rape oils. The linoleic acid and total tocopherol contents of animal and vegetable oils, for which these characteristics were known had a highly significant correlation (Hove & Harris—*J. Am. Oil Chemists' Soc. 28,* 405). A comparison of oil composition of grass of four genera—*Coix, Euchlaena, Tripsacum,* and *Zea* (corn)—showed that they had the same pattern of composition, except that Coix was most saturated (Wiley & Wilken—*J. Org. Chem. 16,* 1536). Peanut oils from different geographic sources had 20-38% linoleic and 60-39% oleic acids in the mixed fatty acids (Crawford & Hilditch—*J. Sci. Food Agr. 1,* 372). Olive oils of Argentine provinces had mixed acids with oleic 51.3-76.0, linoleic 6.4-19.6% and saturated acids 13.0-23.2% (Catteaneo—*Anales asoc. quim. argentina 38,* 383). Analyses showed that small unisexual olives contained a larger percentage of oil per unit weight than normal olives, and that there was very little differences in the chemical characteristics of the oils from the two sources (Liso—*Olivicoltura 5,* No. 1, 8). Analysis of a by-product oil from a fermented soybean-wheat food product indicated that it was too decomposed for use as an edible oil (Kubo—*J. Agr. Chem. Soc. 23,* 335). The tallow of Icelandic sheep had a higher unsaponifiable content (0.9-2%) and lower saponification value than that of sheep of other geographic origin (Bjarnason—*Atvinnudeild Haskol. Rit Idnadardeildar 1950,* No. 1/3, 3). The mesentric fats of Swedish horses contained more linoleic and less linolenic acid than those of English and New Zealand horses (Gupta & Hilditch—*Biochem. J. 48,* 137). In analyses of the fat composition of parts of corn and grain sorghum kernels; gluten, fiber and germ fats had approximately similar amounts of oleic and linoleic acids in each, with gluten and fiber fats showing additional amounts of polyunsaturated acids (Baldwin & Sniegowski—*J. Am. Oil Chemists' Soc. 28,* 24).

Brief discussions on the composition of fats were written by Hilditch (*Brit. J. Nutr. 3,* 347), Daubert (*Inst. Spokeman 15,* No. 9, 18), and Liberman & Mirkin (*Myasnaya Ind. S.S.S.R. 22,* No. 1, 20). The latter report was limited to livestock fats and contained information on fractionating them by crystallization. Many reports on analyses of fats and oils were included in this review by tabulations appended to this section because that was most convenient. A group of analytic reports will not be reviewed in such detail because the data agreed with others easily found in the literature. These analyses were on the avocado pears of Turkey (Yazicioglu—*Fette u. Seifen 53,* 9), castor oil of Africa, America, and Asia (Gupta *et al.*—*J. Sci. Food Agr. 2,* 245), chrysalis oil (Serchi & Mazzoni—*Chimica, Milan 5,* 290), citrus fruit seed oil (Lobo *et al.*—*Ion 10,* 651), Egyption fenugreek oil (Shahat — *Proc. 11th Intern. Congr. Pure Applied Chem., London, 3,* 569), Argentine corn oil (Fortunato—*Ind. y quim. Buenos Aires 10,* 132), New England horse chestnut, *Aesculus hippocastanum,* oil (Ehlers & Hill—*J. Am. Oil Chemists' Soc. 28,* 45), Sudan Africa okra seed oil (Crossley & Hilditch—*J. Sci. Food Agr. 2,* 251), Paraguay palm, *Acrocomia totai* oil (Landmann—*Seifen-Öle-Fette-Wachse 77,* 403), *Parenarium laurinum* seed oil (Riley—*J. Chem. Soc. 1951,* 291), oil from the seed of *Rhus succedanea* (Japanese tallow tree) (Chen—*J. Toiwan Pharm. Assoc. 2,* 17), Indian sesame oil (Chakrabarty & Hilditch—*J. Sci. Food Agr. 2,* 255), sunflower oil produced in North China (Ueno & Wan—*J. Agr. Chem. Soc. Japan 19,* 735), and tung oil of Formosan origin (Chin—*J. Chem. Soc. Japan Ind. Sect. 52,* 215). Among the above reports those by Hilditch and coworkers contained information on the distribution of the fatty acids among the glycerides of the glyceride composition of the fats. Riley (*J. Chem. Soc. 1951,* 291) determined the distribution of the fatty acids among the glycerides of *Parinarium laurinum.*

Some analytical reports on lipides were limited to certain constituents other than fatty acid glycerides. A review and investigations of phosphatide determination by Kaufmann *et al.* (*Fette u. Seifen 52,* 600, 736) indicated there was considerable disagreement of results; so they selected a modified Grossfeld-Zeisset method and analyzed many milk products. With cheese the phospholipide content increased with the fat content. In milk the phospholipide content was unaffected by heating at 100°; acidification before heating caused a 19.8% reduction; and condensation by heating at 100°C. resulted in a 23.8% decrease. Taylor & McKibbin (*J. Biol. Chem. 188,* 677) determined total lipide nitrogen, phosphorus, choline, and sphingosine in the blood plasma of nine species. There were differences between species and within species; however, there was a similarity in pattern, for choline nitrogen comprised 64-79% and sphingosine nitrogen 10-21% of the total nitrogen. A centrifugal acetone foots test was applied to crude soybeans as a rapid method for estimating the phospholipide content (Freyer & Shelburne—*J. Am. Oil Chemists' Soc. 28,* 393).

Cholesterol determinations in skin fat were found to be more exact and simpler by a spectrophotometric method based on the Liebermann-Burchard reaction than by the nephelometric method (Lincke & Kläui—*Arch. Dermatol. u Syphilis 192,* 402). The spectrophotometric method could determine both free and ester cholesterol when higher concentrations of acids and higher temperatures than normally employed were used so as to eliminate the difference in reaction rates.

A method of determining gossypol in cottonseed oil by the color developed with *p*-anisidine was modified by using a 794:206 mixture of hexane:isoproponol as the solvent (Pons *et al.*

—*J. Am. Oil Chemists' Soc. 28,* 8). A number of gossypol-like pigments gave the same reaction. Pominski *et al.* (*Ibid.* 444, 472) demonstrated that gossypurpurin gave the same reaction. This compound had the elemental formula $C_{30}H_{32}O_7$ N.

Budowski *et al.* (*Ibid.* 51) developed a spectroscopic method for determination of sesamin in sesame oil and found that the oils contained 0.50-0.96% sesamin. Their work (*Ibid.* 54) on the sesamin, sesamolin and phytosterol in sesame oil indicated that these compounds were responsible for the optical rotation of the oil and 75-85% of the total unsaponifiable.

The appearance of turbidity in beechnut oils on storage was principally due to precipitation of myricyl lignocerate (Kaufmann *et al.*—*Fette u. Seifen 53,* 531).

Several analytical procedures pertained to color in margarine. Brant-Smith (*Can. J. Technol. 29,* 290, 296, 303) determined the influence of temperature on the Lovibond tintometer color readings, developed charts so that Hunter reflectometer color readings could be converted to Lovibond units, and found that surface dehydration could cause increase in surface color of the commercial products. Schmalfuss (*Fette u. Seifen 52,* 739) used Kuhn & Brockman standard color solutions to rate the color of margarines and butters colored with carotene. Annatto dye used for the same purpose was detected by chromatography on alumina, removing other dyes from the adsorbent by elution with alcohol, and confirmation of the dye by Carr-Price reaction (Thaler & Scheler—*Z. Lebensm.-Untersuch. u.-Forsch. 93,* 220). To include detection of butter yellow in this procedure the eluate was evaporated, taken up with benzene, and chromatographed on "Floridin XXF" (*Ibid.* 286). The presence of butter yellow produced a dark carmine red colored zone. Thaler (*Fette u. Seifen 53,* 132) reviewed chromatographic methods for detection of the artificial coloring in fats. Chromatographic procedures were used to identify three carotene isomers and lycopene in carotene concentrates prepared from palm oil (Löw & Argoud—*Oleagineux 5,* 629).

