Letter to the Editor

September 20, 1956

IT HAS BEEN SHOWN recently (1) that alkali catalyzed methanolysis makes possible the preparation of methyl esters from triglycerides in an almost quantitative yield. The method is applicable only to fats with low acid values since free fatty acids not only escape conversion into esters but, by reacting with the catalyst with the formation of water, affect the methanolysis of glycerides. However even fats which require saponification with alkalis can be converted into methyl esters without first isolating fatty acids. Youngs and Craig (2) suggested direct conversion of soaps into methyl esters by double exchange with dimethyl sulphate, but in most cases the refluxing of methanolic soap solution with a calculated quantity of sulphuric acid will also give satisfactory results.

The conventional procedure of separating free fatty acids before the esterification has been apparently based on the assumption that water formed during the acidification of soaps might interfere with ester formation. If however the alkali hydroxide is used in 50% excess, which is ample, only 0.5 mole of water is formed for each mole of fatty acid on acidification, and this additional amount of water is too small to affect the equilibrium during the esterification to any marked extent. The initial acidity of the fat also contributes to the formation of water, but it will be seen that even in the case of 100% fatty acids interference from this source is not serious.

The fats used were steam-rendered lard, refined peanut oil, soybean oil, and commercial oleic acid. Samples of 100 g. of each fat were refluxed for 1 hr. with 30 g. of potassium hydroxide in 400 ml. of methanol. To the soap solution were added slowly 32 g. of sulphuric acid dissolved in 100 ml. of methanol. This amount of acid is sufficient to neutralize the alkali and to provide 1% (w/v) of free sulphuric acid with regard to methanol. After the addition of some porous clay chips the mixture was refluxed for another hour, whereupon 2.5 g. of anhydrous sodium carbonate were added to neutralize about half of the free sulphuric acid. Two-thirds of the alcohol were distilled off. This partial recovery of methanol is, of course, optional. Then 500 ml. of water were added, and the methyl esters were extracted with two successive 250-ml. portions of ethyl ether. The ethereal extracts were washed with 100 ml. of 10% potassium carbonate solution and afterwards with water. From the alkaline washings the fatty acids were recovered by acidification and extraction with ethyl ether. After distilling off the solvent, both methyl esters and recovered fatty acids were dried *in vacuo* and weighed separately. The free fatty acid content of the methyl esters, estimated as usual, was in all cases below 0.1%.

To compare the above method with the conventional procedure 100-g. samples of fats were saponified with ethanolic potassium hydroxide. The fatty acids were separated as usual and refluxed for two hours with five volumes of methanol containing 1% sulphuric acid. No alcohol was recovered. Table I shows that there was little difference in the yields obtained with either method. It appears therefore that, unless the isolation of fatty acids from the soaps is required for some specific purpose, such as separation into solids and liquids, the *in situ* conversion of soaps into methyl esters saves time and solvents without affecting the final results.

TABLE I

Yields of Methyl Esters and Recovered Fatty Acids Expressed as Percentage of Fats Employed

Sample	Conventional procedure		Direct conversion of soaps	
	% Esters	% Fatty acids	% Esters	% Fatty acids
Lard	99.1	0.8	98.8	1.2
Peanut oil	99.3	0.6	99.1	0.7
Soybean oil	99.2	0.6	99.2	0.9
Oleic acid	104.4	0.5	103.0	1.4

REFERENCES

1. Hartman, L., J. Am. Oil Chemists' Soc., *33*, 129 (1956).
2. Youngs, C. G., and Craig, B. M., J. Am. Oil Chemists' Soc., *28*, 521–522 (1951).

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ABSTRACTS . . . . R. A. Reiners, Editor

• Oils and Fats

S. S. Chang, Abstractor
Sini'tiro Kawamura, Abstractor
Dorothy M. Rathmann, Abstractor

Application of thermal diffusion to separation of aliphatic alcohols and fatty acids from their mixtures. C. W. Blessin, C. B. Kretschmer and Richard Wiebe (U. S. Dept. of Ag., Peoria, Ill.). *Anal. Chem.* 29, 408–409 (1957). Very little separation by thermal diffusion occurs in binary mixtures of lower aliphatic alcohols or fatty acids. This is attributed to hydrogen bonding, which obscures structural differences and prevents separation.

Synthesis of lanosterol in vivo. P. B. Schneider, R. B. Clayton, and K. Bloch (Harvard Univ., Cambridge, Mass.). *J. Biol. Chem* *224*, 175–183 (1957). Acetate-C^{14} was injected into rats and the animals were killed 10 or 75 minutes later. The unsaponifiable fractions of the intestines and of the liver were analyzed by chromatography and found to contain squalene and lanosterol of high specific activity. Under the same conditions the presence of an intermediate in the conversion of lanosterol to cholesterol can be demonstrated.

Polarographic determination of antioxidants in gasoline. V. F. Gaylor, A. L. Conrad, and J. H. Landerl (Standard Oil Co., Cleveland 6, Ohio). *Anal. Chem.* 29, 228–231 (1957). Antioxidants in gasoline may be determined directly by polarography by measuring the oxidation waves at a wax-impregnated graphite electrode. Lithium chloride in isopropyl alcohol is used as the solvent-electrolyte. A single sample can be analyzed in 45 minutes. Standard deviation of the method is approximately 9%.

