

This note describes conditions under which approximately 74% of the vernolic acid in *Vernonia anthelmintica* seed oil can be recovered in the mixed acids obtained upon saponification. A procedure for isolation of pure vernolic acid is also described. Variations of the fractionation procedure were not attempted but could probably be improved to increase recovery of pure vernolic acid.

It was found that the reactive epoxy group could be preserved after saponification of the oil with ca. 1 *N* ethanolic potassium hydroxide, provided the alcoholic alkali after completion of refluxing was rendered only very slightly acidic (pH 4–5) and extraction was carried out immediately. Concentration and purification of vernolic acid were then effected by a sequence of low-temperature crystallization, urea complex fractionation, solvent partitioning, and a final recrystallization. The course of the purification was followed by Durbetaki's method for determining oxirane oxygen (6). The purified acid was a poorly crystalline solid, m.p. 25–28°.

### Experimental

**Oil Extraction.** Coarsely ground, air-equilibrated seeds of *Vernonia anthelmintica* (Willd.) (76.6 g.) were extracted over-night in a Soxhlet apparatus with 30–60° petroleum ether. The bulk of the solvent was evaporated under an atmosphere of nitrogen, and the balance was removed *in vacuo* by means of a rotating evaporator. A yield of 17.9 g. of greenish oil (I) was obtained containing 3.5% oxirane O; infrared showed maxima at 11.85, 12.17  $\mu$  (med. intensity).

**Saponification.** A mixture of 6.3 g. of I, 1.5 g. of potassium hydroxide in 2 ml. of water, and 18 ml. of 95% ethanol was refluxed for 30 min. The solution was then cooled, diluted with water, and extracted with ether to remove unsaponified matter; the yield of such material was 0.4 g. The alkaline liquor was then carefully acidified to pH 4–5 by dropwise addition of dilute hydrochloric acid and was immediately extracted three times with ether. The combined ether extracts were dried with sodium sulfate and evaporated under nitrogen, yielding 4.1 g. of mixed fatty acids (II) containing 4.0% oxirane O.

**Low Temperature Crystallization.** A solution of 4.1 g. of II in 40 ml. of 30–60° petroleum ether was cooled for 3 hr. at –10° with occasional stirring. The super-

natant liquid was then withdrawn with a fritted glass filter stick. The solid material was dissolved in petroleum ether, and the process was repeated three times, yielding concentrate III containing 4.6% oxirane O. The over-all yield in these successive recrystallizations was 57%.

**Urea Complex Fractionation.** A 1.56-g. portion of III was dissolved in 30 ml. of absolute methanol containing 4 g. of urea and let stand over-night at room temperature. After the solution had been chilled at 0° for 2½ hr., a crop of crystals appeared that was removed by filtration. The yield of urea adduct (IV) was 1.98 g., m.p. ca. 102° dec. IV was decomposed with water, and the resulting aqueous mixture was extracted with ether twice. The combined ether extracts were washed with water, dried with sodium sulfate, and evaporated. A yield of 0.47 g. of fatty acid concentrate V, containing 4.8% oxirane O, was obtained.

**Hexane-Acetonitrile Solvent Partitioning.** A 0.35-g. portion of V was dissolved in 10 ml. of *n*-hexane and extracted with 20 ml. of acetonitrile. The acetonitrile layer was then re-extracted twice with hexane. Upon evaporation, the acetonitrile solution yielded 0.22 g. of vernolic acid. After recrystallization from hexane at –20° the acid was a poorly crystalline solid, m.p. 25–28° (Fisher-Johns melting point apparatus).

**Anal.:** Calcd. for C<sub>18</sub>H<sub>32</sub>O<sub>3</sub>: C, 72.9; H, 10.9; oxirane O, 5.4. Found: C, 73.0; H, 10.7; oxirane O, 5.4.

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## ABSTRACTS . . . . R. A. REINERS, Editor

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### • Fats and Oils

DEVICE FOR ISOLATION OF COMPONENTS SEPARATED BY GAS CHROMATOGRAPHY. J. M. Lesser (Barrett Division, Allied Chemical Corp., Philadelphia, Pa.). *Anal. Chem.* 31, 484 (1959). When the desired peak appears on the chromatogram, the collector is attached via the metal connector to the effluent gas exit, care being taken that both holes are open. After the elution of this component, the collector is removed and sealed at both ends with suitable plugs. Additional collectors can be used to collect other components from the same run.

INFRARED DETERMINATION OF HYDROXYL EQUIVALENTS IN STEROIDS. P. Kabasakalian, E. R. Townley, and M. D. Yudis (Res. Division, Sechering Corp., Bloomfield, N. J.). *Anal. Chem.* 31, 375–6 (1959). The number of hydroxyl groups contained in a

steroid molecule can be determined by using the fundamental hydroxyl stretching absorption in the infrared region. Hydrogen bonding and solubility problems which have heretofore complicated the measurement have been overcome by selecting pyridine as the solvent. The absorbance is linear with concentration for the associated band which appears near 3.05 microns and is essentially independent of the type of hydroxyl group. GAS-LIQUID CHROMATOGRAPHY. DETERMINATION OF COLUMN EFFICIENCY. H. W. Johnson, Jr., and F. H. Stross (Shell Development Co., Emeryville, Calif.). *Anal. Chem.* 31, 357–65 (1959). Gas-liquid chromatography (GLC) can be used to determine physical constants as well as for quantitative analyses. For the latter, only peak areas need be accurately known, but accurate determination of physical constants, such as par-

tion and activity coefficients, requires that certain corrections be applied to peak position and width for detector volume.