Various discrepancies in the vitamin A determinations of fats and means for their correction were investigated. The irrelevant spectra absorbing substances originating in the unsaponifiable during vitamin A determination of margarine were eliminated by the use of double chromatography with two different adsorbents in series (Boldingh & Drost—*J. Am. Oil Chemists' Soc. 28,* 480). The influence of presence of tocopherol on the results was compensated for by a correction obtained from a blank determination in which the solution was shaken in hexane solution with 60% sulfuric acid (Fox & Mueller—*J. Am. Pharm. Assoc. 39,* 621). The corrections necessary for spectrophotometric assay of vitamin A were determined by seven collaborative determinations of the $E^{1\%}_{1cm.}$ at 328 mμ and geometric correction of irrelevant absorption (Adamson *et al.*—*Analyst 76,* 445; Gridgeman—*Ibid.* 449). Investigations on kitol and its diacetate isolated from whale oil showed that the former interferes negligibly and the latter considerably in the spectroscopic determination of vitamin A (Chatin & Debodard—*Compt. rend. 233,* 105). Das *et al.* (*Proc. Indian Acad. Sci. 30B,* 299) after comparing biological and tintometric determination of vitamin A in several fish liver oils, found that conversions factors for correcting the latter method were different for the different species. In the colorimetric determination of vitamin A with glycerol dichlorohydrin the sensitivity and accuracy of the method was improved by additions of adequate amounts of antimony trichloride and hydrochloric acid to the reagent (Fujita & Aoyama—*J. Biochem. Japan 38,* 263). These investigators (*Ibid.* 271) also described a new procedure based on the measuring of the yellowish green fluorescence of vitamin A. Abul-Fadl *et al.* (*J. Roy Egypt. Med. Assoc. 33,* 521) reported that the vitamin A content of Samina, the butter of the milk of the Egyptian buffalo, was twice that of ordinary cow butter.

A new chemical method for determination of vitamin D was based on a color reaction produced with furfural in ethanol solution and in the presence of sulfuric acid (Candela *et al.*—*Anales real soc. espan. fis. y quim. 46B,* 509, 609). Vitamin D assays of Indian fish liver oils indicated that the D-potencies of these were negligible in comparison to the vitamin A in the oils (Appanna & Devadatta—*Proc. Indian Acad. Sci. 33B,* 199).

A comparison of determination of sterols in palm oil by separation as complexes with digitonin and with natigine indicated that the latter was unsuitable for separation of pure sterols (Mellier—*Oleagineux 6,* 20). Sterols of formulas $C_{19}H_{16}O$, and probable formulas $C_{27}H_{56}O$, $C_{14}H_{28}O_2$ and $C_{14}H_{18}O$ were chromatographically separated from butter fat unsaponifiable (Morice—*J. Chem. Soc. 1951,* 1200). Meretristerol of crustacea was isolated in purer form than heretofore and its characteristics recorded (Kita *et al.*—*J. Chem. Soc. Japan, Chem. Sect. 71,* 21). The fat of fin back whale liver contained a highly unsaturated hydrocarbon of probable formula $C_{48}H_{78}$ (Tsuchiya & Kato—*Ibid.* 514).

A method for decomposing oils for spectrochemical determination of iron and copper based on chemical oxidation and charring with heat was described by Melvin & Hawley (*J. Am. Oil Chemists' Soc. 28,* 346).

A method for determination of small amounts of trichloroethylene in vegetable oils was a modification of a method of Fujiwara, and was based on codistillation with xylene and water and spectrophotometrically measuring the color produced when a portion of the solution was heated with a pyridine and lye solution (Eisdorfer & Mehlenbacher—*Ibid.* 307).

DETECTION OF ADULTERATION. A method for characterizing olive oils, similar to that of Fitelson, was based on the iodine number of the hydrocarbons of the unsaponifiable material (Hadorn & Jungkunz—*Mitt. Lebensm. Hyg. 41,* 435). This characteristic, named the squalene number, average 300 for commercial olive oils, was 1062 on a freshly laboratory extracted sample, and very low on other oils. The solvent used in extracting olive oils could be identified by the character of a one to five solution of the oil in petroleum ether (Wittka—*Olearia 4,* 405). The petroleum extracted oil gave a clear test, that obtained with carbon disulfide produced a greenish-gray precipitate, and from trichloroethylene a separation of tar and precipitation of a greenish white powder occurred.

A specific test for tung oil, sensitive to mixtures containing over 10%, was based on the gelling produced on treatment with a mixture of sulfuric and nitric acids and ferric chloride (Korotkov—*Gigiena i Sanit. 1951,* No. 1, 38). The test was issued in connection with poisonings caused by adulteration of food oils with tung oil.

A determination of the amount of castor oil in oil mixtures was based on the refractive index of the mixtures before and after acetylating (Achaya & Saletore—*J. Sci. Ind. Res., India, 10B,* 118). A proposed test for detection of dehydrated castor oil was based on identification of the 10,12-octadecadienoic acid in the samples (von Mikusch—*J. Am. Oil Chemists' Soc. 28,* 133). This test is useful in the paint, varnish and related industries.

A method of detecting mineral oils in fatty oils based on the turbidity of a saponified solution of the samples was not sensitive to up to 10% adulteration with white mineral oil (Patzsch—*Pharm. Zentralhalle 89,* 302). A method for estimating the amount of coconut oil in mixtures with mineral oils and other vegetable oils was based on determining Reichert plus Polenske values and reference to standard curves developed from known mixtures (Iyer—*J. Sci. Ind. Res., India, 9B,* No. 4, 93).

The aniline point determination was found to be a poor method for identification of oils or detection of adulteration because the results were affected by moisture, free fatty acids and rancidity (Kane & Ranadive—*Ibid. 10B,* 62).

Detergents

MANUFACTURE. The new patents on continuous soapmaking processes concerned complete systems and an improvement in existing methods. Bradford's (*U. S. 2,539,899*) new system was based on dissolving the fat in kerosene, bleaching, saponifying, and vaporizing to remove solvent and glycerol. Mill's (*U. S. 2,578,366*) system was based on proportionation of molten fatty acids and lye solution, and regulating temperature so that a soap product of 18-22% moisture was formed. Other continuous soap equipment and systems were based on means of emulsifying fat and saponification agent, passing into heated saponifying zones, and through separating steps (Owen—*U. S. 2,566,-359*; Schueller *et al.*—*Fr. 946,740, Fr. 946,746*). A new glycerol recovery step for a continuous process comprised mixing the soap with lye and allowing soap and a glycerol-lye mixture to separate in a quiescent zone (Owen—*U. S. 2,562,207*).

Some new fundamental information for soapmaking pertained to the fatty raw material. Jellinek & Gordon (*J. Applied Chem., London, 1,* 185) recorded data on saponification of the monoglycerides of myristic, stearic, and oleic acids in sodium hydroxide solution containing 75 volume percent acetone over a range of temperatures. The reactions were found to obey, in general, a second order law. Feldpush & Sutker (*Soap Sanit. Chemicals 27,* No. 12, 48) prepared liquid 20% potassium soaps from mixtures of pure oleic, linoleic, stearic, and palmitic acids to determine the limiting combination under which clear commercially acceptable liquid soaps could be made. The effect of silicates on the viscosity of this type of soap was determined (Spencer—*J. Am. Oil Chemists' Soc. 28,* 426). The potassium silicates affected linseed and soybean oil liquid soaps but not those of coconut oil. High alkaline silicates caused greater increases in viscosities. The data were applicable for designing

blending operations for liquid soap manufacture. Bracho (*Bull. mens. ITERG 4*, 418) determined the amount of brine solutions needed to salt out soap from tallows of different hydroxy acid content. The information was applicable to soapmaking from poor grade tallows or tallows that have been bleached by oxidation with air. Reutenauer & Dupin (*Ibid. 5*, 130) evaluated the yellowing effect of three types of rosin at 15 and 20% in tallow-coconut soaps over a period of six weeks. Since antioxidants and nitrogen atmosphere did not retard yellowing whereas high humidity did, it was suggested that the discoloration was due to change in structure of the rosin soap associated with loss of moisture.

The color of cold-made soaps was improved by emulsifying the fatty acid stock in a solution of a weak organic acid of low molecular weight preliminary to saponification (Peterson—*U. S. 2,547,280*). Tall oil soap was refined in aqueous solution by removal of color bodies and non-saponifiable material with ketone solutions (Hasselstrom & Stoll—*U. S. 2,547,208*). The ketone solution of impurities separated from the aqueous soap solution on standing at room temperature. The addition of material yielding phosphate ion during soap-fitting operation, prior to settling, also reduced the color of soap products (Kirschenbauer & Percy—*U. S. 2,567,381*).

A small amount of new information was on the nonsoap constituents of laundry soaps. Special polyphosphate builders were prepared by heating mixtures of mono- and diorthophosphates above 300° but below the melting point of the mixtures (Henkel & Cie—*Ger. 762,903*). In a discussion on laundering compositions, Uhl (*Fette u. Seifen 53*, 84) suggested that presence of 10-15% synthetic detergents in soap maintained dispersion of calcium soap without affecting suds appreciably, that tylose increased detergency only up to 0.3% concentrations, and that when phosphates were used Trilon should be added to inhibit damage to fibers. Certain mixtures of alkali phosphates and silicates, and aluminum salts were recommended as noncaking agents for powdered detergents (Funderburk—*U. S. 2,579,380*). The newly patented optical bleach additives for soaps were derivatives of 4-amino-2,2'-stilbenedisulfonic acid (J. R. Geigy, A.-G.—*U. S. 2,527,425; Swiss 268,381-5, 269,483*), 4-methyl-7-aminocoumarin (Ciba, Ltd.—*Swiss 265,707-15*), and benzimidazolylethylene (Ciba Ltd.—*Swiss 267,582-5*).