Directed interesterification of lard. C. Placek (Associate Editor) and G. W. Holman (The Procter & Gamble Co., Cincinnati, Ohio). *Ind. Eng. Chem.* 49, 162–169 (1957). Directed interesterification of lard is a solution to the plastic-range problem of lard. The process increases the fraction of high melting solids(trisaturated glycerides), and decreases the fraction of intermediate melting glycerides(disaturated glycerides). This change in glyceride composition accounts for the improved plastic range. Procter & Gamble's Fluffo is made at Cincinnati by directed interesterification. Melted lard is mixed with finely divided sodium-potassium alloy and then rapidly cooled to induce the crystallization of trisaturated glycerides. Further interesterification takes place in the liquid phase, causing additional crystallization. When complete, interesterification is followed by conventional oil and fat refining steps.

Characterization of long-chain fatty acids by infrared spectroscopy. R. A. Meiklejohn, R. J. Meyer, S. M. Aronovic, H. A. Schuette, and V. W. Meloche(Univ. of Wisconsin, Madison, Wisc.). *Anal. Chem.* 29, 329–333(1957). Long-chain fatty acids may be identified by infrared spectrophotometry of the solid compounds from their band progressions in the 7.43- to 8.47-micron region using the potassium bromide disk technique. Number of bands in the progression is approximately half the number of carbon atoms in the chain. Soaps of noncrystalline or somewhat impure fatty acids give sharper spectra than the acids. Infrared spectra of high purity fatty acids, C_{10} to C_{36} are given.

The chemistry of some native constituents of the purified wax of myobacterium tuberculosis. H. Noll(Public Health Res. Inst. of the City of New York, Inc., New York). *J. Biol. Chem.* 224, 149–164(1957). The chemical composition of Wax C from various virulent strains of *Mycobacterium tuberculosis* has been investigated by chromatographic partitioning, combined with infrared spectroscopy. A series of compounds designed as Compounds A_1, A_2, B_1, C_1, and D_1 has been isolated in purified form and characterized by their infrared spectra. The chemical structure of the following compounds has been determined by hydrolytic degradation: Compound A_1 as phthiocerol-dimycoceranate, Compound A_2 as a triglyceride of a straight chain fatty acid C_{22} to C_{26}, and Compound B_1 as an α-monoglyceride of mycolic acid.

Occurrence of n-heptadecanoic acid(margaric acid) in unhydrogenated mutton fat. R. P. Hansen, F. B. Shorland, and N. June Cooke(Fats Res. Lab., Dept. Sci. and Ind. Res., Wellington, New Zealand). *Biochem. J.* 65, 18–20(1957). *n*-Heptadecanoic acid(margaric acid) has been isolated from unhydrogenated mutton fat.

Separation of the C_6–C_{12} Fatty Acids by Reversed-phase partition chromatography. J. B. Wittenberg(Albert Einstein College of Med., Yeshiva Univ., New York). *Biochem. J.* 65, 42–45(1957). The method of reversed-phase partition chromatography has been extended to the separation of the C_6–C_{12} fatty acids. This type of chromatography is applicable to the separation of the terpene acids.

Antioxidants in the food industry. C. H. Lea(Low Temperature Research Station, Cambridge). *Research* 9, 472-8(1956). The deleterious changes in foods exposed to air are reviewed briefly. Antioxidants are discussed with particular emphasis on the tocopherols, gallic acid derivatives, caffeic acid, NDGA, flavonoids, BHA and BHT, metal deactivators, ascorbic acid, and aromatic amines. Permissible maximum concentrations of added antioxidants in fats for use in the United States are listed.

Method of making a bakery shortening. L. Kiers(Hunter Packing Co.). *U. S. 2,779,679.* A liquid-mixture is prepared from water and the lipides extracted from brains, pancreas or spinal tissue. A fat-mixture is prepared by melting together glyceryl mono-and distearate and a glyceryl ester of hydrogenated vegetable oils, and adding glycerine and lecithin. The liquid-mixture is blended into the melted fat-mixture. The resultant smooth paste is mixed with lard in the proportion of 5 to 10% of paste to 90 to 95% lard.

Sediment remover for deep frying. E. W. Kleeman, *U. S. 2,780,984.* A cylindrical trap with a hollow plunger is described for the periodic removal of sediment from the bottom of a deep fat frying kettle.

Manufacture of mayonnaise. D. B. Crawford and C. W. Hirsch (American Machine and Foundry Co.). *U. S. 2,781,270.* A continuous process is described for the manufacture of mayonnaise. A thin flowing mixture is prepared by emulsifying the oil, egg, and part of the vinegar water. This mixture is pumped under an inert gas with the remainder of the vinegar water into a closed chamber when the combination may be vigorously agitated under pressure. Finished mayonnaise is withdrawn continuously into a vessed maintained at atmospheric pressure.

Automatic floating covers for fry kettles. H. N. Shaw. *U. S. 2,781,718.* A motor operated cover for a deep fat frying kettle is described.

Refining vegetable oils. L. P. Hayes and H. Wolff(A. E. Staley Mfg. Co.). *U. S. 2,782,216.* Vegetable oil is degummed by a treatment with water followed by separation of the aqueous phase. The separation is improved if there is added to the oil a small amount of at least one acid anhydride selected from the group: acetic anhydride, propionic anhydride, butyric anhydride, maleic anhydride, succinic anhydride, monomethyl succinic anhydride and dimethyl succinic anhydride.