**RADIATION PRESERVATION OF MILK AND MILK PRODUCTS.** V. PRECURSORS TO THE RADIATION-INDUCED OXIDATION FLAVOR OF MILK FAT. J. E. Hoff, J. H. Wertheim, and B. E. Proctor (Dept. of Food Technology, Massachusetts Inst. of Technology, Cambridge). *J. Dairy Sci.* 42, 468-75 (1959). Oxidation flavor produced upon irradiation of milk fat is derived from certain of its component fatty acids. One of these acids has been identified as linolenic acid. Other acids required for production of irradiation-induced oxidation flavor have been isolated in an unidentified mixture. This unknown isolate is believed to contain the vinyl group and branched chains. Linolenic acid produced a fishy flavor upon irradiation while the mixture of fatty acids containing the vinyl group and branched chains gave rise to a flavor characterized as candle-like. These two flavors blended upon mixture to give the typical tallowy oxidation flavor of irradiated milk fat.

**FOOD COMPOSITION AND METHOD OF PRESERVATION.** R. T. Holman (Regents, Univ. Minnesota). *U.S.* 2,875,060. Prior to incorporation into food compositions, essential fatty acid compounds are reacted with urea to form urea-inclusion products. These aid in inhibiting autoxidation and rancidification in food compositions containing the essential fatty acids and oil-soluble vitamins.

**FLUID SHORTENING.** S. W. Thompson (Lever Brothers Co.). *U.S.* 2,875,065. A stable fluid shortening is prepared from an edible oil and 3 to 8% by wt. of a saturated long-chain fatty acid triglyceride containing at least one behenyl radical per molecule.

**RAPID DIRECTED INTERESTERIFICATION OF GLYCERIDES.** G. W. Holman and L. H. Going (The Procter & Gamble Co.). *U.S.* 2,875,066. A process for the low-temperature directed interesterification of mixtures of triglycerides comprises (a) rapidly chilling the mixture so as to cause supercooling and the sudden formation of a cloud of extremely minute crystals of the higher melting triglycerides throughout the flowing stream but without accumulation on the vessel walls, (b) effecting directed rearrangement by continuous agitation of the mixture with a uniform dispersion of an active low-temperature interesterification catalyst so as to facilitate continued crystallization of high-melting triglycerides, and (c) inactivating the catalyst while the temperature of the mass is still at least 3° F. below the maximum to which it would rise if the rearrangement were continued under adiabatic conditions.

**LARD SHORTENINGS.** G. W. Holman and L. H. Going (The Procter & Gamble Co.). *U.S.* 2,875,067. A plastic shortening is prepared from at least 90% of a low-temperature directed rearranged and partially hydrogenated mixture consisting of at least 80% lard and no more than 20% tallow. The remainder of the shortening consists of mono-, di-, and triglycerides of lard, edible tallow, randomly rearranged lard, palm oil, partially hydrogenated cottonseed oil, corn oil, soybean oil, sunflower seed oil, linseed oil and peanut oil, or mixtures of any of these.

**HYDROGENATION OF FAT.** J. W. Bremer, Jr. (Swift & Co.). *U.S.* 2,875,220. In a continuous process for the hydrogenation of a liquid fat, catalyst is removed from the product by magnetized grids and later released to a fresh portion of oil.

**DRY RENDERING OF FATS.** H. C. Dormitzer (Wilson & Co., Inc.). *U.S.* 2,875,232. In a process for the dry rendering of packing-house materials, these are cooked to free the fat. The mixture is centrifuged while hot so as to separate most of the fat. The cracklings are pressed and the fat so obtained is mixed with freshly rendered material prior to centrifugation.

**DEEP FAT FRYING MACHINE.** J. W. Smoot and J. J. Smoot. *U.S.* 2,875,682.

**PROCESS FOR REFINING FATTY OILS.** B. J. Thurman (Benjamin Clayton). *U.S.* 2,876,242. A process for the alkali refining of glyceride oils is described in which the crude oil is mixed with only a slight excess of soda ash so that evolution of carbon dioxide is prevented. The carbon dioxide is then removed from the mixture without dehydration. Finally, soapstock is separated from the refined oil.

**FATTY ACID RECOVERY FROM SOAP STOCK.** T. H. Kelley (The Buckeye Cellulose Corp.). *U.S.* 2,877,249. Vegetable oil soap stock is mixed with a water-insoluble solvent such as an aromatic hydrocarbon, halogenated hydrocarbon or petroleum naphtha. The pH of the mixture is adjusted to 3.0 to 8.5 and the mixture is agitated at a temperature below its boiling point.

Fatty miscella are separated from the aqueous phase, and the solvent is removed.

**FOOD PRODUCT.** J. G. Roberts. *U.S.* 2,878,126. A modified cream is prepared to contain 22 to 28% butterfat, 15 to 16% non-fat milk solids and no more than 60% water.

**NEUTRALIZING AND DECOLORIZING LARD INTERESTERIFICATION REACTION MIXTURE.** L. A. Van Akkeren and H. J. Ast (Swift & Co.). *U.S.* 2,878,274. For the interesterification of lard, sufficient alkali metal alcoholate is added to react with the free fatty acids to form soaps and to furnish an excess of catalyst. After modification of the lard is completed by heating, acid is added in an amount sufficient to neutralize excess alkali but not to decompose the soaps which occlude color bodies.

## • Fatty Acid Derivatives

**REACTIONS OF UNSATURATED FATTY ALCOHOLS. IV. OXIDATIVE DEGRADATION OF LAURYL ISOPROPYL ETHER.** L. E. Gast, C. B. Coleman, and H. M. Teeter (Northern Utilization Research and Development Div., Agricultural Research Service, U.S. Dept. of Agriculture). *J. Organic Chem.* 24, 160-65 (1959). Lauryl isopropyl ether containing catalytic amounts of cobalt naphthenate was degraded with oxygen at 150°. Degradation products were separated by distillation, solvent extraction, and chromatography. Isolated oxidation products indicate attack at both carbon atoms adjacent to the ether oxygen. Degradation products isolated were lauric acid, acetone, isopropyl laurate, lower fatty acids from C<sub>2</sub> to C<sub>11</sub> and their esters, carbon dioxide, isopropyl alcohol, and polymerized acidic products. Lauric acid and isopropyl laurate were oxidized under the same conditions as lauryl isopropyl ether. Lauric acid gave small amounts of lower acids in the homologous series as degradation products. Isopropyl laurate was oxidized to fatty acids from C<sub>2</sub> to C<sub>12</sub> in about one third the quantities found from lauryl isopropyl ether.