New equipment was designed for finishing soaps. Cooling equipment for spray-dried soap was based on causing turbulence of the particles while blowing ambient air through them (Sartorius—*U. S. 2,544,616*). Other patented soap finishing devices were a soap molding frame (Gertson & Rashal—*U. S. 2,569,469*), a bar soap cutting and handling apparatus (Schulerud—*U. S. 2,563,876*), a machine for cutting and spacing soap cakes (van Buren—*U. S. 2,567,041*), a soap press for compressing chips of soap into a bar (Roma—*U. S. 2,558,879*), and means for making inlaid soap (Hoopes—*U. S. 2,563,839*).

A new method of purifying soap lye crude glycerol was entirely different from the existing techniques. This comprised purifying the glycerol solution by ion exchange. Experiences with this method were recorded by Stromquist & Reents (*Ind. Eng. Chem. 43*, 1065), Zager & Doody (*Ibid.* 1070), and Neu (*Fette u. Seifen 53*, 205). Removal of impurities in this manner permitted concentration of glycerol by evaporation, after which the conventional distillation was not required. A method of deodorizing distilled glycerol comprised addition of a volatile acid followed by steaming under vacuum to remove both odorous material and the added acid (Lofdahl & Gunther—*U. S. 2,578,816*). Viscosity data were reported for aqueous glycerol solutions over the entire range of 0 to 100°C. and 0 to 100% glycerol concentrations (Segur & Oberstar—*Ind. Eng. Chem. 43*, 2117). These data were pertinent for designing all processing and handling equipment.

The name "ersatz" soaps seemed applicable to three patented products. Two were reaction products of caustic and, respectively, ground seeds (Freund—*Hung. 138,556*) and whole horse chestnuts (Kiss—*Hung. 135,207*) and the other was a mixture of acid-treated soybean lecithin and waste liquor from sulfite pulp (Chigusa—*Japan 177,456*).

There was only a small amount of journal literature on synthetic detergent manufacture. In investigating sulfonated fatty amides, Paquot (*J. recherches centre natl. recherche sci., Paris, 1950*, 169) found that the foaming capacity of the lauric preparation exceeded those of capric, myristic, palmitic, stearic, or oleic acids, and that the products, neutralized with 10% caustic, were superior to those neutralized with stronger caustic. Schleicher & Rossi (*Bol. Soc. Chilena quim. 2*, 5) recorded experiences in making sulfonated detergents from Chilean pitcoal tar oils. A method for making a light colored product satisfactory for washing wool was developed. Hirschmann's (*Anales direc. nacl. quim., Buenos Aires 3*) investigations on the effect of temperature and time of reaction for sulfonating linseed oil indicated that best products were obtainable at 4°. At higher temperatures less time was required but it was difficult to control the reaction in a manner to obtain suitable products. Other literature on synthetic detergent manufacture was on newly patented products and manufacturing techniques. For convenience of presentation these are listed below with the assignee, or with the patentee where no assignee was given:

Allied Chemical & Dye Corp.
Alkyl aromatic sulfonate (*U. S. 2,525,024*).

American Cyanamid Co.
Condensation product of ethylene oxide and fatty acid amides (*U. S. 2,520,381*). A mixture of fatty acid toluene sulfate (*U. S. 2,535,972*). Alkylol amides of dimerized fatty acids (*U. S. 2,537,493; Brit. 650,939*). Alkali salt of an ester of phosphoric acid and fatty amide (*U. S. 2,545,357*). Monoalkyl sulfosuccinates rendered soluble with lithium, beryllium, cobalt, or chromium salts (*U. S. 2,562,154-6, 2,567,159*). Reaction products of fatty amines, cyanamide, and polyoxyethylenes (*U. S. 2,574,510*). Reaction products of fatty guanidines and alkylene oxides (*Brit. 650,820*).

Atlantic Refining Co.
Reaction products of oxidized hydrocarbon and ethylene oxide (*U. S. 2,542,697*).

Atlas Powder Co.
Polyoxyethylene fatty acid amines (*U. S. 2,559,583-4*).

Badische Anilin & Soda Fabrik.
Diquaternary ammonium compounds (*Ger. 802,694*). Polyesters containing various solubilizing radicals (*Ger. 803,835*). Refining organic sulfonic acids (*Ger. 804,571*).

Bersworth, F. C.
Alkylene polyamine detergent mixtures (*U. S. 2,524,218, 2,530,147*).

Burton T. Bush, Inc.
N-alkyl (2-alkanol)amines (*U. S. 2,541,088-9*).

California Research Corp.
Sulfonation of alkylaromatic hydrocarbons (*U. S. 2,531,324*).

Ciba, Ltd.
Mixture of partial ester of glycol, and condensation product of *N*-methylolamide of a fatty acid with mercaptocarboxylic acid (*U. S. 2,576,896-9*).

Colgate-Palmolive-Peet Co.
Sulfamic acid products stabilized with buffers (*U. S. 2,513,549*). Di-N-alkoxy-substituted piperazine derivatives (*U. S. 2,541,260*).

Dobbelman, N. V.
Mixture of sulfonated hydrocarbons and sulfonated rosin (*U. S. 2,571,689*).

E. F. Drew & Co., Inc.
Polyalkanol amine condensed with coconut oil fatty acids (*U. S. 2,538,929*).

J. R. Geigy A.-G.
Polyglycol ethers of mixtures containing organic hydroxy compounds (*Swiss 267,360*).

General Aniline & Film Corp.
Sulfonated, alkylated aryl glycol ethers (*U. S. 2,536,976*). Sodium salts of oleyl methyl tauride, diethylene glycol mono butyl ether, and ethylene-bis-(iminodiacetic acid mixture (*U. S. 2,542,385*). Mixture of soap and polyalkylene glycol ether derivatives (*U. S. 2,543,744*). Same containing also formaldehyde-alkyl-naphthalene sulfonic acid product (*U. S. 2,555,285*). Similar mixtures containing builders (*U. S. 2,560,839*). Fatty amide derivatives (*U. S. 2,572,809-10*). Mixtures of soap, polyglycol ether derivatives, and quaternary ammonium germicides (*U. S. 2,577,773*).

Hadnagy, Z.
Detergents from aromatic or heterocyclic acids with simultaneous production of furfuryl alcohol (*Ital. 449,451*).

Harvel Corp.
Sulfonated cashew nutshell liquid (*Brit. 645,062; U. S. 2,559,593-4*).

Houghton, E. F.
Water soluble salts of benzoyl sulpho propionic acid (*U. S. 2,548,017-21*).

Hansawerke Luermann, Schuette & Co.
Method of preparing organic sulfonamides (*Ger. 800,411*).

Imperial Chemical Industries, Ltd.
A mixture of condensate of phenol, fatty acid, and ethylene oxide, and condensate of fatty acid with β-monohydroxyethyl amide (*U. S. 2,576,913*). A mixture of the latter and condensate of fatty acids and ethylene oxide (*U. S. 2,577,503*).

Krupp Treibstoffwerk G.m.b.H.
Sulfoxidation of aliphatic or cycloaliphatic hydrocarbons (*Ger. 802,820*).

Kyoritsu Marine Industries Co.
Sulfonation of sperm oil in the presence of boron bisulfite (*Japan 178,560*).

Mannheim, H. S.
Cycloinidine fat acid derivatives (*U. S. 2,528,378-80*).

Micropon.
Condensed product of albumins with organic sulfonic acid halides (*Gr. 955,174*).

Monsanto Chemical Co.
Condensation products of polyoxyethylene containing builders (*U. S. 2,522,446-7*). Condensation products of ethylene oxide and tall oil (*U. S. 2,550,691*). Same containing fatty mercaptan condensed with ethylene oxide (*U. S. 2,572,805*).

Nopco Chemical Co.
Condensation product of 2 molecules of alkylolamine with 2 molecules of fatty acids (*U. S. 2,540,678*). Product of 3 alkyl groups and a fatty acid sulfonated and converted to the amide with butyl amines. (*Brit. 642,836*).

J. B. Niederl & Associates, Inc.
4,4-Dialkylthiamorpholinium alkyl sulfates (*U. S. 2,541,714*).

Nippon Petroleum Oil Co.
Sulfonated pine oil fraction (*Japan 179,657*).

Nussbaum, J. & Lubicz, V.
A mixture of triethanolamine soaps of rosin and sodium lauryl- or cetylsulfonate and builders (*Ital. 444,081*).