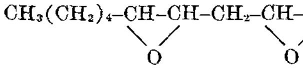
FATTY ACID DERIVATIVES

2-Hexadecenoic acid and related compounds. R. S. Sweet and F. L. Estes(Rutgers Univ., New Brunswick, N. J.). *J. Organic Chem.* 21, 1426–1429(1956). α-Bromo, α-iodo-, and α-hydroxy-palmitic acids and 2-hexadecenoic acid have been prepared and characterized. The infrared absorption spectra of these four acids have been determined.

The synthesis of ω-trifluorostearic acid and ω-trifluoro-n-octadecyl amine. G. Gavlin and R. G. Maguire(Armour Res. Found., Ill. Inst. Tech., Chicago). *J. Organic Chem.* 21, 1342–1347 (1956). ω-Trifluorostearic acid was prepared in an eleven-step program based upon the synthesis of ω-trifluorobutyric acid *via* the intermediates 1,1,1,3-tetrachloropropane and 1,1,1-trifluoro-3-chloropropane, followed by chain-lengthening operations employing reactions between organocadmium compounds and acid chlorides. New compounds isolated pure, as intermediates, were ω-trifluoro-*n*-butyl alcohol, ω-trifluoro-*n*-butyl bromide, methyl 8,8,8-trifluoro-4-ketoöctanoate, ω-trifluoro-octanoic acid, ω-trifluoro-*n*-octyl alcohol, ω-trifluoro-*n*-octyl bromide, methyl 18,18,18-trifluoro-10-ketoöctadecanoate, and ethyl 18,18,18-trifluoro-octadecanoate. Based on 1,1,1,3-tetrachloropropane the yield of ω-trifluorostearic acid was 1.7%. ω-Trifluoro-*n*-octadecyl amine was prepared by reduction of ω-trifluorostearamide.

Use of hydrazine as a reducing agent for unsaturated compounds. II. The hydrogenation of elaidic and ricinoleic acids. F. Aylward and C. V. N. Rao(Borough Polytechnic, Borough Road, London, S. E. 1). *J. Applied Chem.* 6, 559–61(1956). Elaidic and ricinoleic acids were hydrogenated by hydrazine in ethanol at 50°. With an excess of hydrazine (5:1 ratio of hydrazine hydrate to fatty acid) and continuous stirring of the reaction mixture, hydrogenation was over 90% complete in 8 hrs. Under comparable conditions, the rates of hydrogenation of elaidic, ricinoleic and oleic acids were almost identical.

Higher fatty ester diepoxides and process of producing the same. B. Phillips and F. C. Frostick, Jr.(Union Carbide and Carbon Corp). *U. S. 2,779,771.* The compounds claimed have the general formula



$$CH_3(CH_2)_4-\underset{\diagdown O \diagup}{CH-CH}-CH_2-\underset{\diagdown O \diagup}{CH-CH}-(CH_2)_7COOR$$

wherein R is a C_2 or C_3 alkenyl radical.

Preparation of secondary amines. S. H. Shapiro(Armour & Co.). *U. S. 2,781,399.* A mixture of C_8 to C_{22} aliphatic nitriles and amines is preheated with a hydrogenation catalyst until most of the water has been removed. The mixture is then hydrogenated under pressure. Ammonia is retained in the hydrogenation zone until the total amine content becomes constant. The ammonia is then vented and the hydrogenation is continued to completion.

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• Biology and Nutrition

F. A. Kummerow, Abstractor
Joseph McLaughlin, Jr., Abstractor

Antagonism of the inhibitory action of aminosalicylic acid on Mycobacterium tuberculosis by methionine, biotin, and certain fatty acids, amino acids, and purines. L. W. Hedgecock(U. Kansas Medical Center, Kansas City, Kans.). *J. Bacteriol.* 72, 839–46(1956). Inhibition of *Mycobacterium tuberculosis* by *p*-aminosalicylic acid was reversed competitively by *p*-aminobenzoic acid and noncompetitively by methionine and biotin. Methionine and biotin were especially effective when added with C_5 to C_{18} fatty acids, certain amino acids or purines.

Auxotrophic mutants of Mycobacteria dependent on fatty acids derived from the parent strain. J. L. Karlsson(School of Medicine, U. Calif., San Francisco). *J. Bacteriol.* 72, 813–15(1956). Mutants of *Mycobacterium* strain 607 were isolated following exposure of a culture to ultraviolet light. The irradiated culture was spread over an agar plate and then lipides from the parent strain were layered over the bacteria from a dilute solution in petroleum ether. Two mutants were isolated which were dependent for growth on fatty acids extracted from their parent strain. One of these mutants responded to tuberculostearic acid, and the other to hexacosanoic acid.

The phospholipides of fish. J. A. Lovern (Torry Research Station, Aberdeen). *J. Sci. Food Agr.* 7, 729–33 (1956). Phospholipids were isolated from the flesh of cod and haddock. Both phospholipid mixtures were complex, but showed a similar composition. Lecithins comprised 50 to 60% of the total phospholipids. Three unidentified phospholipids are partially described; two of these may be derivatives of *bis*-phosphatidic acid. Three types of inositol phosphatides were detected; these appeared to be particularly resistant to extraction. Phosphatidyl ethanolamine was a minor component. Traces of plasmalogens and, possibly, sphingomyelin were present but phosphatidyl serine was absent. Examination of the fatty acids from the lecithins showed a high proportion of C_{20} and C_{22} polyethylenic acids, but a virtual absence of hexadecenoic acid. A portion of the lecithin was soluble in cold acetone and was found to be more unsaturated than the acetone insoluble lecithin.