**METHOD FOR PREPARING ADHESIVE COATING COMPOSITIONS.** G. D. Duro (The Harshaw Chemical Co.). *U.S.* 2,875,081. A gel suitable for use in adhesives is prepared from a saturated higher fatty acid, an aluminum triacoholate and microcrystalline wax.

**AMIDES.** J. Dazzi (Monsanto Chemical Co.). *U.S.* 2,875,218. A process is described for the preparation of N, N, N', N'-tetraalkyl diamide of dimeric linoleic acid.

**PROCESS FOR PREPARING MONOGLYCERIDES OF FATTY ACIDS.** H. Birnbaum (Hachmeister, Inc.). *U.S.* 2,875,221. A continuous process is described for the preparation of glycerides containing a high proportion of monoglyceride by the reaction of glycerol and a polyglyceride of a fatty acid in the presence of caustic soda at temperatures between 475 and 525° F. A portion of the product is recycled. The remainder is acidified with concentrated phosphoric acid and rapidly cooled. Removal of unreacted glycerol and the precipitated sodium phosphate yields the desired product.

**PREPARATION OF UNSATURATED HIGHER ALIPHATIC ALCOHOLS.** F. F. A. Braconier, H. Le Bihan, and R. Arnould (Soc. Belge de l'Azote et Prod. Chim. du Marly). *U.S.* 2,876,265. A method is described for the production of unsaturated fatty alcohols by continuous hydrogenation of high molecular weight unsaturated aliphatic carboxylic acids or their esters in the presence of a catalyst consisting of chrome oxide, zinc oxide, and aluminum oxide.

**UNSATURATED ACYL ISOTHIOCYANATES.** G. Nischk and H. Holt-schmidt (Farbenfabriken Bayer Akt.). *U.S.* 2,877,244. A process is described for the preparation of isothiocyanates by the reaction of halides of ethylenically unsaturated fatty acids with inorganic thiocyanates.

**PREPARING ANHYDRIDES OF HIGH MOLECULAR WEIGHT LONG-CHAIN FATTY ACIDS.** J. Nichols and E. S. Schipper (Ethicon, Inc.). *U.S.* 2,877,247. Equimolar quantities of a long-chain fatty acid and a lower alkyl chloroformate dissolved in an inert organic solvent are reacted at temperatures below 0° in the presence of an equi-molar amount of a lower aliphatic tertiary amine. The resultant acylalkylcarbonate is reacted with a tertiary alkylamine salt of a long-chain fatty acid, and the desired acid anhydride is isolated.

**PHENOL-ALDEHYDE RESINS ESTERIFIED WITH FATTY ACIDS.** R. A. Mohr and E. L. Capener (General Mills, Inc.). *U.S.* 2,878,199. A higher fatty acid ester of a phenolic resin is prepared by the condensation of a phenyl or alkyl phenol, formaldehyde and a higher fatty acid in the presence of an acidic resinification catalyst.

## • Biology and Nutrition

VARIATION IN APPEARANCE, CHOLESTEROL CONCENTRATION, AND WEIGHT OF THE CHICKEN AORTA WITH AGE AND SEX. H. S. Weiss (Dept. of Poultry Sci., Rutgers Univ., New Brunswick, N. J.). *J. Gerontology* 14, 19-24 (1959). The development of spontaneous aortic arteriosclerosis was studied in 4 generations of standard-fed, uniformly treated genetically similar group of 114 White Leghorns ranging up to 5 years of age. Progressively more severe arteriosclerosis, as measured by score, cholesterol concentration and aortic weight increment, developed in both males and females, although the diet can be considered non-atherogenic, i.e., contained approximately 0.025% cholesterol and 4-5% fat.

EFFECTS OF TESTOSTERONE AND CASTRATION ON CHOLESTEREMIA AND ATHEROGENESIS IN CHICKS ON HIGH FAT, HIGH CHOLESTEROL DIETS. R. Pick, J. Stamler, S. Rodbard, and L. N. Katz (Cardiovascular Dept., Medical Research Inst., Michael Reese Hospital, Chicago, Ill.). *Circulation Res.* 7, 202-04 (1959). Testosterone in large doses in intact cholesterol-fed cockerels partially inhibited hypercholesteremia, without affecting aorta or coronary atherosclerosis. Castration, with and without testosterone administration in young male and female chicks, was without influence on hypercholesteremia and atherogenesis.

OXIDATION OF ALBUMIN-BOUND PALMITATE-1-<sup>14</sup> BY ADIPOSE AND HEPATIC TISSUES OF THE RAT. S. W. Milstein and L. H. Driscoll (Brooke Army Medical Center, Fort Sam Houston, Texas). *J. Biol. Chem.* 234, 19-21 (1959). The oxidation of albumen-bound palmitate-1-C<sup>14</sup> by rat liver and adipose tissue has been demonstrated *in vitro*. Normal adipose tissues are about one-eighth as active as equal wet weights of liver. Prolonged fasting and alloxan diabetes depleted the fat depots and markedly elevated their capacities for fatty acid oxidation. Livers from such animals show normal rates of oxidation. Insulin administration depressed oxidation of the fat complex in the liver, but it had no significant effect on its catabolism in adipose tissue.