N. V. Bataafsche Petroleum Maatschappij.
Deodorizing alkylated aromatic sulfonates (*Dutch 67,091*). Sulfonates of oxidized hydrocarbons (*Dutch 67,931*).

Onyx Oil & Chemical Co.
Fatty acid phenylethyl pyridinium bromide (*U. S. 2,542,642*).

Price, R. H.
Liquid soap of lower alkanolamines of oleic acid (*U. S. 2,551,634*).

Proctor & Gamble Co.
Sulfating fatty alkylolamides (*U. S. 2,551,125*).

Publicker Industries, Inc.
Esters of 4-sulfo-4-cyclohexane-1,2-dicarboxylic acid (*U. S. 2,551,575*).

Rohm & Haas Co.
Quaternary ammonium compounds having pentachlorophenate as an anionic group (*U. S. 2,541,816, 2,541,961*).

Rare-Galen, Inc.
Sulfated and saponified fat with pH of 7.5-8.5 (*U. S. 2,553,716*).

Sandoz, Ltd.
Fatty acid amino sulfonates (*U. S. 2,543,852*).

G. D. Searle & Co.
(Phenylaminobenzoyl) alkanoic acids (*U. S. 2,555,955*).

Sharples Chemicals, Inc.
3-(alkylmercapto)-2-hydroxypropyl quaternary ammonium halide (*U. S. 2,548,679*). Polyethylene glycol thioethers (*U. S. 2,565,986*).

Shell Development Co.
Salting hydrocarbon sulfonates from impurities in polar solvents (*U. S. 2,522,212*). Diamine fatty acid salts (*U. S. 2,539,685*). Mixture of alkyl sulfuric acid esters and soap (*U. S. 2,567,645*). Alkyl aromatic sulfonic acid salts (*U. S. 2,567,854*).

Standard Oil Co.
Removing sulfuric acid from alkane sulfonic acids (*U. S. 2,518,639*). Petroleum sulfonic acid compounds converted to nitro derivatives (*U. S. 2,559,503*).

Ueno, N.
Blown or polymerized fish oil which was then sulfonated (*Japan 176,707-8, 176,912*).

U. S. Secretary of Agriculture
Amides of 9,10-epoxystearic acid (detergent intermediate) (*U. S. 2,567,237*).

Upjohn Co.
7-chloro-4-[1-methyl-4-(1-pyrrolidyl)-butylamino]-quinoline (*U. S. 2,526,417*).

Visking Corp.
Aliphatic 2-amino sulfonic acid (*U. S. 2,510,281*). Aliphatic 2-nitro sulfonic acids (*U. S. 2,510,282*).

Wyandotte Chemicals Corp.
Sodium alkyl benzene sulfonate (*U. S. 2,566,501*). Combining sodium carboxy methyl cellulose with synthetic detergents (*U. S. 2,568,334*). Removal of inorganic sulfates from alkali metal benzenesulfonate (*U. S. 2,572,344*).

Many of the new toilet soaps were designed for use with hard water. Those patented by Preston (*U. S. 2,527,075-8*) were bar soaps containing alkylsulfonates, fatty acid amides, and other synthetic detergents. An all-synthetic detergent toilet bar contained alkylaryl sulfonate, with soybean lecithin as a detackifier, corn starch for hardening, and talc to provide slush resistance (Keenan—*Soap Sanit. Chemicals 27*, No. 5, 27). Sodium fluoride added to ordinary soap inhibited formation of insoluble soaps when used with sea water (Dobbelman N. V.—*Dutch 66,550, 67,413*). A bar soap that was exothermic contained two separate juxtaposed cakes containing different chemicals which, when in contact with water, reacted to heat the water (Elissabide—*Brit. 643,493*).

The new hand cleaner compositions were a mixture of wood flour, powdered soap, borax, and a synthetic detergent (Savidge & Tyrer—*Brit. J. Ind. Med. 8*, 26), a ground mixture of cereal flours and alkali silicates (Del Franco—*Ital. 446,551*), and tablets containing alkyl-aryl sodium sulfonate, sodium sulfate, and sawdust (Emerson & Cuming—*U. S. 2,560,097*). A medicated soap contained soap, lanolin, sulfur, and wood tar (Narasimham—*Indian Soap J. 16*, 223). Soaps germicidal to *Staphylococcus aureus* contained 1-3% of 2,2'-dihydroxy halogenated diphenylmethane (Kunz & Gump—*U. S. 2,535,077*). An antiparasitic soap contained a small amount of "DDT" or a mixture of "DDT" and hexachlorocyclohexane (Paoloni & Eusepi—*Ital. 444,855*).

Several detergents were made for special uses. An antiseptic detergent for the dairy industry or for dishwashing consisted of nonionic detergent, quaternary ammonium compound, and alkali salts (DuBois—*U. S. 2,519,747*). A new glass cleaning composition contained alkali ammonium pyrophosphates and a synthetic detergent (Flaxman—*U. S. 2,524,380*). The inorganic constituent for a similar product was a mixture of sodium carbonate and glassy phosphates (Beiley *et al.*—*U. S. 2,568,-110*). A detergent for cleaning hotel china, glassware, and table silver contained chlorinated hydrocarbon solvent and alkylarylsulfonate dispersed in water (Marshall & Wilson—*Brit. 591,035*). Zincates, beryllates and aluminates were added to alkaline dishwashing detergents to inhibit deterioration of vitreous- and ceramic-ware surfaces (Bacon & Otrhalek—*U. S. 2,575,576*). A metallic wool scouring pad contained soap and sodium tetraborate (Belluschi *et al.*—*Ital. 449,590*). Some general purpose detergents comprised special mixtures of alkali salts (MacMahon & Taylor — *U. S. 2,515,880;* Kovacs — *Ital. 454,657;* Henkel & Cie—*Ger. 753,058*). A gel-like product contained ammonium oleate, sodium oleate, water, and solvent derived from petroleum (Guastavino—*U. S. 2,567,099*). A paste product contained petroleum hydrocarbon, magnesium oxide, soap, triethanolamine, alcohol, glycerol, and water (Duranti—*Ital. 456,807*). A rubber-like cleaner was made from soybean oil, lime, sulfuryl chloride, and petroleum solvent (Blumberg—*U. S. 2,546,383*). Detergent formulas developed by the Mjölkcentralens Centrallaboratorium (*Svenska Mejeritidn. 43*, 99, 111, 123, 158) for glass cleaning machies, dairy use, machine cleaning, and for other industrial purposes contained various mixtures of builders, lye, and alkylaryl sulfonates. The liquid dry cleaning compositions were mixtures of organic solvent materials containing some detergent material (Secrist & Petering—*U. S. 2,576,419;* Davidsohn—*Soap Sanit. Chemicals 27*, No. 8, 47), and a liquid metal cleaner was of the same nature (King—*U. S. 2,528,230*). Denture cleaning composition (Apperson—*U. S. 2,476,205*), well drilling fluids (Fischer—*U. S. 2,573,959-61*), a moisture supplying means for spindles on a cotton picking machine (Rust—*U. S. 2,567,301*), and means of laying dust from mine drills (Nedin & Morgun—*Gornyi Zhur. 1951*, No. 1, 34) were based on detergent compositions.

Many communications on detergents provided historical information, described known processes, products, or raw material, or gave other general information. For convenience these are tabulated under the subjects treated:

Historical information: Soap progress since 1900, McCutcheon—*Soap Sanit. Chemicals 27*, No. 1, 27. Reviews of recent literature, Berlitzer—*Seifen-Öle-Fette-Wachse 77*, 88, 112; Uhl—*Fette u. Seifen 53*, 354. Developments of synthetic detergents in Switzerland, Stüpel—*Soap, Perfumery & Cosmetics 24*, 52, *Seifen-Öle-Fette-Wachse 77*, 23. Future outlook for soap and synthetic detergents, Flett—*Soap Sanit. Chemicals 27*, No. 3, 35.

Manufacture: Continuous methods, Rao—*Soap, India, 1*, No. 10, 12; Kothe—*Seifen-Öle-Fette-Wachse 77*, 87; Nichterlein—*Ibid. 76*, 485; Nelson—*Indian Soap J. 16*, 217. Production of various types, Szmidtgal—*Przemysł Chem. 6*, 229. New methods, Heinz—*Chem. Ing. Tech. 22*, 458. Automatic manufacture of liquid hand soap, Ballantine & Jessop—*Soap Sanit. Chemicals 27*, No. 12, 45. Errors

in soap manufacture, A. K.—*Seifen-Öle-Fette-Wachse 77,* 45. Soap-fitting, Weber—*Ibid. 303.* Soap flakes, Zilske—*Ibid. 76,* 488. Nomograph for determination of soap yields, Stölzle—*Ibid. 77,* 158. Spray drying, Ladisch—*Fette u. Seifen 53,* 349, 413. Drying soap, Smith—*Am. Perfumer Essent. Oil Rev. 58,* 125.

