ever for this series the agitator speed was increased to 1,140 rpm. This gave violent turbulence at the surface of the charge.

Samples 12-15 again illustrate the lack of correlation between flavor stability and oxidative stability. As before, the oils were graded after three days' aging at 60°C. Sample 12 was obtained by high pressure hydrogenation at a very low catalyst level. The final oil, before filtration, contained less than 6 parts per million of nickel.[1]

Runs 16-20 demonstrate the effect of extreme changes in pressure on the rate and selectivity of hydrogenation. Sample 20 was obtained by bubbling hydrogen through the oil while agitating it under 20 inches of vacuum. Note that this oil is evidently higher melting than sample 19 in spite of the fact that it was hydrogenated more selectively than 19. This is probably the result of its high content of trans acids (6).

Effect of Catalyst Concentration

Samples 14 and 15 of Table III show the direct relationship which holds between catalyst concentration and selectivity at normal pressures. That is, the content of polyunsaturated glycerides at a given refractive index or iodine value varies inversely with the amount of catalyst used in the hydrogenation. Samples 23 and 24 of Table IV illustrate how this effect is reversed at high pressure.

Effect of Temperature

The change in selectivity with temperature at moderate pressure is shown by comparing samples 21 and 22 of Table IV. At 300°C. (sample 25) considerable polymerization takes place, which accounts for the small drop in refractive index. Selectivity is low at this extreme temperature. Samples 10 and 11 of Table II seem to indicate that selectivity is almost independent of temperature at 400 psi.

Effect of Agitation

Samples 26 and 27 of Table IV illustrate the effect of changes in agitator speed at low pressure on chemical selectivity. At high pressure, samples 28 and 29, the effect is reversed.

Summary

No correlation was found between the flavor stability of partially hydrogenated soybean oil and its oxidative stability or its content of polyunsaturated glycerides. Fats having unusually high melting and softening points with considerable spread between melting and softening points were obtained by high pressure hydrogenation. At moderate pressures chemical selectivity was found to vary directly with catalyst concentration and inversely with agitator speed, as reported by previous workers. However at high pressures the effect of these variables was reversed. That is, selectivity then varied directly with the amount of agitation and inversely with the catalyst concentration.

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[1] Spectrophotometric method, involving the development of a colored complex with dimethylglyoxime.

ABSTRACTS . . . . Don Whyte, Editor

Oils and Fats

R. A. Reiners, Abstractor

Reagents for idometric determination of peroxides in fats. L. Hartman and Margaret D. L. White (Dept. Sci. Ind. Res., Wellington, N. Z.). *Anal. Chem.* **24**, 527-29(1952). It is suggested that a 10% solution of citric acid in a mixture of tert. butyl alcohol and carbon tetrachloride be used as solvent in the Lea method for peroxide determination in fats in order to reduce the blank.

Estimation of 2- and 3- tert-butyl-4-hydroxyanisole isomers. J. H. Mahon and R. A. Chapman (Dept. Nat. Health Welfare, Ottawa). *Anal. Chem.* **24**, 534-536(1952). The method is based on the fact that the 3-tert. butyl-isomer reacts to form a more highly colored complex with 2,6-dichloroquinone chloroimide-borax reagent than the 2-tert. butyl-isomer and that the reverse is true on reaction with ferric chloride-1,1-bipyridine reagent. The 95% confidence limits for the determination were ± 3.0% units.

The component fatty acids and glycerides of jute-seed oils. M. L. Meara and N. K. Sen (Univ., Liverpool). *J. Sci. Food Agr.* **3**, 237-240(1952). The component acids from the seed fats of *Corchorus capsularis* and *C. olitorius*, two varieties of jute, have been shown to consist of palmitic, 12.2, 16.9; stearic 4.6, 3.7; arachidic 2.2, ...; behenic ...., 1.8; lignoceric 0.9, 1.1; cerotic 1.2, ...; oleic 28.7, 9.1; linoleic 41.3, 62.5; linolenic 4.7, 0.9; and C_{20} monoethenoid 4.1, 4.0% (wt.) respectively. The component glycerides of the *C. olitorius* seed oil, which resembles sunflower seed oil, consist of 11% disaturated-monounsaturated glycerides, 64% monosaturated-diunsaturated glycerides mainly saturated-dilinoleins, and 25% triunsaturated glycerides consisting mainly of oleodilinoleins and trilinolein.

Semimicromethod for the determination of plant sterols. D. Waghorne and C. E. Ball (Ontario Agr. College, Guelph, Ontario). *Anal. Chem.* **24**, 560-564(1952). The method uses dichromate oxidation of the digitonides, followed by titration of the excess dichromate with ferrous solution, and yields results with a precision better than ± 3%. Samples containing as little as 0.1 mg. total sterol are adequate for a single determination. The sensitivity of the method approaches that of the colorimetric methods and is unaffected by slight changes in the structure of the sterol molecule.

The constitution of some wool wax esters. J. Tiedt and E. V. Truter (Univ. Leeds). *Chem. Ind.* **1952**, 403. By fractional crystallization from methyl ethyl ketone there has been isolated from wool grease cholesteryl 24-methylhexacosanoate, cholesteryl 26-methyloctacosanoate, cholesteryl 28-methyltriacontanoate and an as yet unidentified diester of a hydroxy acid.

Lipids of the central nervous system. G. H. S. Stanley (Maudsley Hosp., London). *Biochem. J.* **50**, xxiv(1952). Tracer experiments using deuterium showed that there is a significantly rapid turnover of the lipids in the central nervous system.

The composition of the depot fats of a pig fed on a diet rich in whale oil. G. A. Gorton, T. P. Hilditch and M. L. Meara (Univ., Liverpool). *Biochem. J.* **50**, 517-23(1952). The depot fats from a pig whose diet had included 50% whale oil consisted of a mixture of whale oil with the typical fat synthesized by the pig from non-fatty food. Crystallization of the perinephric fat from acetone at —40° gave 26% of a soluble fraction which approximated the composition of whale oil and 74% of insoluble material which closely resembled normal pig fat.

The branched-chain fatty acids of ox fat. 1. The isolation from ox suet of a C_{17} branched chain saturated fatty acid. R. P. Hansen, F. B. Shorland and N. J. Cooke (Dept. Sci. Ind. Res., Wellington, N. Z.). *Biochem. J.* **50**, 581(1952). A C_{17} methyl branched-chain saturated fatty acid isomeric with n-heptadecanoic acid has been shown to be present in the fat extracted from ox suet.

Determination of high proportions of unsaponifiable matter. K. A. Williams. *Chem. Ind.* **1952**, 379. The British Standard method for the determination of unsaponifiable matter was found to give excellent results on a mixture containing 50% castor oil, 25% mineral oil and 25% oleyl alcohol.

Cholesteryl laurate. D. Kritchevsky and Margaret E. Anderson (Univ. Calif., Berkeley). *J. Am. Chem. Soc.* **74**, 1857-8 (1952). Cholesterol and lauroyl chloride were reacted in pyridine to form an ester melting at 78-78.5°.