EFFECT OF CHYLOMICRONS ON THE FIBRINOLYTIC ACTIVITY OF NORMAL HUMAN PLASMA IN VITRO. T. C. Merigan, J. W. Farquhar, J. H. Williams, and N. Sokolow (Dept. of Medicine, Univ. of California School of Medicine, San Francisco, Calif.). *Circulation Res.* 7, 205-09 (1959). The reported inhibition of fibrinolytic activity of blood plasma after ingestion of fat has been confirmed. It has shown that the chylomicrons present in such plasma are responsible for this phenomenon.

THE EFFECT OF DIETARY FAT, CALORIC INTAKE AND PROTEIN LEVEL ON CAGED LAYERS. A. H. McDaniel, J. H. Quisenberry, B. L. Reid, and J. R. Couch (Dept. of Poultry Science, Texas Agricultural Experiment Station, College Station, Texas). *Poultry Sci.* 38, 213-19 (1959). The feeding of three protein levels (15, 20, and 25%) at energy levels of 750-960 Calories per pound, was without appreciable effect on rate of egg production. Histological study of arteries showed that neither the productive energy and protein levels used nor the use of fat had any effect on the results obtained with reference to atherosclerotic conditions involving the arteries. The observed morphological changes were not found to be related to total serum cholesterol level. Significant differences in serum cholesterol were found to exist between protein levels fed and to be significantly correlated (positive) with total liver weight, percent liver fat, serum lipid phosphorus, total serum protein and total serum albumin. Dietary protein and energy levels were without effect on total serum lipid levels, and the use of fat in the diet had no effect on total lipid content of the blood serum.

A BIOCHEMICAL STUDY OF SPONTANEOUS ATHEROSCLEROSIS IN PIGEONS. H. B. Lofland and T. B. Clarkson (Dept. of Biochemistry, Wake Forest College, Winston-Salem, N. C.). *Circulation Res.* 7, 234-37 (1959). Two breeds of pigeons (White Carneau and Silver King) have been found to have a high incidence of spontaneous atheromatous lesions in the aorta, while two breeds of homing pigeons (Racing Homers and Show Racers) seemed to be resistant to atherosclerosis. The weight of the aorta, and its cholesterol content, paralleled the severity of the disease. The levels of serum cholesterol and phospholipids, and the cholesterol:phospholipid ratio, however, appear to be unrelated to the incidence and severity of atherosclerosis in pigeons. The differences observed among the four breeds of birds showed little correlation with age, sex, diet, or physical activity.

INCIDENCE OF FAMILIAL HYPERLIPEMIA. K. Hirschhorn (Dept. of Medicine, New York Univ., Post-Graduate Medical School, New York), Rochelle Hirschhorn, M. Fraccaro and J. A. Böök. *Science* 129, 716-17 (1959). Familial hyperlipemia is an in-

herited disease associated with early onset of coronary atherosclerosis. In a survey of a student population in Sweden, an estimated case incidence of 2 to 3 percent was discovered. This study also demonstrates that there is probably a heterogeneity of causes for the primary elevation of blood triglycerides in man.

EFFECT OF TAURINE, GLYCINE AND  $\beta$ -SITOSTEROLS ON SERUM AND TISSUE CHOLESTEROL IN THE RAT AND RABBIT. R. G. Herrmann (Lilly Research Lab., Indianapolis, Ind.). *Circulation Res.* 7, 224-27 (1959). Rats and rabbits were fed a hypercholesteremic diet until elevated serum cholesterol levels were observed in all animals. The animals were then divided into groups, one group remaining on the original diet and the others receiving the same hypercholesteremic diet fortified with either taurine, glycine, or  $\beta$ -sitosterols. In the rat, 4 per cent taurine in the diet significantly decreased the serum, liver, and aorta cholesterol concentration, but glycine was without significant effect. In the rabbit, 2 per cent  $\beta$ -sitosterol in the diet significantly reduced the serum, liver, and aorta cholesterol, while taurine or glycine produced no significant effect. Neither taurine, glycine, nor  $\beta$ -sitosterols produced any signs of toxicity.

FURTHER STUDIES ON NUTRITIONAL FACTORS INFLUENCING THE EXPRESSION OF VITAMIN E DEFICIENCY SYMPTOMS IN CHICKS. S. V. Amato and H. M. Scott (Illinois Agricultural Experiment Station, Urbana, Illinois). *Poultry Sci.* 38, 176-180 (1959). A casein-cerelose purified diet low in vitamin E, containing 3 per cent unsaturated fish oil, failed to induce vitamin E deficiency symptoms in either depleted or non-depleted chicks. When the cerelose was replaced by degerminated white corn meal, both depleted and non-depleted chicks developed encephalomalacia. Water washing of the white corn meal was without effect, but solvent extraction of degerminated white corn meal decreased the incidence of encephalomalacia to a low level in some unknown manner. When corn starch served as the carbohydrate source in this diet, symptoms of vitamin E deficiency were not observed.

LIPIDE METABOLISM. INTRODUCTION. A. C. Frazer (Univ. Birmingham, Engl.). *Brit. Med. Bull.* 14, 197-200 (1958).

BIOSYNTHESIS OF FATTY ACIDS. Priscilla Hele (Hammersmith Hosp., London). *Ibid.* 201-6.

FAT ABSORPTION AND ITS DISORDERS. A. C. Frazer (Univ. Birmingham, Engl.). *Ibid.* 212-20.

BIOSYNTHESIS OF CHOLESTEROL. J. W. Cornforth (Natl. Inst. Med. Research, London), and G. Popják. *Ibid.* 221-6.

THE ABSORPTION AND METABOLISM OF STEROLS. J. Glover and R. A. Morton (Univ. Liverpool, Engl.). *Ibid.* 226-33.

REMOVAL OF LIPIDE FROM THE BLOOD STREAM. J. E. French, B. Morris, and D. S. Robinson (Univ. Oxford, Engl.). *Ibid.* 234-8.