Soap ingredients: Pisa fat as a substitute for coconut oil in soap, Gupta—*J. Sci. Ind. Res., India, 9B,* 275. Fatty acids for soap manufacture, Smith—*Am. Perfumer Essential Oil Rev. 57,* 131; *58,* 53. Silicates, Smith—*Ibid. 57,* 305. Phosphates, Smith—*Ibid. 57,* 223. Cellulose derivatives, Smit & Nieuwenhuis—*Witwasserij—Ind. 5,* No. 3, 9; Davis—*Pharm. J. 165,* 347; Voss—*Melliand Textilber. 29,* 382; *30,* 197; Muhr—*Chemia, Switz., 2,* 242. Enhance detergency with natural hydrophilic colloids, Rordorf—*Seifen-Öle-Fette-Wachse 77,* 283. Superfatting agents, Zilske—*Ibid. 77,* 263. Optical bleaches, Stearns *et al.*—*Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 144; Uhl—*Fette u. Seifen 53,* 545. Detergent additives, Bergwein—*Seifen-Öle-Fette-Wachse 77,* 118. Acetal in soap perfuming, Schmidt—*Fette u. Seifen 53,* 42.

Soap products: Liquid soaps, Vallance & Wells—*Indian Soap J. 16,* 147, 179. Hand washing pastes, Augustin—*Seifen-Öle-Fette-Wachse 77,* 308. Incompatibilities of surface active agents in pharmacy, Nixon & Cheetham—*Pharm. J. 165,* 46. Review of the sulfonation reaction, Stüpel—*Angew. Chem. 63,* 461. Sulfonated fatty alcohols, Price—*Australasian J. Pharm. 31,* 960. Sulfonated fish oils for leather fat-liquoring, Das & Sarkar—*Tanner, India 5,* No. 8, 15. Alkyl aryl sulfonates, Kinder—*Fette u. Seifen 53,* 213; Kircher — *Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 140. Alkyl sulfates, Stüpel & Segesser—*Soap, Perfumery & Cosmetics 24,* 556. Nonionic products, Vaughn *et al.*—*J. Am. Oil Chemists' Soc. 28,* 294; Suter & Kramer —*Soap Sanit. Chemicals 27,* No. 8, 33; Cross—*Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 135. Nonionics in drycleaning, Barker & Ranauto — *Soap Sanit. Chemicals 27,* No. 6, 43. Fatty acid esters of glycol, Dollinger—*Rayon Textile Monthly 29,* No. 4, 98. Cationic surface-active agents, Cardew—*Soap, Perfumery & Cosmetics 24,* 154; Sato—*Science, Japan 13,* 403. Amine detergents, Smith—*Am. Perfumer Essent. Oil Rev. 57,* 53. Building synthetic detergents for cotton detergency, Sanders & Lambert—*Textile Res. J. 21,* 680. High-viscosity detergent solutions, Sanders & Knaggs—*Soap Sanit. Chemicals 27,* No. 2, 41; *Proc. Chem. Specialties Mfrs. Assoc. Dec. 1950,* 130. Oxyethylated alkylphenols, Lindner—*Fette u. Seifen 52,* 613.

Specific cleaners: Bath products, Lesser—*Soap Sanit. Chemicals 27,* No. 3, 46. Hand cleaners, Lesser—*Ibid.* No. 6, 30; *7,* 34. Transparent and glycerol soaps, Kesel—*Seifen-Öle-Fette-Wachse 76,* 560. Shave preparations, Lesser—*Soap Sanit. Chemicals 27,* No. 10, 44. Hard surface cleaners, Harris—*Ibid. 27,* No. 6, 49. Cleaning the Eastman Kodak Co. Plant, Weller—*Ibid. 27,* No. 9, 127. Detergent sanitizers, Lesser—*Ibid.* No. 8, 37; Mallmann—*Proc. 3rd Conf. Res., Council on Res. Am. Meat Inst. Chicago 1951,* 54. Detergents for the food industry, Nieman—*Chem. en Pharm. Tech., Holland, 6,* 67. Detergent sanitizers for the dairy industry, Puhle—*Proc. Chem. Specialties Mfrs. Assoc. Dec. 1950,* 172; Elliker—*J. Milk Food Technol. 13,* 156; Dahberg *et al., Ibid.* 5; MacWalter—*J. Soc. Dairy Technol. 4,* 110. Bottle cleaning compounds, Lesser — *Soap Sanit. Chemicals 27,* No. 9, 39. Auto cleaning products, Lesser—*Ibid.* No. 4, 37; No. 5, 31. Detergents for metal cleaning, Strow—*Proc. Chem. Specialties Mfrs. Assoc. Dec. 1950,* 135.

Description of detergents: Soaps versus synthetics, Rossner—*Seifen-Öle-Fette-Wachse 77,* 191; Mudzhiri—*Tekstil. Prom. 10,* No. 7, 26. Constitution of detergents, Elliott—*Textile Record. 68,* 102; Edelstein—*Soap Sanit. Chemicals 27,* No. 9, 35; No. 10, 51.

Laundry technique: Counter-current washing, Uhle—*Fette u. Seifen 53,* 317; *Seifen-Öle-Fette-Wachse 77,* 375. Aralkyl sulfonates in laundry, Wulkow—*Melliand Textilber 30,* 248.

Discussions on washing action: Surface active agents, Waddams—*Soap, Perfumery & Cosmetics 23,* 1019; Groth —*Iva 22,* 9. Emulsifiers and detergents, Guasch—*Afinidad 27,* 494. Classification and structure of surface active agents, Raabe—*Przemysl Chem. 6,* 272; Sisley—*Tinctoria 46,* 569; Dervichian & Lachampt—*Bull. soc. chim., France, 1951,* 289; Stüpel—*Chem.-Ztg. 75,* 473, 500; *Seifen-Öle-Fette-Wachse 77,* 182, 233, 256; Kling—*Melliand Textilber 29,* 275. Calculation of the work of laundering, Kling & Koppe—*Ibid. 30,* 23. Foam action in detergency, Augustin —*Seifen-Öle-Fette-Wachse 77,* 120. Colloids, emulsions, suspensions, and their significance for soap, Nabell—*Ibid. 77,* 37. Sweating of soaps, Srivastava & Sethumadhava—*Indian Soap J. 16,* 241.

General information on analysis and evaluation of detergents: Household soaps of 1928, 1939, and 1951 compared, James—*Soap, Perfumery & Cosmetics 24,* 698. Determination of free sodium hydroxide and sodium carbonate in soaps, Wolff—*Riv. ital. essenze, profumi piante offic. olii vegetali, saponi 32,* 94, 134. Measuring detergent strength, Davis—*Food Manuf. 26,* 13, 53. Practical use tests, Kilber —*Fette u. Seifen 53,* 353; Wurzschmitt—*Ibid. 53,* 209; Snell—*Brit. Rayon Silk J. 27,* No. 314, 60. Qualitative and quantitative analysis of synthetic detergents, Balthazar —*Ing. chim. 32,* No. 182, 169; Marconi—*Chimica, Milan 6,* 251. Measurement of surface tenison, wetting, and foaming, Baldacci — *Ann. chim., Rome, 40,* 358, 372. Light transmission of fibers as supplement to evaluation of laundering course, Walter—*Fette u. Seifen 53,* 322. Evaluation of paint and linoleum cleaners, Harris—*Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 125. Testing quaternary ammonium compounds, Black—*J. Milk Food Technol. 12,* 224.

CONSTITUENT ANALYSES OF DETERGENTS. In an investigation of moisture determinations on alkyl aryl sulfonates, the Karl Fischer method was most satisfactory; oven desiccation did not remove all the moisture, and azeotropic distillation methods were not recommended because of lack of accuracy (Compton & Liggett—*J. Am. Oil Chemists' Soc. 28,* 81).

The procedures published on rapid determination of fatty acids in soap were similar to the butyrometric fat determination used in the dairy industry (Bruno—*Ann. chim., Rome, 40,* 91; Aquarod—*Anales bromatol., Madrid, 3,* 39). A qualitative test for castor oil in soaps was based on heating a sample with pellets of sodium hydroxide and identification by the odor of octyl alcohol which evolves in the presence of castor oil (Mangold—*J. Chem. Ed. 28,* 266). A colorimetric method for rosin and rosin esters comprised heating the sample in benzene with sulfuric acid and acetic anhydride and matching the violet color which develops in the acid layer against standards made from purified rosin (Swann—*Anal. Chem. 23,* 885).