Regeneration of used vegetable oil. P. I. Kulikov. *Rybnoe Khoz.* **27**, No. 5, 45-6(1951). Vegetable oil used for cooking shows a gradual rise of acid no. with use. In order to regenerate the oil for further use, the oil is washed with water, warmed to 70-5°, and neutralized with stirring by means of 20% NaOH (total 3% by weight) and 10% Na_2CO_3 (5% by weight). The oil is allowed to settle and after separation is ready for further use. The resulting soap floats on top and is readily separated. The oil after recovery should be washed with hot water and its acidity tested before being sent back to the frying plant. The final product should have acid no. under 1.1. (*Chem. Abs.* **46**, 4694)

Polymerizable derivatives of long-chain fatty acids. VI. Preparation and applicability of urea complexes of vinyl esters. D. Swern and W. S. Port (Eastern Reg. Res. Lab., Philadelphia). *J. Am. Chem. Soc.* **74**, 1738-9(1952). Vinyl esters of long-chain fatty acids, such as vinyl pelargonate, laurate, palmitate and stearate, form urea complexes in good to excellent yields (56-99%). Advantage has been taken of this technique to separate vinyl pelargonate from mixtures containing monomer, polymer, inhibitor and other unknown compounds.

Chemistry of epoxy compounds. XIII. Urea complex formation in determining the configuration of the 9,10 dihydroxystearic acids. D. Swern, L. P. Witnauer and H. B. Knight (Eastern Reg. Res. Lab., Philadelphia). *J. Am. Chem. Soc.* **74**, 1655-57 (1952). Based on the ability of low-melting 9,10-dihydroxystearic acid (m.p. 95°) to form a crystalline complex readily with urea in quantitative yield and the reluctance of high-melting 9,10-dihydroxystearic acid (m.p. 131°) to form a complex, it has been shown that the hydroxyl groups in the high melting isomer are on opposite sides of the chain whereas in the low melting isomer they are on the same side. Thus hydroxylation with $KMnO_4$ proceeds by *cis* or normal addition. Opening of the oxirane ring of the isomeric 9,10-epoxystearic acids involves inversion.

2-octadecenoic acid. II. Preparation of the 2,3-epoxystearic acids and the configurational relationships between 2,3-epoxy-, bromoacetoxy-, and dihydroxystearic acids. G. S. Myers (Queen's Univ., Kingston). *J. Am. Chem. Soc.* **74**, 1390-93 (1952). The reaction scheme described by Swern, to correlate the configurations of the compounds formed in the conversion of oleic and elaidic acids to the 9,10-dihydroxystearic acids by way of the epoxy and chlorohydroxy compounds has been used to correlate the stereochemical relationships involved in the conversion of *cis-* and *trans-*2-octadecenoic to the 2,3-dihydroxystearic acids, both by direct hydroxylation and by way of the bromoacetoxy and oxirane compounds. Positions have been assigned to the bromine and acetoxy groups in three 2,3 (3,2) bromo-acetoxystearic acids.

Investigations on the mechanism of catalytic hydrogenations. XVI. Studies on unsaturated aliphatic acids. W. P. Dunworth and F. F. Nord (Fordham Univ.). *J. Am. Chem. Soc.* **74**, 1457-59(1952). A kinetic study of the rate of hydrogenation of various unsaturated fatty acids has shown that the degree of substitution of the double bond, the molecular weight of the compound and the position of the double bond are important in determining the velocity of reduction, when colloidal Pd and Rh are employed as catalysts in 70% ethanol solution. It has been established that α-β-unsaturated acids hydrogenate most slowly with Pd while β-γ-unsaturated acids hydrogenate most slowly with Rh.

Paper chromatography of steroid compounds. G. M. Shull, J. L. Sardinas and R. C. Nubel (Chas. Pfizer & Co.). *Arch. Biochem. Biophys.* **37**, 186-198(1952). Separation of most steroids was achieved on alumina-impregnated paper with non-aqueous solvents as developing agents but some of the slower-moving adrenal cortical steroids were best separated on plain paper with benzene-water as the solvent. The most generally useful method for the detection of steroids on developed chromatograms involved examination under ultraviolet light after chlorination and spraying with antimony chloride-acetic anhydride reagent. Quantities ranging from 0.5 to 5.0 µg. of most of the steroids could be detected.

Rendering fat according to the method of Kuznetsova. L. Gutman (Moscow Meat Combine). *Myasnaya Ind. S. S. S. R.* **23**, No. 1, 23(1952). Rendering under vacuum is rapid and produces a better-quality lard. (*Chem. Abs.* **46**, 4694)

Behavior of certain edible oils in the frying pan. P. Sen, C. V. N. Rao and T. N. Mehta (Nagpur Univ.). *Indian Soap J.* **17**, 107-12(1951). Data are given on the changes in physical and chemical characteristics of raw peanut oil, raw and refined coconut oil, and hydrogenated fat, when used for frying at 190-200°. (*Chem. Abs.* **46**, 4694)

Pisa fat, a coconut oil substitute. J. G. Kane (Univ. Bombay). *Indian Soap J.* **17**, 72-4(1951). The fat is recommended for soapmaking and for the preparation of pure lauric acid and wetting agents. (*Chem. Abs.* **46**, 4815)

Component acids of pig and sheep bones. J. Holmberg and Ulla Rosenqvist-Bergqvist (Forskningslab. LKB, Appelviken, Sweden). *Svensk Kem. Tid.* **63**, 272-5(1951). Analysis of pig- and sheep-bone fats has confirmed previous findings that bone fats form a distinct class of fats. Both pig, and sheep-bone fats have less palmitic acid than the corresponding body fats, and the ratio of palmitic to stearic acid is high. Short-chain acids (lauric and lower) are present in both instances. Pig-bone fat is high in linoleic acid. (*Chem. Abs.* **46**, 4814)

Hydrogenation of fatty oils. VII. Saponified sardine oil in an aqueous solution. T. Tsuchiya and S. Kinomura (Govt. Chem. Ind. Research Inst., Tokyo). *Repts. Govt. Chem. Ind. Research Inst.* **46**, 249-52(1951). The fatty acids saponified from sardine oil in aqueous NaOH were hydrogenated with Raney Ni at 28-30° and 1 atmosphere for 4 hrs. until the iodine no. decreased to 63.9 (from 202.8). During the hydrogenation the fatty acids capable of forming ethyl ether-insoluble bromides vanished when the iodine no. reached 92.4. Only then did solid fatty acids begin to appear. (*Chem. Abs.* **46**, 4815)

Esterification (of fatty acids). E. Stalmann (Hamburg, Ger.). *Seifen-Ole-Fette-Wachse* **77**, 423-5(1951). This review on the esterification of distilled fatty acids with glycerol includes the description of an apparatus which can be used both for esterification and for deodorization of the esters. (*Chem. Abs.* **46**, 4815)