The fatty acid composition of milk fats from beef cows fed on different winter rations. G. A. Garton and W. R. H. Duncan (Rowett Research Inst., Bucksburn, Aberdeenshire). *J. Sci. Food Agr.* 7, 734–9 (1956). Four composite milk fat samples from groups of stall-fed beef cows were analyzed by the ester-fractionation technique. Fatty acid compositions are reported in terms of per cent composition and also on a molar basis. The composition of the milk fat from cows fed grass silage was similar to that from animals at pasture. Milk fat from cows fed wholly or mainly on roots (turnips) was somewhat more saturated, due to a higher content of palmitic acid and lower content of oleic acid. Although oleic and palmitic acid contents were markedly influenced by diet, the contents of C_{18} dienoic and trienoic acids were relatively constant.

Nutritive value of leaf protein concentrates. I. Effect of addition of cholesterol and amino acids. S. J. Cowlishaw, D. E. Eyles, W. F. Raymond and J. M. A. Tilley (The Grassland Research Inst., Hurley, Berkshire). *J. Sci. Food Agr.* 7, 768–74 (1956). The value of leaf protein concentrates as supplements to cereal diets for chicks was assayed by the "gross protein value" technique. In all cases concentrates were inferior to casein. The concentrate from lucerne (alfalfa) depressed chick growth; this effect was counteracted by the addition of cholesterol to the diet. The growth-depressant factor was absent from concentrates of Italian ryegrass or white clover. Addition of lysine increased the value of diets containing leaf protein concentrates.

II. Effects of processing methods. *Ibid.*, 775–80. The toxic factor could be removed by washing the lucerne protein concentrates with hot water. No appreciable improvement in the protein value of leaf concentrates was achieved by varying the pH at which the curd was precipitated, by varying drying conditions, or by removal of pigments, waxes, etc. by solvent extraction.

Determination of carotene in silages and forages. H. G. Wiseman, H. M. Irvin, and L. A. Moore (Dairy Husbandry Research Branch, U. S. Dept. of Ag., Beltsville, Md.). *J. Agr. and Food Chem.* 5, 134–136 (1957). A rapid chromatographic method for determination of carotene in silages and forages is described. Necessary conditions for separation of carotene from impurities on magnesium oxide-Celite columns in the presence of small amounts of alcohol have been established. The analysis combines the advantages of splash-free blending extraction afforded by alcohol-Skellysolve B foaming mixtures and the elimination of epiphasic washing to remove alcohol. Additional advantages include faster and more compact elutions gained by presence of alcohol on the column. Direct collections in smaller volumes eliminate concentration and transfer operations.

Studies in vitamin E deficiency. *III.* The estimation of tissue tocopherol with phosphomolybdic acid. H. Rosenkrantz (Worcester Foundation for Experimental Biology, Shrewsbury, Mass.). *J. Biol. Chem.* 224, 165–174 (1957). A sensitive micromethod was developed for the estimation of tocopherols in which phosphomolybdic acid was used. The procedure was found to be relatively specific except for vitamin A substances. Characteristic rates of color development were observed for the various tocopherols. Application to tissue tocopherol measurements was made, and the limitations of the method were evaluated. The reagent can be used to detect minute quantities of tocopherol on paper chromatograms. The nature of the colored product was discussed.

Nutritive value of the proteins of stored instant nonfat dry milk. A. Z. Hodson and C. B. Miller (Research Lab., Pet Milk Co., Greenville, Ill.). *Food Tech.* 11, 89–92 (1957). The protein efficiencies for rat growth of pasteurized milk proteins and those of instant nonfat dry milks stored under favorable conditions for one or two years are similar. The lysine contents of the proteins of pasteurized milk and of properly stored instant nonfat dry milks are similar.

Metabolism of cholestanol. I. Fate of cholestanol-4-C^{14} in the rat. II. Metabolic interrelations of cholestanol, epicholesterol, and Δ^4-cholestenone. F. M. Harold, D. D. Chapman, and I. L. Chaikoff (Univ. of Calif. School of Medicine, Berkeley, Calif.). *J. Biol. Chem.* 224, 609–620 (1957). Evidence is presented which indicates that the conversion of epicholesterol to cholestanol proceeds via Δ^4-cholestenone and cholestanone. A tentative metabolic scheme showing the interrelations of epicholesterol, Δ^4-cholestenone, and cholestanol is proposed.

Isolation of lanosterol from "isocholesterol." J. D. Johnston, F. Gautschi and K. Bloch (Harvard Univ., Cambridge, Mass.). *J. Biol. Chem.* 224, 185–190 (1957). A method is described for the separation of lanosterol from the C_{30} sterol mixture of "isocholesterol" in the form of the sparingly soluble lanosterol dibromide. Acetylation, debromination, and saponification yield pure lanosterol. Small quantities of the structurally related C_{30} sterols of wool fat can be separated by alumina chromatography of the acetates.