HORMONAL CONTROL OF THE CIRCULATING LIPIDES. G. S. Boyd (Univ. Edinburgh, Scot.). *Ibid.* 239-42.

THE EFFECT OF DIETARY FAT ON BLOOD LIPIDES AND THEIR RELATION TO ISCHEMIC HEART DISEASE. B. Bronte-Stewart (Radcliffe Infirmary, Oxford, Engl.). *Ibid.* 243-52.

FATS IN BLOOD COAGULATION. J. C. F. Poole (Univ. Oxford, Engl.). *Ibid.* 253-8.

ESSENTIAL FATTY ACIDS AND THE SKIN. H. M. Sinclair. *Ibid.* 258-62.

ESSENTIAL FATTY ACIDS AND THE SKIN. A. T. James and J. E. Lovelock (Natl. Inst. Med. Research, London). *Ibid.* 262-6. (*C.A.* 53, 533)

OBSERVATIONS UPON THE TOXIC PRINCIPLE IN THE SEEDS OF DICHAPETALUM TOXICARIUM. SEPARATION OF A LONG-CHAIN FATTY ACID CONTAINING FLUORINE. R. A. Peters (Dept. Biochem., Univ. Oxford), R. W. Wakelin, A. J. P. Martin, J. Webb, and F. T. Birks. *Biochem. J.* 71, 245-8 (1959). By reversed-phase chromatography the toxic principle from ratsbane was isolated and provisionally identified as a fluoro-saturated acid containing at least 16 carbon atoms. When injected into rats or fed, it induced large citric acid accumulations, especially in the heart, presumably by conversion into fluorocitric acid. When incubated with kidney particles from the guinea pig, the fluoro-fatty acid induced citric acid accumulation.

STUDY OF LYMPH LIPIDS FOLLOWING ADMINISTRATION OF OLEIC-1-C<sup>14</sup> ACID WITH OR WITHOUT CHOLESTEROL. G. E. Clement and J. F. Mead (Univ. Calif., School of Medicine, Los Angeles). *Proc. Soc. Exptl. Biol. Med.* 100, 285-8 (1959). In rats, cholesterol feeding apparently decreased the proportion of triglycerides in lymph and increased those of sterol ester and phospholipid containing oleic-1-C<sup>14</sup>. Although glyceride fatty acid distribution was similar to that of the fed acids, both

sterol ester and phospholipid fatty acids were diluted with endogenous oleic, linoleic and stearic acids.

**METHOD OF PREPARING SUSTAINED RELEASE PARTICLES.** G. M. Grass, Jr. and M. J. Robinson (Smith, Kline & French Labs). *U.S. 2,875,130*. A finely powdered pharmaceutical is mixed with a molten water-insoluble lipid having a melting point above 85°. The mixture is solidified, powdered, and dispersed in a second molten lipid having a melting point at least 5° below that of the first lipid. The mixture is dispersed in warm water so as to form an emulsion at a temperature above the melting point of the second lipid. The emulsion is finally cooled to precipitate the desired sustained release pharmaceutical powder.

**PROCESSES OF PRODUCING A BAKED CEREAL PRODUCT.** G. H. Blomqvist and A. I. Malm (P. O. Stokkebyes Kvarnaktiebolag). *U.S. 2,868,648*. Formation of bitter substances in a baked product containing a considerable proportion of oats is inhibited by the incorporation of 0.1 to 12.0% of coconut oil.

**FEED YEASTS FROM WASTE LIQUOR OF SOAP MANUFACTURE.** Ken'ichi Otsuka and Seiji Tada (Yamanashi Univ., Kōfu). *Nippon Nōgei-Kagaku Kaishi* (J. Agr. Chem. Soc. Japan) 32, 805-10 (1958). On artificial synthetic medium containing 2.5% glycerol all strains of *Hansenula* tested, some of *Pichia*, *Mycoderma*, and *Candida* grew well. On the medium from industrial soap waste liquor after neutralization, filtration, and dilution to 4 volumes (glycerol 2.02%, NaCl 2.66%, pH 6.0) *Pichia* sp. grew best (*Hansenula* sp. followed) after shaking culture at 30° for 48 hrs. Examination of the composition of the medium showed that Mg<sup>++</sup> and to a lesser degree Mn<sup>++</sup> were effective in the propagation of yeasts. Only 0.2% phosphate was required, as N source NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> was best, 0.1% as N was sufficient; 2.62% NaCl was optimal in the range of 0.62-5.62%, and in the range of 1.23-4.63% glycerol, the yield of yeasts against glycerol was higher at lower concentration of glycerol. At higher concentrations of glycerol the time for propagation was lengthened. Thus addition of more precultured suspension increased the yield. A pilot plant test with a vessel of 300 l. was successful. An iron tank was as good as a stainless steel tank. Dry yeasts suitable for food and feed in the flavor could be obtained in the yield of 50% against glycerol. A patent is pending.

## • Paints and Drying Oils

**METHOD OF ESTERIFYING EPOXY RESINS WITH UNSATURATED ACIDS.** W. C. Stoesser, E. H. Sommerfeld, and W. R. Surine (The Dow Chemical Co.). *U.S. 2,875,165*. Epoxy resins prepared by the reaction of 4,4'-dihydroxydiphenyl dimethyl methane and epichlorohydrin are esterified with drying or semi-drying oils in the presence of a minor amount of a chelating agent.

**MODIFIED OIL COPOLYMERS.** J. C. Konen and R. A. Boller (Archer-Daniels-Midland Co.). *U.S. 2,877,194*. A coating vehicle is prepared from a partial ester of a polyhydric alcohol with a nonconjugated drying or semi-drying oil or the corresponding acids by reaction in the presence of an acidic catalyst with an  $\alpha,\beta$ -ethylenic dicarboxylic acid or anhydride and then in the presence of an organic peroxide catalyst with a polymerizable vinyl benzene compound.