Investigation of the fatty oil from the seeds of the Spirea ulmaria. I. A. M. Nordstrom and C. G. Nordstrom (Svenska Handelshogskolans Lab., Helsingfors, Finland). *Finska Kemistsamfundets Medd.* **59**, 11-24(1950). Hexane extraction of the seeds of *Spirea ulmaria* yielded a dark-green liquid (14% oil and 3% wax, dry basis). The oil had n^{16} 1.4848, d^{20} 0.9331, saponification no. 191, acid no. 4, iodine no. 182, OH no. 10, hexabromide no. 39, Polenske no. 0.27, and Reichert-Meissl no. 0.42. The per cent of acids were: saturated acids (not identified) 8, oleic 28.2, linoleic 16.2, linolenic 46.8. The oil had 5% unsaponifiable matter. Use of the oil for soap and margarine and as a drying oil and the possibilities for growing the seeds for such purposes are discussed. (*Chem. Abs.* **46**, 4815)

A simultaneous esterification and hydrogenation of fatty oils. C. F. Holmboe. *Avhandl. Norske Videnskaps-Akad. Oslo I. Mat.-Naturv. Klasse* **1951**, No. 5, 5-15. To improve the quality of low grade palm oil the excess free fatty acids were esterified with glycerin, but the product had an undesirable color. Simultaneous esterification and hydrogenation with Zn-Ni catalyst at up to 218° removed the free fatty acids and also gave an oil of lighter color. The combination process worked better when 30% of a highly unsaturated fish oil was added to the palm oil because hydrogenation after initiation was rapid and generated enough heat for the reactions. (*Chem. Abs.* **46**, 4815)

Rapid method for the determination of moisture in oil seeds. M. Fauve and P. Lacoste. *Bull. mens. ITERG* **6**, 70-2(1952). With the infrared drying method requiring 15 min. 1500 determinations were made on 5-g. samples. At moistures of 8-13% the amount of moisture was less, in most cases, than that found by the oven-drying method. (*Chem. Abs.* **46**, 4816)

Fatty oils from the seeds of Caesalpinia separia. V. V. Mhaskar, B. V. Bhide and N. L. Phalnikar (S. P. Coll., Poona). *J. Univ. Bombay*, Sect. A, **19**, Pt. 5 (Science No. 29), 16-20 (1951). *Caesalpinia separia* seeds contained 8.87% of a yellow fatty oil free from N and S. Saponification of the oil and separation of the solid and liquid acids yielded mixtures of fatty acids which contained palmitic 5.59, stearic 7.25, linoleic 63.11, oleic 23.51, and probably lignoceric acid 0.54%. A sterol m.

133-4° was obtained from the unsaponifiable matter and a bitter principle m. 181-3° was also isolated by alcohol extraction of the seeds. (*Chem. Abs.* 46, 4816)

Neutralization of vegetable oils—mathematical study of economics. G. P. Pinto (Inst. agron. do Norte, Belem, Para, Brazil). *Bol. tec. inst. agron. norte* (Belem, Brazil) 21, 3-29 (1950). Equations are derived for economic control of neutralizing animal and vegetable oils. A graph relates operating costs to the acidity (per cent oleic acid) of the oil to be treated. Application to babacu oil is illustrated. (*Chem. Abs.* 46, 4816)

SINITIRO KAWAMURA

Rice Oil. VII. Separation of fatty acids of rice oil by urea complexes. I. Formation of complexes by warming with methanol as the solvent. Hiroshi Sakurai (Osaka Univ., Sakai). *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 16-18(1952). Rice oil fatty acids were reacted with urea in methanol. As the amount of urea decreased, the amount of the complexes decreased and the selectivity of addition (for palmitic and oleic acids) increased. As the amount of MeOH decreased, the amount of the complexes increased and the selectivity decreased. The acid composition based on these analyses is palmitic 20.3, oleic 44.2, and linoleic 35.5%.

Rice Oil. VIII. Separation of fatty acids of rice oil by urea complexes. 2. Formation of complexes by stirring at room temperature. Hiroshi Sakurai. *Ibid.* 80-2. The reaction of urea with fatty acids or their Me esters of rice oil both dissolved in MeOH by stirring at room temperature showed no sharp separation of fatty acids; the iodine no. of fatty acids in the complex and nonreacted portion was 59-62 and 106-129, respectively. When Me esters were used, the separation was better as there were 2 layers. Reactions in 2 layers (urea in water and fatty acids in benzene or trichloroethylene) were not convenient when urea soln. was concentrated. The reaction of fatty acids without the solvent with aqueous urea soln. was good in separating the complex with iodine no. 31-39 from the nonreacted portion with iodine no. 115-122.

Odoriferous substances of whale oil. I. Ryûzaburô Nozu, Keigo Onoe, and Tarô Hori (Kyoto Univ.). *J. Chem. Soc.*, Pure Chem. Sect. 73, 193-6(1952). The sample was the oil of *Balaenoptera musculus* (acid no. 2, sapon. no. 189, iodine no. 108). Vacuum distillation could collect the odoriferous substances. They contained no N or S. They seemed to consist of free fatty acids, carbonyl compounds, and/or peroxides.

II. Free fatty acids. Ryôzô Gotô, Keigo Onoe, and Tarô Hori. *Ibid.* 196-7. The volatile odoriferous free fatty acids were isovaleric acid which was isolated, and formic, acetic, propionic, and butyric acids which were identified by paper chromatography. In addition unsaturated fatty acids with 1, 3, 4, and 5 double bonds were present as free acids.

The rate of oxidation of 9-, 12-, and 15-octadecenoic acids by oxygen and by perbenzoic acid. Yoshiyuki Toyama and Tsutomu Yamamoto (Nagoya Univ.). *J. Chem. Soc. Japan*, Ind. Chem Sect. 55, 164-5(1952). Comparison of oxygen absorption by oleic, elaidic, 12-octadecenoic (chiefly cis-form), and 15-octadecenoic (chiefly cis-form) acids dissolved in acetic acid with added Co resinate in the stream of oxygen at 30° revealed only slight difference in the rate of oxidation. In the case of oxidation of these octadecenoic acids dissolved in chloroform by perbenzoic acid, oleic and elaidic acids gave apparently greater rate of oxidation, than 12- and 15-octadecenoic acids. After 15, 30, 60, and 360 min. the oxygen absorption (calculated as iodine no.) was, respectively, oleic, 55.0, 71.2, 82.0, 89.8; 12-octadecenoic, 46.0, 58.3, 71.6, 84.2; 15-octadecenoic, 45.5, 56.5, 69.8, 84.0. The rate for elaidic acid was almost the same as for oleic acid.