On the structure of an intermediate in the biological demethylation of lanosterol. F. Gautschi and K. Bloch (Harvard Univ., Cambridge, Mass.). *J. Am. Chem. Soc.* 79, 684–689 (1957). A new intermediate in the biological demethylation of lanosterol to cholesterol is described. It is isolated by chromatography of the nonsaponifiable fraction of liver and intestinal tissue of rats which had been injected with C^{14}-acetate. Evidence is presented that the intermediate contains a *gem*-dimethyl substituent at C_4, a double bond between C_{24} and C_{25} and an inert nuclear double bond. On enzymatic conversion to cholesterol it yields two moles of CO_2. These properties suggest that the intermediate is a 4,4-dimethylcholestadienol.

Plasma phospholipide synthesis in the eviscerated rabbit. D. B. Zilversmit, Esther L. McCandless, and M. L. Shore (Univ. of Tenn.). *Proc. Soc. Exptl. Biol. & Med.* 93, 542–544 (1956). In the rabbit, plasma phospholipide synthesis continued at a reduced rate in absence of liver, intestine and spleen. Removal of kidney further reduced plasma phospholipide synthesis. The rate of synthesis of kidney, lung or muscle phospholipides from phosphate precursors does not appear to be altered by evisceration.

Methods for measuring fat absorption. H. C. Tidwell and J. M. Johnston (Dept. Biochem., Univ. of Texas, Southwestern Med. Sch., Dallas). *Proc. Soc. Exptl. Biol. & Med.* 94, 150–1 (1957). Gastric retention of a lipid may lead to erroneous conclusions when rates of fat absorption are measured by the generally accepted methods. Separate recoveries of the remaining lipids from the stomach and intestines after an absorptive period permits a correction for the lipid unavailable for absorption in relative rate calculations.

Effect of alloxan, insulin, and thyroxine on cholesterol and fatty acid synthesis by rat liver homogenates. J. F. Scaife and B. B. Migicovsky (Dept. Agriculture, Ottawa, Ontario). *Can. J. Biochem. & Physiol.* 35, 15–24 (1957). The *in vitro* effect of alloxan and insulin on the synthesis of cholesterol and fatty acids from 1-C^{14}-sodium acetate by rat liver homogenates has been examined. Alloxan caused a reduction in the incorporation of acetate into cholesterol, fatty acids, and $C^{14}O_2$, but an increase in the oxygen consumption and carbon dioxide production. The addition of insulin to homogenates caused a reduction in cholesterol synthesis but an increase in fatty acid synthesis both for normal and diabetic animals. Homogenates from thyrotoxic rats exhibited a marked reduction in cholesterol synthesis when compared with normal animals. $C^{14}O_2$ production by homogenates from starved rats was appreciably lower than for those from normal animals.

Studies on the sea anemone Gyrostoma sp. Lipids of Gyrostoma sp. M. V. Rajagopal and Kamala Sohonie (Dept. Biochem., Inst. Sci., Bombay, India). *Biochem. J.* 65, 34–36 (1957). The concentrations of cerebrosides, free and total cholesterol, total phospholipid, lecithin and sphingomyelin have been determined in the whole anemone, *Gyrostoma* sp., as well as in its mesenteries, gonads and tentacles.

Metabolism of short-chain fatty acids in the sheep. R. J. Pennington and W. H. Pfander (Rowett Res. Inst., Bucksburn, Aberdeenshire). *Biochem. J.* 65, 109–111 (1957). Some interrelationships exist in the metabolism of fatty acids and glucose by sheep-rumen epithelial tissue. Butyrate lowered the uptake of propionate by sheep-rumen epithelium, and acetate lowered the uptake of butyrate. Ketone-body production from acetate was suppressed by propionate or glucose. Some acetate was produced when propionate and butyrate were metabolized together.

Increased metabolism in fat-deficiency: relation to dietary fat. T. C. Panos, J. C. Finerty, and R. L. Wall(Univ. of Texas Med. Branch, Galveston). *Proc. Soc. Exptl. Biol. & Med.* 93, 581–584(1956). Disturbance in energy metabolism, as indicated by increased basal oxygen consumption and growth retardation, is prevented when rats receiving a fat-free diet are given daily supplements of methyl linoleate(100 mg.) or cottonseed oil (200 mg.). However, similar supplementation with a saturated fatty acid(methyl palmitate, 100 mg.) in no way modifies the development of the fat-deficiency syndrome.

Prefeeding of High Fat Diet and Resistance of Rats to Intense Cold. J. LeBlanc(Army Chem. Med. Labs., Md., U.S.A.). *Can. J. Biochem. & Physiol.* 35, 25–30(1957). Three groups of 16 albino rats were fed for 45 days: group I, a normal diet of pellets containing 3.5% fat; group II, a diet containing 17% fat in the form of oil; and group III, a diet containing 17% fat in the form of lard. On exposure to cold, the drop in rectal temperature in group I was faster and more pronounced than in group II or group III. It was shown that the larger amount of fat accumulated in the animals fed a high fat diet could not explain, either as a source of energy or as an insulator, the superiority of these diets in maintaining the rectal temperatures at higher levels in the cold. It is postulated that prefeeding of a high fat diet induces changes in the organism which permit higher sustained rates of heat production in the cold.