**VINYLATED FATTY ACID ESTERS.** J. W. McNabb (American Can Co. and Kienle & Co.). *U.S. 2,877,195*. An interpolymers is prepared by heating in the presence of a peroxide catalyst an alkyl ester of acrylic or methacrylic acid and an unsaturated C<sub>12</sub> to C<sub>22</sub> fatty acid polyester of a polyether.

**COATING COMPOSITION COMPRISING AT LEAST TWO VINYL POLYMERS AND LECITHIN.** G. J. De Cristoforo (E. I. du Pont de Nemours & Co.). *U.S. 2,877,922*. A baked coating for beverage cans is prepared from at least two vinyl polymers and 0.1 to 0.75% by wt. of lecithin.

## • Detergents

**AN ECONOMIST'S VIEW OF THE BRITISH SOAPMAKING INDUSTRY.** R. Brech (Unilever Ltd.). *Soap, Perfumery, Cosmetics* 32, 159-64 (1959). Vital statistics concerning the British soap and detergent industry are reviewed. Tables include consumption of detergents from 1781-1958, relation between real income and consumption of detergents from 1870 to 1957, comparison of total retail prices and prices of detergents from 1900 to 1958, and the personal consumption of detergents from 1780 to 1958. It is concluded that there is an ever-expanding market for detergents in the United Kingdom.

**OXO ETHER ALCOHOLS—PROMISING DETERGENT COMPOUNDS.** J. H. Bartlett, I. Kirshenbaum, and C. W. Muessig (Esso Research & Eng. Co., Linden, N. J.). *Ind. Eng. Chem.* 51, 257-8

(1959). To obtain a better understanding of the oxo reaction, by-products formed in oxo bottoms have been studied. These compounds boil higher than the principal alcohol and/or aldehyde products. The principal compounds in oxo bottoms are ether alcohols, presumably formed via acetal and subsequent oxonation. Nonionic surfactants prepared from the ether alcohols show advantages over nonylphenol nonionics in laboratory surfactant tests.

**THE PRODUCTION OF DETERGENTS FROM OLEFINS.** E. M. Kheifetz, N. V. Milovidova, E. B. Zelvyanskaya, B. I. Il'in, R. N. Yudakova, and I. B. Rapoport. *Khim. i Tekhnol. Topliv i Masel* 3(9), 48-54 (1958). Results of laboratory experiments on the sulfonation of olefins boiling in the range 180-320° synthesized from carbon monoxide and hydrogen with Cu-Ni catalysts, from the thermal cracking of paraffins or the dehydrogenation of paraffin hydrocarbons, are tabulated. Optimum sulfonation conditions were: acid 98%; ratio of C<sub>m</sub>H<sub>m</sub> to H<sub>2</sub>SO<sub>4</sub> 1:5 to 1:2, temperature less than 10°. The degree of conversion ranged from 71 to 81%, with 5% appearing as polymers. Percentage conversion falls with rise in molecular weight of the olefin. The products are satisfactory surface-active agents and detergents. A scale drawing of an elaborate glass apparatus is shown. (C.A. 53, 3740)

**ANIONIC SURFACTANTS IN LIGHT DUTY DETERGENTS.** W. M. Bright and L. P. McKenney (Lever Brothers Co., New York). *Soap Chem. Specialties* 35(1), 43-6, 107 (1959). The history of anionic detergents in light duty products is reviewed. The early products were soap flakes and beads, while newer products are now liquids. The various types of anionic active ingredients, additives and solvents used in these formulations are described, and their future growth outlined.

**ANIONIC SURFACE AGENTS IN HEAVY-DUTY INDUSTRIAL CLEANERS.** J. B. Davidson (Cowles Chemical Co., Skaneateles Falls, N. Y.). *Soap Chem. Specialties* 35(1), 47-8 (1959). The use of anionic surfactants in industrial cleaners used for maintenance and manufacturing processes is reviewed. The two main types are diphasic cleaners and alkali-type. The various classifications of anionics used in these cleaners are listed and comparative surface-active properties of the anionics versus nonionics are described.

**DETERGENT ADSORPTION ON SOIL AND SUBSTRATE.** J. C. Harris (Monsanto Chem. Co., Central Research Laboratories, Dayton, O.). *Soap Chem. Specialties* 34(11), 40-3, 101; (12) 59-62, 233 (1958); 35(1), 49-52, 108-10; (2) 57-8, 103 (1959). This is an extensive review in which an attempt is made to correlate surfactant adsorption in the detergency process with other measurable detergent functions such as critical micelle concentration, zeta potential, and detergency. 115 references.

**OPTICAL BRIGHTENING AGENTS IN THE SOAP AND DETERGENT INDUSTRY.** G. Durig and R. Kaufmann. *Fette, Seifen, Anstrichmittel* 60, 838-43 (1958). A summary is given of the performance of "Uvitex," a brightening agent for soaps and detergents. (C.A. 53, 3735)

**SUSPENDING ACTION BY DETERGENTS IN THE PRESENCE OF SUBSTRATE.** J. C. Harris (Monsanto Chem. Co., Dayton, Ohio). *Textile Research J.* 29, 99-118 (1959). Soil suspension tests in the presence of substrate are superior to those made in its absence, but at the expense of increased complexity of the system. Among factors mentioned as affecting the soil-substrate relationship are mechanical, chemical, and electrical forces affecting both soil and substrate; adsorption of surfactant (or antiredeposition agent) on substrate and/or soil; sedimentation; deflocculation; soil particle size; solvation; and several of lesser potential. Surfactants are enhanced in soil deposition prevention by builders such as polyphosphate and silicates. Outstanding in preventing carbon soil deposition are nonionic surfactants. Interpretation of mechanisms proposed are for the action of sodium carboxymethylcellulose in preventing deposition indicates probable action by adsorption on the cellulose substrate. Use of radiotracer tagged soils, only recently begun in this field, promises aid in elucidating the soil suspension mechanism.