The rate of oxidation of oleic, linoleic, and linolenic acids by oxygen and by perbenzoic acid. Yoshiyuki Toyama and Tsutomu Yamamoto. *Ibid.* 176-7. Similar experiments of oxygen absorption showed the greatest rate of oxidation for linolenic acid, and the smallest rate for oleic acid, when compared for each ethylenic bond. The oxygen absorption by perbenzoic acid oxidation was in the order, linolenic > linoleic > oleic acid; however, the rate of oxidation per one ethylenic bond was in the order, oleic > linolenic > linoleic acid.

Thiourea derivatives of fatty acid. Tokushige Suzuki and Ryôhei Oda (Kyoto Univ.). *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 178(1952). Methyl 9,10-dibromostearate in *n*-BuOH with thiourea at 100° after 10 hrs. gave a dithiuronium salt. The α-bromostearic acid in *n*-BuOH with thiourea gave after 6 hours a pseudothiohydantoin.

Chrysalis oil. III. Brown factice from chrysalis oil as the chief raw material. Senjirô Maruta and Fumio Iwama (Yamanashi Univ., Kôfu). *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 127-8(1952). Brown factice was prepared from chrysalis oil and chem. and phys. tests were performed. The product from which acetone can extract 30-40%, and which has free S less than 4%, and ash less than 0.3% was suitable as rubber additive. The addition of 0.5% vulcanization-accelerating agent of the thiazole or guanidine types is useful in preparing factice.

Methanol extraction of rice oil. I. Color of the methanol-soluble fraction. Jun'ichi Kawai, Juichi Hayakawa, and Harue Ukitani. *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 125-7 (1952). Methanol added to rice oil (acid no. 92) and stirred at 22-25°; after standing the upper MeOH-sol. layer contained the oil with higher acid value (e.g. 152) and the lower MeOH-insol. layer contained deacidified oil (acid value 51.0). The Lovibond values of the crude rice oil were 14.0 red, 36.1 yellow, and 6.1 blue; those of the acidic oil sol. in MeOH were 1.7 red, 20.9 yellow, and 0.40 blue. This color was darkened by excessive heating on MeOH recovery or by the use of Fe vessels. The continuous extraction with MeOH was superior to the batch method.

Preparation of unsaturated higher alcohols by catalytic reduction. XIII. Preparation of lead salts and preparation of higher alcohols by reduction under pressure. Saburô Komori, Yoshihiro Shigeno, and Tadao Tsujimura (Osaka Univ.). *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 103-5(1952). Lauric acid with PbO at 120-130° for 20-30 min. produced a 99% yield of Pb laurate. Pb salts could be obtained also from coconut fatty acids. Lauryl alc. was obtained in the yield of 85.4% by hydrogenating Pb laurate at 330° (initial pressure of H, 80 atm.).

Glycerides. IV. Monoacetin in the process of the reaction of acetic acid with glycerol. Issei Nakamori (Kyushu Univ., Fukuoka). *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 36-8(1952). Analysis and calculation of the course of the reaction of CH_3CO_2H with glycerol showed that the monoacetin produced was chiefly α-monoacetin, and the diacetin was chiefly α,α'-diacetin.

Separation and reduction of coconut oil fatty acids. Ryûzaburô Nozu, Hiroshi Watanabe, and Tadaichi Nishikawa (Kyoto Univ.). *J. Chem. Soc. Japan*, Ind. Chem. Sect. 55, 78-80(1952). The mixture of methyl esters of coconut oil fatty acids was analyzed by distillation. The % of Me ester to the original coconut oil was caproate 7.1, caprate 5.2, laurate 42.7, myristate 13.8, and palmitate 4.9. These esters were converted to corresponding alcohols by catalytic hydrogenation with Cu chromite at 195-87 atm. and 255°. The yield was over 90%. The conditions of hydrogenation and the physical properties of the products are tabulated.

Marine animal oils for edible purpose. Hideo Higashi (Fisheries Research Inst., Tokyo). *J. Japan Soc. Food Nutrition* 4, 4-7(1951). Some fish oils and shark-liver oils with iodine value lower than 130 and containing no squalene were as nutritious as soybean oil. Hydrogenation will improve sardine and other oils with iodine values around 166.

PATENTS

Nonadecyl glycols. C. H. McKeever (Rohm Haas Co.). *U. S. 2,599,468.* A product is claimed which consists of a mixture of 9-hydroxymethyl-1-octadecanol and 10-hydroxymethyl-1-octadecanol.

Insolubilizing and solidifying fatty acid esters of alcohols. M. Kronstein. *U. S. 2,599,397.* Higher fatty acid esters of alcohols characterized by their solubility in coal tar and petroleum solvents are treated with at least 4.5% of an organic peroxide at at least 90° to effect immediate and complete release of O_2 therefrom. A solidified reaction product is formed which is insoluble in coal tar and petroleum solvents.

Refining soybean lecithin. H. O. Renner (J. R. Short Milling Co.). *U. S. 2,599,016.* A process of refining commercial soybean lecithin is disclosed which consists of extraction of lecithin with 2-methoxy ethanol or 2-ethoxy ethanol and removal of the extract from the residue.

Recovery of sterols. H. G. Kirschenbauer (Colgate-Palmolive Peet Co.). *U. S. 2,598,269.* The gummy still bottoms residue obtained by distilling fatty acids obtained by hydrolysis of fatty materials at high temperature and pressure is saponified with alkali and the resulting mass is subjected to distillation in the presence of an inert gas at a temperature of at least 225° to vaporize the sterols.

Dehydration of castor oil. R. J. Carter and Mary Bristow (Imperial Chem. Ind. Ltd.). *U. S. 2,598,108.* A process for dehydrating castor oil is described in which the oil is heated in the presence of ortho-phosphorous acid.

Process for reducing organic compounds by hydrogenation. E. De Bartholomaeis and V. DeNora. *U. S. 2,597,074.* A batchwise system of catalytically hydrogenating fatty materials is claimed in which the reaction zone is maintained at operating temperature and pressure between runs. Volatile reduction products are removed from the hydrogen stream before it is recirculated.

Solvent extraction of admixed fatty acids. Pittsburgh Plate Glass Co.). *British 658,967.* A simple convenient method has been developed for separating saturated and unsaturated fatty acid mixtures into fractions having different degrees of saturation. This process comprises bringing the mixture into contact at room temperature with a 2-phase solvent system comprising a hydrocarbon solvent and a polar solvent combined, separating the immiscible solvents while the 2 phases are in the liquid state, and eliminating the solvents from the fatty acids in which they are dissolved. (*Chem. Abs.* **46**, 4820)

Separation of fatty acids and fatty acid derivatives. (Lever Brothers & Unilever Ltd.). *British 660,017.* The components of fatty oils or of the derivatives containing oily constituents can be separated or extracted by dialysis through a membrane of a high polymeric substance, such as rubber, which exhibits swelling with the substance to be dialyzed or with a solvent used in the dialysis. This process is used for (1) demucilaging of crude ground nut oil; (2) separation of phosphatides and oil; (3) extraction of Vitamin A fatty acid ester; (4) extraction of polymers; (5) separation of partial glycerides; (6) purification of extraction fat from the Ni catalyst; (7) purifying of poor herring oil; and (8) purifying refinery acid oils. (*Chem. Abs.* **46**, 4820)

• Biology and Nutrition

R. A. Reiners, Abstractor

The estimation of vitamin A in low potency oils. D. H. Laughland (Can. Dept. Agr., Ottawa). *Can. J. Tech.* **30**, 116-129 (1952). The use of the Morton-Stubbs correction in the analysis of low potency vitamin A containing oils may give rise to some difficulties unless the limitations of the method are understood. The results of a collaborative assay have been reported to provide information regarding interlaboratory differences. The nature of the absorption curve for the whole oil and nonsaponifiable fraction has been investigated. The necessity for extreme care in estimating the absorbancy of oil solutions and the importance of correct calibration of spectrophotometric equipment has been emphasized.