In a new procedure designed for determination of alkaline carbonates and free alkalies in soap containing neutral glycerides and/or resinous soaps, the soap in aqueous solution was first extracted with ethyl ketone to remove the materials which may interfere with the titration (Accinelli — *Olii minerali, grassi e saponi, colori, e vernici 27,* 51, 61). Machemer (*Fette u. Seifen 53,* 150) recommended that the hydrochloric acid used in the volumetric determination of carbonates be replaced by sulfuric acid because the presence of surface active agents increased the solubility of carbon dioxide in the former acid. This was not as pronounced with sulfuric acid. As qualitative tests for the various phosphates, alkyl-dimethylbenzyl ammonium chloride gave precipitates with metaphosphates and tripolyphosphates in acetic acid solution; the latter dissolving in excess reagent (Neu—*Fette u. Seifen 53,* 148). Pyrophosphates did not react with the reagent. In another scheme for identifying phosphates in detergents, the organic matter was removed by extraction; orthophosphates gave a yellow precipitate with silver nitrate solution; pyrophosphates were deposited as characteristic crystals when in solution with sodium hydroxide and acetone was added; and tripolyphosphates were identified by the characteristic of their zinc salt crystals when crystallized slowly (Heinerth—*Ibid.* 31). Crystals used as identification criteria in this work were illustrated. A quantitative method for determining the bactericide, hexachlorophene, in soap was by spectrophotometric evaluation at 550 mμ after development of the ferric compound (Larson—*J. Am. Oil Chemists' Soc. 28,* 301).

Analyses of synthetic detergents were by various procedures. Miller *et al.* (*J. Applied Chem. 1,* 523) analytically separated surface active agents by adding sodium carbonate to aqueous solutions of samples and extracting with *n*-butanol. Evans (*J. Soc. Chem. Ind. 1950,* S76) estimated the amount of anionic synthetic detergent in sewage by extracting with chloroform as the methylene-blue complex and measuring the color intensity of the solution. Total amounts of surface-active material in a solution were determined by titration with suitable dyes; cationic dyes being used with anionic soaps and vice versa (Klevens—*Anal. Chem. 22,* 1141). The method depended on the marked changes in color or fluorescence intensity of the dye in the region of the critical micelle concentration. Swanston & Palmer (*J. Soc. Dyers Colourists 66,* 630) investigated the procedure wherein anionic detergents were titrated with cationic material and vice versa in presence of dye and suggested

modifications to eliminate the effect of sodium carbonate and other salts when present. The methods of Epton and of Barr (Weatherburn—*J. Am. Oil Chemists' Soc. 28,* 233) and the *p*-toluidine titration (Stüpel and Segesser—*Fette u. Seifen 53,* 260) for the determinations of anion-active material were improved in accuracy by new means of calculating corrections from blank determinations and from determinations with known compositions, respectively. A qualitative test for "Tweens" comprised hydrolysis, acidification, extraction with benzene, filtration of extracted solution, addition of barium chloride and silicotungstic or phosphomolybdic acid, and heating (Newburger —*J. Assoc. Offic. Agr. Chemists 34,* 109). Formation of a precipitate was a positive test; but positive tests were also obtained with polyoxyethylene compounds and some basic organic nitrogen compounds. For new schemes for systemic analysis of commercial detergent mixtures the readers are referred to Simmons *et al.* (*Analyst 76,* 279), Gilby & Hodgson (*Mfg. Chemist 21,* 371, 423), and Reutenaur (*Bull. mens. ITERG 4,* 557).

PHYSICAL PROPERTIES. McLaren (*J. Soc. Dyers Colourists 66,* 521) studied the relation between detergency and adsorption by wool. Detergency increased from 50 to 90°C. with no adsorption of detergent. In presence of acids detergency decreased at the higher temperatures and adsorption of soap increased as the acidity was increased. Sodium chloride improved detergent efficiency and decreased adsorption of detergent up to concentrations where precipitation occurred. Similar information was developed by Swanston and Palmer (*Ibid. 632*) using sodium hexadecyl sulfate as the detergent and the effects of both salt and sodium carbonate were determined. Wool containing some of the detergent when transferred to an alkaline solution first lost the adsorbed detergent and after six minutes the detergent started to return and eventually the wool adsorbed more than the original amount.

Stainsby & Alexander (*Trans. Faraday Soc. 46,* 587) calculated the change of heat content and change in entropy for soap in solutions with soap molecules in a spherical micelle by considering the factors involved in the aggregation process. These data showed that aggregation of the fatty acids in the solutions of the soap was associated with heat changes of similar magnitude; that is, when soap molecules aggregated the ions had little effect on heat content. The heats of wetting were determined for several soaps in an adiabatic colorimeter (Dumanskii & Demchenko—*Doklady Akad. Nauk S.S.S.R. 78,* 277).

In solubilization studies of solutions of various detergents for cyclohexane, *n*-hexane, cyclohexene, and benzene, slight variations were caused by excess acid or base (McBain & Lissant—*J. Phys & Colloid Chem. 55,* 655). Sulfonated castor oil containing 28% moisture was more miscible in aromatic- than in paraffinic-type solvents (Davidsohn—*J. Am. Oil Chemists' Soc. 28,* 84). This behavior was related to the micellar structures of the systems.

Several studies were on the effect of salts on solutions of detergents. With the nonionic detergents made by condensing polyoxyethylenes with fatty alcohols or acids, the salting-out effect of ions increased in the order: lithium, potassium, and sodium, whereas salting-in effect of multivalent cations increased in the order: magnesium, barium, calcium, and aluminum (Doscher *et al.*—*J. Colloid Sci. 6,* 223). Nitrates and chlorides had equivalent effects. Comparable studies with sodium dodecyl sulfate as the surface active material related the salt cation and anion concentrations to the critical concentration for micelle formation (Lange—*Kolloid-Z. 121,* 66). The results were said to agree with the law of mass action. The order of increasing effect of the anions on the decrease of critical concentration for micelle formations was Cl^-, NO_3^-, Br^-, I^-, and SCN^-; which was the same for increasing flocculation. Hobbs (*J. Phys. & Colloid Chem. 55,* 675) discussed the effects of salts on the critical concentration, size, and stability of the soap micelles in the light of the soap-micelle theory of Debye. He suggested that the increase in size of soap micelles upon addition of salt to dilute solutions was related to the ionic strength of solutions of the micelles having surfaces of low charge densities. The effect of salts on the elastic viscous oleate systems were studied by de Jong *et al.* (*Proc. Koninkl. Nederland-Akad. Wetenschlap. 53,* 975, 1122, 1319; *54,* 1) by observations on the elastic modulus, turning point, and reciprocal of the damping. Hydrocarbons had a sparing action on the salt in this system. The minimum concentration of salt required to form an elastic system increased in the order: sodium, potassium, rubidium, and cesium salts. The data in this work was plotted and the effects of various materials were discussed with regard to substances that produced deviations from normal curves. Booij *et al.* (*Ibid. 53,* 1169, 1413) determined the influence of fatty acids, alcohols, and halogen derivatives of paraffins on coacervates of sodium oleate. The results were explained by the manner in which the materials fitted into the coacervate micelle. Their observations on films paralleled the above results. They suggested that film spreading pressure changed because the film either absorbed or squeezed out material. Krishnappa *et al.* (*Proc. Ann. Convention Oil Technol. Assoc India 3,* 6) recorded the maximum spreading obtained with purified "Igepon T" on various salt solutions. With sodium chloride the area spread increased with solutions up to 0.2 *N* concentration and then diminished at higher concentrations. With sodium sulfate solutions the maximum spreading was at a higher concentration, whereas with salts that cause precipitation the area was small.

McHan *et al.* (*J. Phys. & Colloid Chem. 55,* 311) recorded the vapor pressure lowering in terms of osmotic coefficient for aqueous solutions of 19 detergents at various concentrations at 30°. Where the experimental data permitted, thermodynamic properties of the detergents were calculated. Cook (*Ibid.* 383) mathematically interpreted curves of degree of hydrolysis of soap at various concentrations. A model was developed to explain the hydrolysis behavior. In a study pertaining to foaming and hydrolysis of soap solutions, Raison (*Compt. rend, 232,* 1660) found that the foam contained an adsorbed layer of acid soap, and this property of taking up acid by the foam displaced the hydrolytic equilibrium, particularly at low concentrations.

"Polysoaps," which were defined as polymers to whose chain soap molecules were attached, exhibited reduced viscosities in aqueous solutions which were smaller than the parent polymer and polysoap in ethanol (Strauss & Jackson—*J. Polymer Sci. 6,* 649; *7,* 473). These soaps dissolved isooctane proportionally to their concentrations from zero to three percent; rather than requiring a critical concentration as occurs with ordinary soap. This suggested that polysoaps formed micelles at the lowest concentrations. The polysoaps in this work were derived from poly-2-vinyl-pyridine by partial quaternization with *n*-dodecyl bromide. Singleterry & Weinberger (*J. Am. Chem. Soc. 73,* 4754) demonstrated that oil soap micelles in non-polar solvents could be rapidly determined from the depolarization of fluorescence emitted from a dye adsorbed by the micelle. The technique was applicable at much higher dilutions than viscometry, osmometry, or light scattering.