Effect of dietary fat on requirement of vitamin B_{12} by the chick. M. R. Spivey Fox, Legia O. Ortiz, and G. M. Briggs (Nat. Inst. Health). *Proc. Soc. Exptl. Biol. & Med.,* 93, 501–504(1956). Incorporation of 20% fat into a corn-soybean oil meal diet increased 10- to 20-fold the vitamin B_{12} requirement of non-depleted chicks for optimal growth at 4 weeks of age. The requirement was between 0 and 5 γ per kg. of diet(3% fat) and between 50 and 100 γ with the high fat diet. The high dietary fat did not deplete the chick of its vitamin B_{12} liver store or alter the absorption of dietary vitamin B_{12} and its subsequent storage in the liver.

Fractionation of plasma non-esterified fatty acids. V. P. Dole (Hosp. of the Rockefeller Inst., New York City). *Proc. Soc. Exptl. Biol. & Med.* 93, 532–33(1956). Palmitic and oleic acids are the predominant non-esterified fatty acids of human plasma. Their concentrations fall proportionately after an injection of insulin.

Rape oil and cholesterol metabolism in different species with reference to experimental atherosclerosis. K. K. Carroll(Univ. Western Ontario). *Proc. Soc. Exptl. Biol. & Med.* 94, 202–205 (1957). Rape oil caused a marked increase in adrenal cholesterol when fed to rats or mice but had little or no effect in rabbits, guinea pigs, chickens and dogs. This contrasts with results obtained by feeding cholesterol since dietary cholesterol tends to be deposited more readily in the latter four species. The differences in response to rape oil do not appear to be due to species differences in digestibility of rape oil.

Browning in phosphatide-containing fat emulsions. R. R. Benerito, Katherine M. Formusa, J. L. White, and W. S. Singleton (Southern Regional Res. Lab., New Orleans). *Proc. Soc. Exptl. Biol. & Med.* 94, 47–51(1957). This investigation gives experimental evidence that at autoclaving temperature (121°) a type of browning occurs which involves reaction of dextrose with lecithins as well as with amino-groups of cephalins. Therefore, use of pure lecithin as an emulsifier in an oil emulsion containing dextrose in the water phase would not eliminate formation of the brown material. The complex browning reaction is responsible for formation of a colloidal material found in phosphatide-containing fat emulsions prepared for intravenous alimentation, and might be a possible explanation of certain adverse physiological results occasionally experienced with the use of these emulsions.

• Drying Oils and Paints

Raymond Paschke, Abstractor

Method of producing metal resinates. J. Drew and C. S. Lampo (Crosby Chemicals, Inc., DeRidder, La). *U. S. 2,753,330.* This patent covers the improvement in the fusion process of making a non-gelling, high melting point, metal resinate from rosin, having a metal content in excess of 3.5% of the weight of rosin, including the steps of reacting rosin material with a compound of the type capable of forming a rosin adduct by the Diels-Alder reaction and directly reacting said rosin adduct in a molten state with a reactive compound of a metal of the group consisting of zinc and calcium, capable of yielding its metal to the rosin.

Process for the preparation of copals for use in the production of lacquers and varnishes. L. Lacroix(Gino Vuagnat, Geneva, Switzerland). *U. S. 2,753,331.* This patent covers the method of refining a raw natural copal resin, which comprises the steps of dissolving said resin at an elevated temperature not higher than 100° in an organic solvent medium selected from the group consisting of butyl alcohols and mixtures of butyl alcohols with benzene. The solvent medium having a boiling point of about 100°, reducing the hot solution with a reducing agent selected from the group consisting of hydrogen and formaldehyde, filtering the reduced solution and removing the solvent medium by distillation.

Modified synthetic drying oil. A. H. Gleason(Esso Res. and Eng. Co., Delaware). *U. S. 2,753,385.* This patent covers a process for preparing a drying oil which comprises copolymerizing at 40–90° a mixture of from 75 to 85% of butadiene and 25 to 15% of styrene in 150 to 300 parts of a hydrocarbon diluent per 100 parts of monomers and 10 to 40 parts of an ether chosen from the group consisting of mono- and diethers having 4 to 8 carbon atoms and having on O-C-C-O group in the molecule. The catalyst is 1.2 to 4 parts of finely divided metallic sodium based on monomers. Polymerization is continued until substantially 100% conversion is obtained whereby a product is obtained which has a viscosity somewhat less than that ultimately desired. The polymerization is stopped by killing and removing the catalyst, and the product is heated at a temperature between 100 and 175° in the presence of an organic peroxide until a drying oil having the desired viscosity is obtained.

Tall oil esterified with mixture of epoxy resin and polyhydric alcohol used as an ink vehicle. J. W. McNabb(American Can Co., New York). *U. S. 2,754,277.* This patent covers a lithographic ink vehicle comprising about 39 to 40 per cent by weight based on the vehicle of chinawood oil, and about 60 to 61 per cent of a mixed ester of tall oil. The tall oil is esterified with a mixture of alcohols consisting of (1) a resinous polyalcohol resulting from the reaction of bis-(4 hydroxyphenyl)2,2-propane and an epihalohydrin, said resinous polyalcohol having a molecular weight of about 2,500 and being present in a concentration of about 9 per cent by weight based on unreacted ester forming materials and (2) about 10.7 per cent by weight of pentaerythritol.