Research on processing procedures to improve the nutritive value of cottonseed meal. N. B. Knoepfler, H. L. E. Vix and F. H. Thurber (So. Reg. Res. Lab., New Orleans, La.). *Oil Mill Gaz.* **56** (11), 29(1952). Screw press cottonseed meals in which the cooking temperatures during processing were 200°F. had a much higher nutritive value than those cooked at 240°F. and the latter were much better than those cooked at 280°F. The gossypol content of oils produced at low temperature was as great at 1.0% as compared with 0.3-0.4% in oil from high temperature processing. Good oil could be produced from the high gossypol content crude oil only if it was immediately refined and bleached. Excellent hydraulic-pressed meals were prepared in the pilot plant by severe rolling of the moistened flakes followed by low-temperature, moist cooking, drying, and pressing.

Polyethenoid fatty acid metabolism. V. Prooxidant-antioxidant effect. P. W. Witten and R. T. Holman (Texas Agr. Exp. Station, College Station). *Arch. Biochem. Biophys.* **37**, 90-98 (1952). Growth response in fat-deficient rats was greatest when the diet was supplemented with linoleate and benzoyl peroxide (2 mg. daily), and was least when benzoyl peroxide was fed alone. The conversions of linoleate to arachidonate, linolenate to arachidonate, and linolenate to hexaenoate are not affected by supplementation with either tocopherol or benzoyl peroxide under the test conditions. However, conversion of linoleate to hexaenoate is greatly increased by feeding benzoyl peroxide with the test doses of linoleate.

Studies on vitamin-A in solution. Part V. Aqueous system. S. Bhattacharya and U. P. Basu. *Jour. Indian Chem. Soc.* **28**, 693-697(1951). Vitamin-A when dispersed in aqueous medium is found to be less stable at a pH of 3 than at 6. Hydroxy containing phenolic anti-oxidants such as propyl gallate showed no anti-oxidant properties.

Standardization of methods for the analysis of milk and dairy products in the Netherlands II. Determination of the percentage of fat in milk by Gerber's butyrometric method. II. Anon. *Neth. Milk & Dairy J.* **6**, 62-74(1952). The fat percentage of Netherlands cow milk is about 3.75% which gives an unsatisfactory result with Gerbers original milk pipette. A modified pipette of 10.77 ml. was adopted as the standard by a central committee for standardization in the Netherlands. This paper gives the complete details of their method.

Routine method for determining the fat contents of viscous milk products (gruels and custards) by means of the butyrometer. P. C. den Herder and F. Dijkstra. *Neth. Milk & Dairy J.* **6**, 18-35(1952). A routine butyrometric method was developed for determining the fat content of gruels and custards. A small calibrated glass replaced the milk burette because of the high viscosity of the products being tested. The sample was heated in the glass with H_2SO_4 to destroy the proteins, and then the Gerber method was followed from this point on.

Chromatographic investigations on cheese. I. A qualitative analysis of substances giving positive ninhydrin reactions. P. Simonart and S. Mayaudon. *Neth. Milk & Dairy J.* **6**, 1-17 (1952). The composition of a wide variety of cheeses was examined particularly for their amino-acid content.

The importance of the pH of culture media for the determination of the number of yeasts and bacteria in butter. K. Holwerda. *Neth. Milk & Dairy J.* **6**, 36-52(1952). The types of bacteria and/or yeasts which may be found in butter depends very often on the pH of the butter. A wide variety of bacteria and/or yeasts may be found at any time.

SINITIRO KAWAMURA

Synthetic study of vitamin A and related compounds. II. Synthesis of vitamin A acid. Saburô Akiyoshi and Keihei Ueno (Kyushu Univ., Fukuoka). *J. Chem. Soc. Japan*, Pure Chem. Sect. **73**, 126-8(1952). β-Ionylideneacetaldehyde (prepared by the method of Arens and Van Dorp [*Rec. trav. chim.* **67**, 973 (1948)]) with ethyl α-bromo-β-methylcrotonate gave δ-hydroxy ester, which was dehydrated to form the ethyl ester of the vitamin A acid. The product was purified by chromatography with active alumina.

• Drying Oils

Stuart A. Harrison, Abstractor

Physicochemical effects during the oxidative film formation from fatty acid esters. K. Meier. *Farbe u. Lack* **58**, 55(1952). Solutions in xylene of the methyl esters of oleic, linoleic, eleostearic, licanic, ricinoleic, dehydroricinoleic, and linseed fatty acids were run from 5 ml. pipets to measure comparative efflux viscosities at 20°. Below 25% ester concentration all viscosities were alike. As the ester concentration increased, however, the viscosity of the nonconjugated esters rose only moderately whereas the viscosity of the esters of conjugated acids and even more of the oxygen containing acids rose much more sharply. The conjugated double bonds appear to exert short-range intermolecular attractions similar to those exerted by the oxygen containing groups. Linseed oil samples blown with air for 0, 2, 4, 6, and 8 hours were used to make films on glass plates and their daily increase in weight determined. Contrary to expectation the induction period was lengthened rather than shortened by blowing. This is thought to indicate that the condition necessary for accelerated autoxidation is created only under the action of the van der Waals field of the substrate. (*Chem. Abs.* **46**, 5336)

Correlation of viscosity measurements. A. J. Ziegler. *Official Digest Federation Paint Varnish Production Clubs* **No. 329**, 378(1952). A chart is presented which correlates a large number of the viscosity measuring scales through the absolute unit, the poise.

The component fatty acids and glycerides of jute-seed oils. M. L. Meara and N. K. Sen. *J. Science Food Agric.* **3**, 237 (1952). The component seed fatty acids of two varieties of jute *corchorus capsularis* and *corchorus olitorius* were determined. The first resembles cottonseed oil in composition while the second more nearly resembles sunflower seed oil. Low temperature crystallization of the oils gives a non drying and drying oil fraction.

Water permeability of paint materials for wall construction. E. J. Dunn. *Official Digest Federation Paint Varnish Production Clubs.* **No. 329**, 365(1952). Reviews published work on the study of permeability of paint films. This includes description of permeability measurement methods, correlation between different tests and practical importance of permeability in home construction.