Milligan *et al.* (*J. Phys. & Colloid Chem. 55,* 44) in studies on dehydration isobars of soaps demonstrated that the α-forms existed as hemihydrates, and that β-, δ-, and ω- crystalline phases were not definite hydrates, the water content being desorbed continuously. A study of transitions of soap-oil systems by Doscher & Davis (*J. Phys. & Colloid Chem. 55,* 53) indicated that the stabilization of a sodium stearate-cetane system with water was both to peptize the liquid crystalline aggregates above 117° and to promote the transition from liquid crystalline phase to an intermeshed network of crystalline soap, on cooling the system below this temperature. Neither the presence of cetane nor small quantities of water appeared to change the characteristic transitions of solvent-free sodium stearate.

Some communications pertained to the interaction of surface-active agents and proteins. Abelin & Pfister (*Schweiz. med. Wochschr. 81,* 34) titrated serum proteins with aerosol standard solutions. The results in relation to Kjeldahl values were off from zero to five percent. Two kinds of proteins were precipitated from serum by Zephiran, a cationic soap, by precipitation at pH 7-8 and at pH 4.4, respectively (Polonovski & Macheboeuf—*Ann. inst. Pasteur 74,* 196). Titration with a quaternary ammonium detergent permitted fractionation into four components (Loomeijer—*Nature 166,* 951). The first transmission in the pH curve was caused by α-globulin (pH 4-5), the second by β-globulin (pH 5-7), γ-globulin was precipitated at pH 8.5-9.5, and finally albumin at pH 9.5-10.5. A clear distinction was observed between serums of different protein compositions. Mayer & Eisman (*Proc. Soc. Exptl. Biol. Med. 77,* 452) found that serums from patients with various diseases showed different titration patterns when titrated with cationic detergents and suggested that the reaction may be a helpful clinical test. Bronfin *et al.* (*Proc. Soc. Exptl. Biol. Med. 77,* 456) on using the technique on the serums of 200 patients found heavier than average precipitation in malignant than in nonmalignant diseases; but the test was not regarded as specific.

The interaction of surface-active agents and protein was investigated as a means of inactivating botulinum toxin in bovine plasma (Glassman—*Ann. N. Y. Acad. Sci. 53,* 91). Nonionic surface-active agents were ineffective, whereas others reacted when present in appropriate mass ratio.

The solubilization action of 31 surface-active agents on calf thymus nucleohistone was recorded (Renoll & van Winkle—*J. Am. Chem. Soc. 73,* 2504). Adsorption of the surface active agent, dodecyl sulfate, reduced the rate of denaturation of β-lactoglobulin by heat (Groves *et al.—Ibid.* 2790). This was discussed with regard to association of denaturation with ionization of the amino group. The catalytic effect of alkyl sulfonates on hydrolytic splitting of peptides in dilute acids was also assumed to be due to the effect of the surface active agent on the hydrogen ion activity of the peptides (Hartmann & Hübner—*Z. Elektrochem. 55,* 225).

PERFORMANCE TESTS. Tests on foaming capacity, wetting capacity, laundering, skin compatability, bactericidal effect, etc., are being considered as pertinent to the performance of detergents.

In two new apparatus for measuring foaming capacity of a detergent, foam was produced, respectively, by agitation with a stirrer (Schlachter & Dierkes—*Fette u. Seifen 53,* 207) and by bubbling of a gas (Sinsheimer—*Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 128). The inhibition of foaming of soap solutions by saponin was attributed to conversion of the soap to free oleic acid which displaced the soap from the surface (Shkodin & Tikhomirova—*Kolloid Zhur. 13,* 134).

A new method of evaluating wetting agents was based on densities of skeins of yarn determined in dilute solutions of the wetting agents (Chala *et al.—Proc. Ann. Convention Oil Technol. Assoc., India 2,* 12). The results were comparable with their Herbig numbers.

In investigations on washing test methods, Harris & Brown (*J. Am. Oil Chemists' Soc. 28,* 96) obtained positive correlations between the Launderometer and the Terg-O-Tometer methods with three wash-test soiled fabrics while the results with another test fabric showed low degree of soil removal by the Launderometer test, but gave a satisfactory amount of removal by the Terg-O-Tometer. This communication contained a record of results obtained with soap, built alkylaryl sulfonate, built nonionic, and "loralkyl" sulfate in waters of 50 and 300 ppm. hardness. Collaborative results of testing German laboratory laundering methods were recorded by Machemer (*Fette u. Seifen 53,* 35). In a new detergency testing machine the test cloths were framed into paddles and used as stirrers for the test solution so that uniform contact with washing solution was made (Bernstein—*U. S. 2,568,707*). In a communication on evaluation of washing compounds, Kuckertz (*Fette u. Seifen 52,* 412) suggested that several factors should be considered in determining their efficiencies. He developed an equation in which the efficiencies of compounds were defined in relation to their laundering ability, concentrations giving a defatting effect, concentrations required, and a "threshold value" due to inefficiency below a certain concentration.

Reutenauer & Dupin (*Bull. mens. ITERG 5,* 84) recorded detergency tests on many commercial products as obtained with a Launderometer on fabric soiled according to the method of the official Swiss Laboratory. Stüpel (*Fette u. Seifen 53,* 627) recorded the washing capacities of mixtures of standard soap with several synthetic compounds. With 90% soap and 10% mersolat, lauryl sulfate, Teepol, or alkylaryl sulfonate, washing capacities were at a minimum. In some cases mixtures of two synthetic compounds gave better washing action than either alone; but the results were considered of limited importance for application because presence of builders altered the washing capacities. In a comparison of combinations of fat solvents with surface-active agents for washing wool, by Kroemer & Erhard (*Textile Proxis 6,* 427), those which formed clear solutions were superior to those which formed emulsions.

Data on detergent properties of sodium carboxymethyl cellulose-soap-builder systems were recorded by Vaughn & Kramer (*J. Am. Oil Chemists' Soc. 28,* 317), and by Nieuwenhuis (*Mededeel. Proefsta. Wasend. No. 66,* 10 pp.). The data were pertinent to manufacture of detergent compounds containing carboxymethylcellulose. A record of performance of different types of commercial detergents contained data on detergency, foaming power, Draves test, surface tension, interfacial tension, spreading coefficient, lime soap dispersion, viscosity, corrosiveness, and toxicity (Sanders—*Soap, Sanit. Chemicals 27,* No. 12, 39). The purpose of the work was to develop data to serve as an aid in selecting the wetting agent for a given purpose.

In one report on wool scouring, optimum results were obtained with sodium secondary alkyl sulfates at pH 7–8 and 20-60° with carboxymethylcellulose or tetrasodium pyrophosphate to assist in dirt suspension (Wilkinson—*Dyer 105,* 627, 691). In similar work using soap-soda mixtures as the scouring agents it was found that addition of small amounts of a benzene solution of oleic acid to the scouring mixture permitted grease and wax removal at room temperature (Mansfield—*Australian J. Applied Sci. 1,* 330).

Koehler & Herrmann (*Fette u. Seifen 53,* 146) designed a method for testing laundering compositions for skin compatibility. The appraisal of skin damage by visual, photographic, microphotographic, and light reflection means were discussed. In similar activity, Bober (*Ibid.* 548) determined whether several toilet soaps and detergent powders caused reddening or more intense injury on many subjects and statistically analyzed the results. Like work by Neuhaus (*Ibid.* 552) was based on skin surface fat regeneration after a washing test. The lipide regeneration was significantly better after use of synthetic detergent than after soap base powder. Schwartz (*Soap Sanit. Chemicals 27,* No. 12, 43) in a discussion on cleansers and dermatitis suggested that the better the detergent with regard to wetting, emulsification, and surface activity lowering, the more was it likely to damage the skin.

Ruehhoft & Norris (*Public Health Repts. 66,* 655) correlated various test results with performance of detergents for dishwashing. The factors most significant for each type were as follows: for soap—phenolphthalein alkalinity, total alkalinity, surface tension; for alkaline detergents—pH, inactive alkalinity, surface tension; and for combined detergents—pH, total alkalinity, surface tension, interfacial tension, and emulsification of mineral oil. Certain synthetic detergents were adapted for domestic dishwashing machines by addition of 0.05% potassium pyrophosphate (Sanders & Yeager—*Ind. Eng. Chem. 43,* 866). The combinations were effective dish detergents and caused less filming of glassware than did inorganic alkali products. An investigation on detergents that would remove air-dried milk films, indicated that ethylenediamine and condensed phosphate salts were equally effective whereas other alkaline material gave poor results (Jensen & Claybaugh—*J. Dairy Sci. 34,* 865).