**ANALYSIS OF PETROLEUM SULFONATES.** W. F. Maass, E. Buchspiess-Pauleutz, and F. Stinsky. *Seifen-Ole-Fette-Wachse* 54, 575-8 (1958). A review of German and U.S. standard analytical methods. (C.A. 53, 133)

**HYDROLYSIS OF ALKYL SULFATES.** R. R. Read and W. G. Fredell (Warner-Lambert Research Inst., Morris Plains, N. J.). *Drug & Cosmetic Ind.* 84, 178, 256-7 (1959). Sodium lauryl sulfate is inherently stable, being neutral in solution. Higher temperatures accelerate decomposition. Solutions having pH values higher than 3 do not exhibit rapid change and, when the pH is above 5, do not show hydrolysis even at highest "shelf" temperatures. At very high temperatures and hydrogen ion concentrations, breakdown can be very rapid. Since hydrolysis

is slow at pH levels above 4, buffering at these levels should result in prolonged stability.

RESEARCH ON WASHING PROBLEMS. G. Weder. *Textile Rundschau* 13, 637-42 (1958). Comparisons of 6 commercial detergents and of 3 laundry machines are given, and ability of detergents to remove fat from glass and metal are determined by a simple gravimetric method. Lauryl sulfate performed best. Among inorganic chemicals, all phosphates were superior to silicates, the meta-phosphate being the best. (*C.A.* 53, 3740)

CRITICAL MICELLE CONCENTRATION OF NONIONIC SURFACTANTS. III. THEORY OF MICELLE FORMATION. Toshio Nakagawa and Kaoru Kuriyama (Shionogi & Co., Amagasaki, Hyōgo-ken). *Nippon Kagaku Zasshi* (J. Chem. Soc. Japan, Pure Chem. Sect.) 78, 1568-73 (1957). As the length of hydrophilic part increases, the critical micelle concentration (c.m.c.) increases and the number of molecules consisting the micelle decreases. This increase in c.m.c. with the increase in the length of hydrophilic part is explained to be due to the increase in the free energy of the hydrophilic part and the decrease in the lowering degree of interface free energy between hydrocarbon and water.

IV. EFFECT OF OXYETHYLENE CHAIN LENGTH ON CRITICAL MICELLE CONCENTRATION. Toshio Nakagawa, Kaoru Kuriyama, and Kazuo Tori. *Ibid.* 1573-7. The theoretical conclusion of Part III was proved by experiments. The c.m.c. of methoxy-polyethyleneglycol capric acid esters was 8.0, 10.5, 14.0, and  $16.0 \times 10^{-4}$  mole/l., respectively, when the number of oxyethylene groups was 7.0, 10.3, 11.9, and 16.0. The similar result was obtained also for lauric acid esters.

MIXED MICELLES OF IONIC AND NONIONIC SURFACTANTS. Toshio Nakagawa and Hideo Inoue (Shionogi & Co., Amagasaki, Hyōgo-ken). *Nippon Kagaku Zasshi* (J. Chem. Soc. Japan, Pure Chem. Sect.) 78, 636-40 (1957). Mixed solutions of Na laurylsulfate and Brij 35 (Atlas Co., U.S.A.) (polyoxyethylene lauryl ether) were measured for electrophoresis, diffusion, and viscosity. It was assumed that such mixed solutions contained only one kind of mixed micelles of low degree of dispersion. At lower concentration of the ionic surfactant in the mixture, the charge and  $\zeta$ -potential of micelles, and association number of the ionic surfactant consisting the micelles decreased, while the effective radius and amount of micelles, and association number of the nonionic surfactant consisting the micelle increased.

PENETRATION OF DETERGENT SOLUTIONS INTO FILTER PAPER. Kyozo Nakasima and Kazumune Nakao (Osaka-furitsu Kōgyō Shōreikan, Osaka). *Nippon Kagaku Zasshi* (J. Chem. Soc. Japan, Pure Chem. Sect.) 78, 594-7 (1957). Studies were made with solutions of Aerosol OT and of Na dodecylsulfate. The penetration obeyed the equation of Kozeny-Carman [*Discuss. Faraday Soc.* 8, 72 (1948)].

THE FACTORS AFFECTING DETERGENCY. II. MIXTURES OF SATURATED FATTY ACID AND OLEIC ACID SOAPS. Seizuro Hayashi, Toyozo Fuchizawa, and Yoshiro Namba (Nihon Yushi Co., Amagasaki, Hyōgo-ken). *Yukagaku* (J. Japan Oil Chemists' Soc.) 6, 208-13 (1957). Detergency was compared with Launder-O-Meter (Atlas Co., U.S.A.) for Na oleate, and mixtures of Na oleate and one of Na soaps of  $C_{10}$ - $C_{18}$  saturated fatty acids (0.25% solns.) at 35°. The detergency of Na oleate was similar with that of Na myristate. Addition of Na oleate decreased the effect of C length of the soap on the detergency, all the mixtures giving similar detergency. Thus the detergency of the mixture of Na oleate with Na caprate or laurate was much enhanced. Detergency was best correlated with dispersibility and solubilization ability.

III. SODIUM SALTS OF SATURATED FATTY ALCOHOL SULFATES. Seizuro Hayashi, Takashi Yamamoto, Toyozo Fuchizawa, and Yoshiro Namba. *Ibid.* 213-17. Saturated alkyl sulfates of  $C_{10}$ - $C_{18}$  were studied. Detergency was better correlated with dispersibility, emulsifying power, solubilization power, and prevention of redeposition than with surface and interface activity, foaming power, and penetrating power. The former properties increased with the increase in chain length of alkyls, while the latter properties showed maximum at  $C_{14}$ .