Copolymers in surface coatings. A. E. Errico. *Paint Varnish Production* **42**, 26(1952). A symposium on copolymers in surface coatings which discusses the theories of copolymerization, the type of materials which can be copolymerized, and the properties of products.

An investigation of the gaseous thermal-decomposition products of linseed oil. J. M. Horspool, K. A. R. Julian, and G. Oldham. *J. Applied Chem.* 2, 283(1952). The gases given off by linseed oil when used as a core binder were studied at different temperatures. It was found that the quantity of gas given off varied with the baking time at 230°. The most satisfactory baking time was one hour.

Tailor-made drying oil or alcohol-soluble boiled oil. Om Prakash, A. C. Gupta, and S. N. Chatterji. *Proc. Oil Technol. Assoc., India* 6, 57(1950). Linseed oil was interesterified with glycerol to render it compatible with alcohol or turpentine solutions of shellac. Optimun conditions were glycerol 10, linseed oil 100, and caustic 1 pt. heated to 240° for 4-5 hours. (*Chem. Abs.* **46**, 5336)

Styrene copolymers—recent work on the course of the reaction. F. Armitage and S. Kut. *J. Oil and Colour Chem. Assoc.* **35**, 195(1952). It is shown that when sorbic acid and β-eleostearic acid are reacted with styrene in xylene solution, a free radical copolymerization of the monomers proceeds simultaneously with the formation of a 1:1 Diels-Alder adduct. The former reaction predominates. The copolymerization is believed to proceed via a 1,4 addition of the styrene to the conjugated systems of the acids. A method is described for separating free polystyrene and the copolymer of styrene with fatty acid.

Ship-bottom paints. H. Rabaté. *Peintures pigments, vernis* **27**, 619(1951). The advantages and deficiencies of anticorrosive paints consisting of red lead and linseed oil, zinc yellow and other anticorrosive pigments are discussed. The effectiveness of different toxic agents is reviewed. (*Chem. Abs.* **46**, 5333)

The viscosity of standoils. M. Binde. *Farbe u. Lack* **58**, 63 (1952). Three methods of determining viscosity, Engler viscometer, Zeidler consistency scale, and Hoppler viscometer are described. A method for converting to absolute viscosity is given. (*Chem. Abs.* **46**, 5336)

Maleinization with maleic anhydride and fumaric acid. M. B. Hundert. *Am. Paint J.* **36** No. 38, 78(1952). Reviews the reaction of maleic anhydride and fumaric acid with rosin and with unsaturated fatty esters including esters of oleic, linoleic, and linolenic acid.

PATENTS

Styrenated products. R. W. H. Tess, R. H. Jakob, and T. F. Bradley. *U. S. 2,596,737.* A polyether is made from 2,2-bis (4-hydroxyphenyl) propane (4 mols), epichlorohydrin (5 mols) and sodium hydroxide (6.43 mols) in aqueous solution. The reaction is carried out at 100-105° for 80 minutes. The resulting polyether (1,200 pts.) is esterified with linseed fatty acids 1,875 pts.) by heating to 230° and holding for 6 hours. The ester is styrenated at 135° using di-t-butyl peroxide as catalyst. About 50 per cent of polymerized styrene is combined in this manner. The product has blood drying and film properties as well as excellent alkali resistance. Numerous variations in the procedure are given.

Insolubilizing and solidifying fatty acid esters of alcohols. M. Kronstein. *U. S. 2,599,397.* Oils, fats, waxes, and polyhydric alcohol esters are solidified by heating with an organic peroxide. For example, linseed oil is solidified by heating with 10 per cent t-butyl-perbenzoate until the temperature reaches 150°.

Modified styrene-linseed oil interpolymers. (B) Interpolymers of drying oils, mono alkenyl aromatic hydrocarbons, and divinyl benzene. *British 656,993.* Linseed Oil (1 pt.) is interpolymerized with a mixture of C_{8-10} alkenyl aromatic hydrocarbons (.5-1.2 pts.) consisting mainly of styrene mixed with α-methyl styrene (0-35%), and divinyl benzene in small percentages (approximately 2-4% of α-methyl styrene acid.) This gives a varnish suitable for brushing or dipping. (*Brit. Abs.* B11, 294 [1952])

Synthetic drying oils. *Dutch 68,954.* Polymers of allyl type alcohols are prepared by saponifying polymeric allyl esters. The polyalcohols form drying oils when esterified with unsaturated fatty acids. (*Chem. Abs.* **46**, 4813)

• Waxes

E. H. McMullen, Abstractor

Studies in waxes. IV. The saponification of wool wax and the recovery of the wax alcohols. C. S. Barnes, R. G. Curtis, and H. H. Hatt (Commonwealth Sci. & Ind. Research Organization, Melbourne). *Australian J. Applied Sci.* **3**, 88-99(1952). It is shown that wool wax is rapidly and completely saponified by either NaOH or KOH in aqueous EtOH and that severe methods of saponification are unnecessary. The products are readily separated by extracting the alcohols from the soaps and aqueous alcohol liquors by an immiscible solvent. This operation must be done under very carefully controlled conditions, otherwise unwanted phase distributions and troublesome emulsions are formed. Ten gallons of a 4% NaOH solution in 90% aqueous EtOH was refluxed, stirred, stirring and refluxing for a further 40 minutes, water was added to give a water-EtOH ratio of 2. The wool-wax alcohols were extracted with 20-gallon portions of light petroleum, the seven fractions weighing 5.13, 3.31, 2.70, 1.81, 1.19, 0.81, 0.44 lb., respectively. Acidification of the soaps gave 19.56 lb. of fatty acids. The apparent loss was 3.4%. (*Chem. Abs.* **46**, 4818)

Waxes used to process and protect metal. K. Rose. *Materials & Methods* **34**, No. 4, 104-107(1951). Four types of wax are described: animal, vegetable, mineral, and synthetic. A list of the applications available for these is given including electrical insulation, waterproofing, plasticising of rubbers, various lubricating applications, and surface coating for anticorrosion purposes. Possible modes of application are dipping or spraying with water and organic emulsions or solutions. (*Brit. Abs.* BI, Feb.. **1952**, 199)

Wax chemistry and technology. I. Waxes, a specific group. L. Ivanovszky. *Soap, Perfumery & Cosmetics* **24**, 999-1005(1951). Review and discussion concerning history, definition, classification, methods of production, uses, functions, and characteristics.