Drying oils. A. R. Hempel and P. E. Marling(Monsanto Chemical Co.). *U. S. 2,754,307.* This patent covers a drying oil comprising the pentaerythritol ester of an adduct of fumaric acid and a non-conjugated, unsaturated, non-hydroxylated fatty oil having from 10–24 carbon atoms in the carbon chains of the fatty acids of the oil, said adduct being obtainable by heating the fatty oil with from 3 per cent to less than 10 per cent by weight of fumaric acid.

Method of bodying oils. O. J. Grummitt(Sherwin-Williams Co., Cleveland). *U. S. 2,754,308.* This patent covers a process for bodying vegetable oils which comprises heating a vegetable oil selected from the group consisting of drying and semidrying vegetable oils with from 0.5 to 10% of acetic anhydride at a temperature of from 140° to 180° for a period of time of from 0.5 to 20 hours, and then blowing a body of oil so treated with air.

Preparation of epoxidized acids, esters, and nitriles. E. F. Riener(Rohm and Haas Co., Philadelphia, Pa.). *U. S. 2,756,242.* This patent covers a process for preparing epoxidized acids, nitriles and esters which comprises reacting a strong base with a member of the class consisting of saturated aliphatic nitriles containing 16 to 22 carbon atoms, saturated aliphatic acid containing 16 to 22 carbon atoms and esters of said acids, said member also containing groupings having the structure H-CHX-CH(OCOR)- in which X is an atom of a halogen from the class consisting of chlorine and bromine, and R is a member of the class consisting of a hydrogen atom and a methyl group.

Process for the preparation of copolymers from linseed oil and styrene and the copolymers thus prepared. W. J. Taat(Naamloze Vennootschap Scado Dunstharsindustrie, Zwolle, Netherlands). *U. S. 2,761,850.* This patent covers a process of copolymerizing linseed oil with styrene to form clear polymers which dry to clear films, comprising the steps of heating linseed oil at a temperature above 200° with 0.05–0.2% by weight of selenium; and copolymerizing the selenium-treated linseed oil with styrene to form a clear copolymer of linseed oil and styrene which upon drying forms a clear film.

Polymerization of aliphatic conjugated diolefins. C. R. Greene (Standard Oil Co., Chicago). *U. S. 2,762,790.* This patent covers a process for polymerizing an aliphatic conjugated diolefin, which process comprises contacting said aliphatic conjugated diolefin in the presence of a liquid hydrocarbon reaction medium with catalytically effective amounts of the hydride of an alkaline earth metal and an oxide of a metal of group 6a of the Mendeleef periodic table, the ratio of said hydride to metal oxide catalyst being between about 0.1 and about 5 by weight, at a polymerization reaction temperature between about 100° and about 300° under a pressure sufficient to maintain a liquid phase, for a period of time sufficient to effect substantial polymerization of said aliphatic conjugated diolefin, and separating a polymer thus produced.

Butadienoid drying oil and process for preparing same. A. H. Gleason(Esso Res. and Eng. Co., Delaware). *U. S. 2,762,851.* This patent covers a polymerization process which comprises mixing 75 to 85 parts of butadiene-1,3 and 25 to 15 parts of styrene, 100 to 500 parts of an inert hydrocarbon diluent boiling between —10° and 200°, 10 to 45 parts of a co-diluent selected from the group consisting of open-chain ethers having 4 to 8 carbon atoms wherein the two oxygen atoms are separated by at least two carbon atoms, and 1 to 3 parts of finely divided sodium; and maintaining the resulting mixture at a temperature between 25 and 95° until 100% conversion of the monomers is reached.

Manufacture of epoxide resins. J. E. Masters(Devoe & Raynolds Co., Louisville). *U. S. 2,767,157.*

Stabilizing drying oils. A. H. Gleason(Esso Res. & Eng. Co.). *U. S. 2,767,229.* This patent covers a process for preparing a drying oil which comprises copolymerizing a mixture of from 75 to 85% of butadiene and 25 to 15% of styrene in 200 to 300 parts of a hydrocarbon diluent per 100 parts of monomers and 20 to 30 parts of a member of the group consisting of aliphatic ethers of 4 to 8 carbon atoms and cyclic diethers of 4 to 8 carbon atoms, in the presence of 1 to 5 parts of finely divided sodium at a temperature between 40 and 85°, continuing the polymerization until substantially 100% conversion is reached and a product having an intrinsic viscosity between 0.05 and 0.4 is obtained, stopping the polymerization by killing and removing the catalyst and then heat bodying the product at a temperature between 150° and 275° in the presence of 0.001 to 0.3% of an alkylated phenol.

Color improvement in the preparation of drying oils. J. K. Mertzweiller(Esso Res. and Eng. Co.). *U. S. 2,768,984.* This patent covers a process for preparing a substantially colorless synthetic drying oil which comprises heating to 25 to 90° a mixture of 75 to 85 parts of butadiene, 25 to 15 parts of styrene, 200 to 300 parts of hydrocarbon diluent and 20 to 30 parts of an ether selected from the group consisting of aliphatic ethers of 4 to 8 carbon atoms and cyclic diethers of 4 to 8 carbon atoms in the presence of finely divided sodium at a tempehature between 40 and 85° until substantially complete conversion is obtained, then continuing the heating in the presence of the unused sodium at 50 to 120°, for 0.5 to 1.5 hours.