A test for floor cleaner evaluation was based on cleaning artificially soiled panels of light yellowish-white linoleum squares and measuring cleanliness by light reflectance (Maglio—*J. Am. Oil Chemists' Soc. 28,* 267). Trusler (*Soap Sanit. Chemicals 27,* No. 9, 49) investigated such a test for evaluating detergents for washing waxed floors. In this work some commercial detergents removed the wax whereas others did not.

A test method for metal cleaners was based on removal of artificial soil containing radioisotopes; the residual dirt being measured electronically (Harris & Kamp—*Metal Finishing 48,* No. 11, 75). Sodium silicates containing 2.5-5% of certain commercial synthetics were suitable metal cleaners. Hazel & Stericker (*Ind. Eng. Chem. 43,* 919) tested alkaline metal detergents by determining their ability to remove stearic acid from various metal and glass surfaces.

Several communications on performance of detergents pertained to their sanitizing capacity. Among 2-alkylpyridines and 2-alkyl-1-methylpyridinium iodides, maximum antibacterial power was exhibited by the salts of 2-pentadecylpyridines (Birchenough—*J. Chem. Soc. 1951,* 1263). The effectiveness of benzylalkonium chloride as a disinfectant surface-active agent in hard water was demonstrated on several species of bacteria (Dennis—*Soap Sanit. Chemicals 27,* No. 3, 117). Quaternary ammonium germicidal compounds were found to be about one-fifth as active at pH 3.0 as at pH 10.0 (Mueller & Seeley—*Ibid.* No. 11, 131). In this work mono-, di-, and trivalent cations interfered with the disinfectant power in the respective ratio of 1:100:10000. Benigno & Berti (*Farm. sci. e tec, Pavia 6,* 7, 15) measured the inhibiting intensity of various materials on the antibacterial potency of the surface-active agent, cetyltrimethylammonium bromide. It was interesting to note that presence of anionic detergent inhibited the bactericidal action of this cationic material. The anionic detergents also inhibited the fungistatic action of other antiseptics, and in some cases even favored the development of molds (Bolle & Mirimanoff—*J. Pharm. Pharmacol. 2,* 685). In tests on the effect of soaps on Koch's bacillus, those that extracted most wax from the bacillus were most bactericidal (Luzzati—*Compt. rend. 231,* 308).

Practical evaluation tests for dish washing detergents based on measuring bacteria remaining after washing were investigated. Flett & Guiteras (*Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 96) modified the Guiteras test of this type in order to duplicate more closely restaurant conditions. Toennies *et al.* (*J. Lab. & Clin. Med. 38,* 163) did not consider washing of glassware with bacteriologic tests a suitable means for evaluating detergents because cleaning agents adhered very persistently to glass surfaces. Bishop *et al.* (*J. Inst. Brewing 57,* 106) found that 1:12,800 concentrations of didecyldimethylammonium bromide killed 99% of the organisms on beer glasses;

but the cleaned glasses were deleterious on foam retention of beer.

New work on hand soaps containing hexachlorophene antiseptics confirmed their effectiveness for maintaining a low count of resident bacteria on the skin (Browers—*Proc. Chem. Specialties Mfrs. Assoc. June 1950,* 90; Cade—*Ibid.* 92; Blank & Coolidge—*J. Invest. Dermatol. 15,* 257), and demonstrated that these antiseptics did not delay wound healing (Best *et al.*—*Arch. Surg. 62,* 895). Quaternary ammonium compounds as skin cleansers and antiseptics formed a film over the skin under which bacteria were retained (Blank & Coolidge—*J. Invest. Dermatol. 15,* 249).

Sewage processing engineers were concerned with the increased use of synthetic detergents because it may affect conventional methods of treating sewage. Sperry (*Sewage & Ind. Waste 23,* 1469) observed that the following effects were produced by the synthetics: they reduced the amount of suspended solids deposited, thus diminishing removal from tanks; gas to be expected was diminished; grease tended to emulsify; and excessive frothing that occurred might be an annoying nuisance. Degens *et al.* (*Inst. Sewage Purif. J. and Proc. 1950,* 63) found that concentrations of five parts per million of four common synthetic detergents were lethal to tadpoles, sticklebacks, and *Daphnia,* whereas several other fauna and aquatic plants were unaffected. Some of the detergents decomposed under the conditions of the "biological oxygen demand" test. Waddams' (*Ibid.* 32) work on the problem included laboratory bactericidal tests and the reconciliation of results of these with washing practices and sewage treatments. He summarized the work as follows: The presence of 100 ppm. of synthetic detergents in sewage would be most unlikely; at 200 ppm. it did not affect settling of sewage; up to 500 ppm. it did not have bactericidal nor bacteriostatic action on bacteria common to crude sewage; it affected protozoan, *Euplotus patella,* at 100 ppm. only after 60 hours; it had no effect on sludge digestion below 750 ppm.; and flocculation of sewage by alum was not affected.

Rapid Test for Trichloroethylene in Vegetable Oils. Modified Beilstein Test

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A NEW modification of the Beilstein test is recommended for the semiquantitative determination of small amounts of chlorinated solvents in vegetable oils. The test will detect 0.01% trichloroethylene in crude vegetable oils. The technique should also prove valuable in increasing the sensitivity of other applications of the classical copper wire test.

Trichloroethylene is gaining widespread use as a solvent for the extraction of vegetable oils because of elimination of the fire hazard always present when using inflammable solvents. It has the disadvantage of interfering with hydrogenation by reducing the catalyst activity. Amounts as low as 0.005% have been shown to be definitely harmful, and concentrations above 0.03% make the oil very difficult to hydrogenate. A practical maximum limit is felt to be 0.01%.[1]

Only two published methods for trichloroethylene in vegetable oil have been found. Arnold and Hollowell (1) report a sensitivity of 0.02% when using a modification of Fujiwara's method for chloroform. Two drops of the oil sample are added to a hot mixture of sodium hydroxide and pyridine. A red color shows the presence of halides and may be compared to standards. This test is neither as rapid nor as sensitive as the proposed flame test. Eisdorfer and Mehlenbacher (2) collect the solvent by distillation and measure the red color with a spectrophotometer to get quantitative results. The determination requires two hours.

The classical qualitative test for halogen is the Beilstein test which involves heating over a burner a copper wire dipped in the sample. A green flame shows the presence of halogen. Two modifications of the test have been suggested for the determination of volatile organic halides. Ruigh (3) tested for volatile halides by adding a sample dropwise to a warmed bottle, through which the gas supply to the burner passed, and observed the green color near a copper screen in the flame. Stenger, Shrader, and Beshgetoor (4) used a copper strip in the flame to detect methyl bromide vapors in the air.

[1]After this note was submitted for publication, Norris, F. A., Mattil, K. F., and Lehmann, W. J., J. Amer. Oil Chem. Soc., *29,* 28-32 (1952), published results showing that concentrations of trichloroethylene greater than 0.003% retard hydrogenation and that refining and bleaching processes greatly reduce the trichloroethylene content.

Neither of these procedures appeared applicable to crude vegetable oils, and a number of means of uniting the sample and hot copper in a flame were tried. The method adopted as most sensitive employs a 40-mesh copper gauze[2] cut in a strip one-fourth inch by two inches. The oil is applied with a glass stirring rod by spreading along the center of the length of the strip, keeping the edges free of oil. The strip is grasped by crucible tongs at one end and held horizontally in the colorless flame of a burner. A green flame shows the presence of halide.

To determine the sensitivity of the test, known mixtures of trichloroethylene and "expeller crude" soybean oil were made and tested. Results are summarized in the table below:

Trichloroethylene in Oil, Wt. %	Result of flame test
0.1	Bright green flame—persists during burning of oil.
0.05	Green flame—usually disappears before the oil ignites.
0.01	Faint green flash as gauze is put in flame.
0.005	Green can rarely be detected.

The test must, of course, be carried out in air free of halides. Hydrochloric acid fumes, in concentrations which cannot be detected by odor, give a bright green flame with the bare copper.

If the copper strip is dipped in the oil sample, sensitivity is reduced and a 0.05% sample shows the same flame as a 0.01% mixture tested in the suggested manner. The solvent present in unknown samples may best be estimated by dilution with expeller or hydraulic crude soybean oil until the test is comparable to a 0.01% known mixture.

Each analyst using the test should prepare his own calibration by testing known mixtures. Independent tests in different laboratories have verified that 0.01% trichloroethylene is a practical limit to set for the test.

[2]Central Scientific Company, No. 19956-D is satisfactory.

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[Received July 26, 1951]