II. Chemistry, physics, and analysis. *Ibid.* 1107-12, 1130.

III. Individual waxes. *Ibid.* **25**, 176-81, 281-7(1952). (*Chem. Abs.* **46**, 4818)

Sugar-cane wax. I. L. F. Wiggins. *Proc. Brit. West Indies Sugar Technol.* **1949**, 24-8. Processes for recovering and refining cane wax from defecation muds are discussed. Benzene is a better solvent for the wax than toluene, naphtha, or heptane. West Indies wax resembles Louisianan and Australian waxes, but differs from South African and Philippine waxes. During vacuum distillation the wax probably undergoes inter- or intramolecular changes (esterification). (*Chem. Abs.* **46**, 4825)

Distribution of waxes in guayule. T. F. Banigan, Jr., J. W. Meeks, and R. W. Planck (U. S. Natural Rubber Research Sta., Salinas, Calif.). *Botan. Gaz.* **113**, 231-4(1951). The wax contents of nine parts of the guayule plant, segmented horizontally and each separated into bark and wood where possible, are reported. The wax occurs almost exclusively in the phloem above the crown and in the actively growing parts, and is constant in shrubs of the same age and variety harvested at different seasons. (*Chem. Abs.* **46**, 4833)

Plant wax. I. Constituents of the wax of Juniperus rigida. Atsushi Fujita and Toshiyoshi Yoshikawa (Univ. Kumamoto). *J. Pharm. Soc. Japan* **71**, 913-16(1951). The wax was found to contain caprylic, capric, lauric, palmitic, and montanoic acids in combination with ceryl and myricyl alcohols. It contains a compound having a molecular weight of approximately 2600, and on saponification gives ω-hydroxytetracosanoic acid which polymerizes into a gummy substance and the "etholide" of Bougault and Bourdier (a 7 molecule polymer). (*Chem. Abs.* **46**, 1783)

The waxy state. J. Scheiber. *Farbe u. Lack* **55**, 69-73(1949). This is considered as an intermediate between the fatty and resinous states, and its characteristic properties are the result of a mixture of microcrystalline and liquid material. The contention is elaborated with reference to beeswax and carnauba wax. (*Chem. Abs.* **46**, 3776)

Chemical composition of the wax of the white wax scale. Ceroplastes destructor. R. H. Hackman (Commonwealth Sci. Ind. Research Organization, Canberra, Australia). *Arch. Biochem. Biophys.* **33**, 150-4(1951). The wax extracted from the insects with hot $CHCl_3$, melted at 48.8°. The saponifiable fraction yielded colorless plates, melting at 83.8°, equivalent weight 419 and 420; ethyl ester, colorless plates melting at 64°. The unsaponifiable fraction contained no paraffin hydrocarbons but yielded a primary alcohol, melting at 80.0°; acetate melting at 62.6°. The alcohol on oxidation yielded an acid melting at 83.1°. The wax has an acid value of 21.5 and an acetyl value of 37.0. It is a mixture of C_{26} and C_{28} alcohols and acids free and combined. A similar wax was found in other members of the genus *Ceroplastes*. (*Chem. Abs.* **46**, 1661)

Evolution of analytical methods for the determination of the degree of purity of carnauba wax. Giampiero Camperio. *Ind.*

vernice (Milan) 5, 156-60 (1951). A discussion of the study of Marsel (*Chem. Specialties Mfrs. Assoc., Chicago*, Apr., 1951) on the selective solvent extraction of 10 g. of carnauba wax with 100 ml. of toluene or $CHCl_3$ at 25°. (*Chem. Abs.* **46**, 1783)

Diffusion of waxes through rubber. L. L. Best (Northern Polytech., London). *Brit. J. Applied Phys.* **2**, 237-8 (1951). A simple apparatus is described for accomplishing part of the diffusion operation. It is used in conjunction with a microscope to view the results. The rubber mix is vulcanized, one edge being confined by a straight piece of Al. After vulcanization the Al is removed and molten wax is placed along the straight edge. The wax is cooled rapidly to limit its diffusion into the rubber. The sample is then placed in the apparatus mentioned above where it is cooled to room temperature under controlled conditions. The wax crystals farthest from the interfacial boundary form a plane front through the rubber parallel to the interfacial boundary. The crystals are viewed under polarized light. (*Chem. Abs.* **46**, 3333)

Hydroxy acids in beeswax. Y. Toyama and H. Hirai (Univ. Nagoya). *Fette u. Seifen* **53**, 556-7 (1951). In an attempt to isolate an isomer of 14-hydroxypalmitic acid, further work was done on the lower-melting hydroxypalmitic acid fraction of beeswax fat acids. Oxidation of the mixture yielded a mixture of dibasic acids consisting mainly of HO_2C-$C_{12}H_{24}CO_2H$. Thus the CO_2H of the sought isomer is probably near the C_{14}. Lactonization of 14-hydroxypalmitic acid with 2-naphthalenesulfonic acid in benzene yielded 14-ethyltetradecanolide, b_3 140-50°, d^{20} 0.9368, n^{20} 1.4690. (*Chem. Abs.* **46**, 3776)

A method for determining the apparent melting point of resins, waxes, etc. J. Pickthall. *J. Soc. Cosmetic Chemists* **2**, 157 (1951). Fifty grams of Hg is poured on top of the sample which has been melted and allowed to solidify in a test tube. A thermometer rests on the sample under the Hg. The sample tube is heated slowly in a stirred bath. The apparent melting point is the temperature at which the sample rises through the Hg. (*Chem. Abs.* **46**, 3330)

Characterization of petroleum waxes by solvent fractionation methods. F. H. McLaren (Standard Oil Co. of Indiana, Whiting). *Tappi* **34**, 462-8 (1951). Two methods are given for the solvent fractionation of waxes. In the first, the wax is dissolved in 50-50 solution of acetone and hexane. The solution is chilled to −60°F. and filtered. The first fraction is recovered from the filtrate. The precipitate is redissolved, chilled to −55°F., and filtered, etc. In the second method the wax is dissolved in hexane, cooled to 80°F. and filtered to recover the first fraction, the filtrate cooled to 60°F. and refiltered, etc. The first method is applicable to all petroleum waxes; the second is unsatisfactory for paraffin waxes, but excellent for residual waxes and their blends with polymers and resins. Typical melting point distribution curves are shown and interpreted. For instance, a brittle microcrystalline wax not suitable for laminating use had much less low-melting material than did two other microcrystalline waxes which were excellent for laminating. (*Chem. Abs.* **46**, 4778)

PATENTS

Amides of θ, ι-epoxystearic acid. J. T. Scanlan, D. Swern, and E. T. Roe (to U.S.D.A.). *U. S. 2,567,237.* Derivatives of θ,ι-epoxystearamide and of N-substituted θ,ι-epoxystearamide, and amides of other monoethenoic acids such as elaidic, ricinoleic, petroselaidic, myristoleic, palmitoleic, vaccenic, etc., useful as high melting-point waxes and as intermediates for wetting agents, plastics, etc., are made by AcO_2H oxidation of the corresponding amide, where R may be H, or an alkyl, hydroxyalkyl, aryl, or acyl group. The N-(hexyl)oleamide on oxidation gives a 49% yield of N-hexyl-θ,ι-epoxystearamide, melting at 66.0°, and with an oxirane 0 content of 4.14%. The melting point of N-substituted θ,ι-epoxystearamides where R = H, Me, decyl, dodecyl, Ph, 1-$C_{10}H_7$, $HOCH_2CH_2$, and Ac are, respectively, 94.0°, 65.5°, 75.0°, 78.5°, 83.0°, 81.0°, 81.5°, 74.0°. (*Chem. Abs.* **46**, 3560)