• Detergents

Lenore Petschaft Africk, Abstractor

Determination of trace amounts of alkyl benzenesulfonates in water. Assoc. Am. Soap and Glycerine Producers, Inc. *Anal. Chem.* **28**, 1822–6(1956). An analytical subcommittee of the AAS&GP. Inc. has cooperatively developed a specific quantitative infrared procedure for the determination of trace concentrations of alkyl benzenesulfonate in raw and treated water supplies. Such a method is needed because of the lack of specificity in procedures currently used in analyzing most household synthetic detergents. The method involves concentration of alkyl benzenesulfonate through adsorption on activated carbon, desorption, removal of interfering materials through several purification steps, and final quantitative estimation with infrared spectrophotometry.

Role of interfacial electric conditions in detergency. K. Durham(Unilever Ltd., Port Sunlight, Engl.). *J. Appl. Chem.* (London) **6**, 153–61(1956). A detergent system is treated as a lyophobic colloid system, and the interaction between soil particles and fibers in an electrolyte solution is represented by the superposition of the potentials due to London-van der Waals attraction, the Born repulsion, and double-layer repulsion. The potential energy curves show the effect of electrical forces in soil removal and redeposition. The particle size of the soil and the concentration and valence of the electrolyte are important parameters that determine the effect of interfacial electrical conditions in detergency. Easy soil removal is incompatible with a high degree of stability against redeposition. (*C. A.* **51**, 805)

The electrolytes and soap. A. Finizia(Society PURO, Istambul, Turkey). *Indian Soap J.* **22**, 104–111(1956). The effect of the concentration of electrolytes on the final composition of soap and during the different phases in the pan operation(separation, boiling and liquidation) is described.

Measuring relative abrasive characteristics of household cleanser. C. A. Gerardi(United States Testing Co., Hoboken, N. J.). *Soap & Chem. Specialties* **33**(2) 47–50(1957). A new procedure for measuring the abrasiveness of household scouring cleansers is based on determining an abrasion index which measures the amount of gloss retained on a glass panel abrasion by the cleanser being tested under standardized conditions using a Ro Tap machine.

Synthetic anion-active detergents. A. I. Gershenovich. *Khim. Nauka i Prom.* **1**, 298-311(1956). A review of principal synthetic anion-active detergents: salts of synthetic carboxylic acids: primary and secondary alkyl sulfates; alkylbenzenesulfonates, alkanesulfonates, based mostly on American and German sources. It includes chemical, technological, and statistical data, and production flow sheets of these 4 products. (*C. A.* **51**, 1629)

The corrosion of metals occasioned by aqueous solutions of some surface-active agents. IV. Aluminum. H. Holness and R. D. Langstaff(S. W. Essex Tech. Coll., London). *J. Appl. Chem.* (London) **6**, 115–24(1956). The experimental technique and surface-active agents in distilled and tap water were the same as those used in earlier experiments. Several of the anionic agents produced a water-repellent and protective coating on the Al; where attack occurred it was of the pitting type. Of the cationic agents, triethanolamine was the most active, producing uniform attack. Weight losses were negligible with the other 2 cationic agents. The nonionic agent had little effect. Many of the specimens in tap water solutions acquired a deposit of $CaCO_3$ crystals. (*C. A.* **50**, 15397)

Development problems of chemical concerns supplying detergent alkylate. J. C. Kirk and E. R. Baker(Continental Oil Co., Ponca City, Oklahoma). *Soap & Chem. Specialties* **33**(2) 51–3, 118–19(1957). The requirements of alkylates as used by detergent manufacturers are reviewed. Manufacturers have to control the boiling range of commercial dodecylbenzenes, improve sulfonation and neutralization techniques. The increase in manufacture of liquid detergents has resulted in change in sulfonation procedure. Methods of evaluating these alkylates in finished products are described.

The production of toilet soap containing 80% fatty acids. L. A. Klepikov. *Masloboino-Zhirovaya Prom.* **22**(6) 17–18 (1956). At the end of 6-month open-shelf storage both the moisture-caused swelling of toilet soap and its loss in weight were significantly lower when the soap contained 80 instead of 72% fatty acids. (*C. A.* **51**, 1630)

Secondary alkyl sulfates as washing substances. A. Ju. Rabinovich and E. S. Skripchenko. *Masloboino-Zhirovaya Prom.* **22** (6) 19–20(1956). Data are presented to show that the secondary alkyl sulfates with satisfactory surface-active properties could be derived from a raw material made up of distillate from phenol-free shale oil and olefin fractions C_{10}–C_{14}, C_{14}–C_{16}, C_{16}–C_{18}. (*C. A.* **51**, 1630)

Improvements in dishwashing detergents. Atlas Powder Co. *Brit. 762,154.* An improved dishwashing detergent for automatic dishwashers consists of 50–70% alkali metal carbonate, 10–30% of a sequestering phosphate, 10–20% of a soluble alkali metal silicate, 2–10% Na aluminate and 1–5%(all based on weight) of a non-ionic surface active agent or synthetic anionic detergent.

Improvements in detergent compositions. B. J. Hazzard(Unilever Ltd.). *Brit. 762,472.* A non-tarnishing detergent composition contains a synthetic detergent and as an anti-tarnishing agent, a substance which in the hydrated form has a specified X-ray powder diffraction pattern, and is obtained by heating dicyandiamine, ammonium thiocyanate or melamine to 150–330°.