BEHAVIOR OF DYE IONS IN DETERGENT SOLUTIONS. III. INVESTIGATION OF THE INTERACTION OF DYES WITH DETERGENTS BY THE AID OF DYE-DETERGENT COMPLEX. Tamotsu Kondo (Mushashi Univ., Tokyo). *Nippon Kagaku Zasshi* (J. Chem. Soc. Japan, Pure Chem. Sect.) 78, 1093-6 (1957). Studies were made with Na dodecylsulfate and dodecylpyridonium bromide as detergents and eosine G and rhodamine 6G as dyes. At higher dye concentrations the apparent critical micelle concentration of the detergent measured from electric conductivity curve decreased, but that measured from the change in fluorescence intensity increased. This apparent discrepancy could be explained.

METHOD FOR PURIFYING SYNTHETIC ORGANIC DETERGENTS. P. Becher (Colgate-Palmolive Co.). *U.S.* 2,863,887. Impurities such as inorganic metal salts of sulfated partial glyceryl esters of long-chain fatty acids having from 8 to 24 carbon atoms by treating the alcohol extract of these compounds with magnesium sulfate. This results in the formation of two layers, the upper layer containing the detergent, and the lower layer the inorganic impurities.

GERMICIDALLY ACTIVE SOAP CONTAINING SILVER SALT OF AROMATIC SULFINIC ACID. G. A. Lutz and R. E. Sharpe (Perma-chem Corp.). *U.S.* 2,864,769. It has been found that soaps containing a silver salt of an aromatic sulfonic acid kill skin bacteria rapidly under ordinary washing conditions. The silver salt is incorporated in small amounts in a conventional soap. The silver salts of either aromatic monosulfonic or polysulfonic acid may be used.

LIQUID DETERGENT COMPOSITIONS. H. W. McCume and O. T. Quimby (Procter & Gamble Co.). *U.S.* 2,864,770. A concentrated, thixotropic liquid detergent is prepared consisting of fully water-soluble organic vehicle such as a glycol or glycerine, a colloidal dispersion of a calcium sequestering phosphate having a particle size not greater than about 0.5 micron, and a fully soluble non-soap detergent having a viscosity in the range from about 0.5 to 2.0 poises.

PROCESS OF MAKING NONSOAP DETERGENT BARS AND PRODUCT. L. F. Henderson and F. E. Carroll (Lever Brothers Co.). *U.S.* 2,868,731. An improved nonsoap detergent bar contains a small amount of a free fatty acid of high molecular weight acting as a plasticizer for the detergent, and water-soluble fatty acid soap which serves to improve the soaplike feel and texture of the bar as well as its lathering properties.

MANUFACTURE OF FATTY ACID MONOESTERS OF GLYCEROL MONOSULFURIC ACID AND SALTS THEREOF. F. W. Gray (Colgate-Palmolive Co.). *U.S.* 2,868,812. A detergent composition intermediate, which on neutralization in an aqueous medium with caustic will yield a detergent salt, is prepared by reacting one molecular proportion of a saturated fatty triglyceride, two molecular proportions of glycerol trisulfuric acid and a small amount of sulfuric acid monohydrate.

DETERGENT COMPOSITION. W. F. Tomlinson (Stepan Chem. Co.). *U.S.* 2,870,091. An improved liquid detergent having superior deterative, emulsifying and foam stabilizing properties, capable of retaining large amounts of inorganic detergents in suspension, consists of the inorganic detergents, a higher fatty acid amide detergent and foam stabilizer such as cocount fatty acids (diethanolamide, and a lower fatty acid amide such as palar-gonic diethanolamide which permits large amounts of the above components to remain in aqueous solution.

POLYPHOSPHATE COMPOSITIONS CONTAINING TALLOW METHYL TAURIDE AND 2-MERCAPTOTHIAZOLINE. E. E. Ruff (Lever Brothers Co.). *U.S.* 2,870,092. It was found that spray-dried triphosphosphate detergent compositions containing at least one organic nonsoap detergent and from about 10 to 20% of an alkali metal carbonate could be provided with improved tarnishing properties upon German silver when such compositions contain 2-mercaptothiazoline and an alkali metal tallow methyl tauride as synergistic tarnish inhibitors. *U.S.* 2,870,093. This patent covers the use of 2-mercaptothiazoline and an alkali metal coco- $\beta$ -alanine as the synergistic tarnish inhibitors.

PRODUCTION OF NONIONIC SURFACE ACTIVE AGENTS. C. A. Carter (Union Carbide Corp.). *U.S.* 2,870,220. Primary and secondary alkanols having 10 to 17 carbon atoms can be readily converted with good yield into corresponding monoalkyl ethers of ethylene glycol and polyethylene glycols which are more efficient surface active agents, by a novel two-stage process in which the alkanol and ethylene oxide are first reacted in the presence of an acidic catalyst, followed by reaction with further ethylene oxide in the presence of an alkali metal alcohol of the initial alkanol.

SHAMPOO COMPOSITIONS. H. Henkin (Colgate-Palmolive Co.). *U.S.* 2,871,193. A liquid detergent shampoo of desirable viscosity and good foaming and cleansing properties consists of a water-soluble higher fatty acid monoglyceride monosulfate and a minor proportion of water-soluble hydroxypropyl methylcellulose in an aqueous medium.

ANTI-SOIL REDEPOSITION AGENTS. E. A. Vitalis (American Cyanamid Co.). *U.S.* 2,874,124. A detergent composition capable of preventing redeposition of soil in the cleaning of fabrics comprises from about 5 to 95 parts by weight of a water-soluble organic detergent such as a fatty acid soap or synthetic detergent and, as a soil redeposition inhibitor, from about 0.5 to 20 parts by weight of a polyvinylloxazolidone having a molecular weight of at least 200.