Emulsions. Mathijs van der Waarden (to Shell Development Co.). *U. S. 2,577,218.* Stable, non-gelling, W/O emulsions for cosmetic, industrial, and pharmaceutical uses can be prepared from wool-fat acids, made by saponifying wool grease under pressure with KOH in EtOH. The fatty acids can be recovered by precipitation with mineral acids. Four parts of the fatty acids with 1 part of rosin and 95 parts of spindle oil formed a clear oil which produced a W/O emulsion with 600 parts of H_2O. White oils and fixed oils can be used in place of the spindle oil. (*Chem. Abs.* **46**, 4257)

Solvent fractionation of wax-containing mixtures. E. W. Clarke (Atlantic Refining Co.). *U. S. 2,578,510-2.* A wax-containing mixture is separated into fractions of higher and lower melting point by countercurrent extraction with a solvent, such as $PhNO_2$, PhOH, C_5H_5N, or Me_2CO, and a solvent additive, such as *paraffinic naphtha*, furfural, $PhNH_2$, $MeCH_2NO_2$, $EtCH_2NO_2$, CH_2:CH_2PhOH, cresol, or diethylene glycol, at a temperature such that two liquid phases are formed. (*Chem. Abs.* **46**, 3754)

Wax compositions. J. M. Butler (Monsanto Chemical Co.). *U. S. 2,580,996.* Wax composition for the impregnation and coating of fibrous products, such as textiles, leather, wood, and in particular paper, for use in wrapping food stuffs is improved in gloss, adhesiveness, hardness, tensile strength, and elongation characteristics by the addition of from 60 to 0.02% (preferably 40 to 0.02%) of an alkylated polystyrene to a hydrocarbon wax or such earth waxes, as ceresin or ozocerite. The polystyrene of molecular weight 60,000 to 1,000,000 is alkylated with a polymerized propylene (preferably the trimer of boiling point 175-225°) and the product mixed thoroughly with the hydrocarbon or other wax. (*Chem. Abs.* **46**, 4256)

Bringing wax into contact with clay to deodorize and decolorize the wax. P. F. Dougherty and C. H. Brooks (Sun Oil Co.). *U. S. 2,585,895.* A slurry of petroleum wax and clay is heated to a temperature of 50 to 300°F. above the melting point of the wax, flashed into a vacuum, and then quickly cooled to a temperature 20 to 40°F. above the melting point of the wax to cause vaporization and absorption on the clay of compounds which otherwise would later develop into odoriferous substances. The molten wax is separated from the clay by a rotary vacuum filter operated under an inert atmosphere. (*Chem. Abs.* **46**, 4256)

Solvent extraction of oils, fats, and waxes from discrete organic substances, such as oleaginous seeds. Bamag Ltd. and B. Bregman. *British 667,262.* (*Chem. Abs.* **46**, 3304)

• Detergents

Lenore Petchaft, Abstractor

Measurement of detergency. M. Ringeissen. *Ind. Textile.* **68**, 38-9 (1951). Fabrics soiled with a grease containing a dye are compared in six spectral regions after laundering under standard conditions. Results are reproducible and correlate well with those obtained in practice. (*Chem. Abs.* **46**, 5345)

Toilet soap—problems in production. P. F. Spencer. (Cussons Sons & Co., Ltd., Manchester, England). *Perfumery, Essent. Oil Record.* **43**, 154-6, 183 (1952). Review of problems involved in production of high-grade toilet soap caused by improper moisture content, and addition of such additives as colors and perfumes.

Detergents from petroleum. S. F. Birch. *J. Inst. Petroleum* **38**, 69-87 (1952). Methods of producing these detergents are reviewed, largely on the basis of patent specifications. The method of action of the various classes of detergents are discussed. The bulk of the anionic detergents now being produced from petroleum fall into 3 main chemical types: (1) secondary alkyl sulfates, (2) alkanesulfonates, and (3) alkylarylsulfonates. Flow sheets are presented for the manufacture of these detergents. Since the commercially available detergents of the nonionic type are all derivatives of ethylene oxide, and much of the ethylene oxide is now of petroleum origin, these detergents as a class can be partly or wholly derived from petroleum. Flow sheets are given for two types of nonionic products. (*Chem. Abs.* **46**, 5345)

Study of detergent power of detergents chosen for scouring of grease wools. M. van Overbeke, L. Lefebvre and L. Raux. *Bull. inst. textile France* **No. 21**, 59-66 (1950). Detergents for wool scouring are evaluated by a 5-bath trial procedure duplicating mill practice. Thirty-g. samples of grease wool are scoured with the detergents in question, and are then tested for completeness of scouring and for damage to the wool. Ethylene oxide condensates with lauric acid are found most desirable. (*Chem. Abs.* **46**, 4806[1952])

Toxicity to eye mucosa of certain cosmetic preparations containing surface-active agents. J. H. Draize and Elsie A. Kelley (Food and Drug Administration, Washington, D. C.). *Proc. Sci. Sect. Toilet Goods Assoc.* **No. 17**, 1-4 (1952). Surface active agents penetrate the eye mucosa freely to produce lesions. The combination of surface active agents in a preparation may produce irritation in excess of that which might be inticipated by the simple addition of the irritant affects of the individual agents. Although wide variations in toxicity were observed in the anionic and nonionic groups, the most generalized statement concerning toxicity remains cationic > anionic > nonionic.

Relation of surface active properties to irritation of the rabbit eye. L. W. Hazleton (Hazleton Laboratories, Fall Church, Va.). *Proc. Sci. Sect. Toilet Goods Assoc.* **No. 17,** 5-9(1952). In this survey some physical, chemical, and physiological characteristics of surface active agents have been reviewed in terms of their capacity to produce eye injury. It appears that neither pH nor surface tension contribute to eye irritation. Good wetting powers within the nonionics appear to correlate with irritation, but this is not true with the cationics. On the other hand, while foaming per se is not critical, most of the irritant materials are at least relatively good foamers. Perhaps the best partial correlation is that good wetters which are good foamers are likely to be irritant. From these tests it appears that the previously recognized order of cationics > anionics > nonionics is still the best initial criterion for prediction of eye irritant potential. Within each of these groups, chemical and physical properties may serve as guides in predicting the potential but none is sufficiently critical to substitute for the eye as a final test.

Phase transitions in alkaline-earth soaps. G. V. Vinogradov. *Doklady Akad. N auk S.S.S.R.* **58,** 73-5(1947). Thermal analysis was applied to Ca, Ba, and Sr stearates. These salts are microcrystalline solids that fused into glassy solids above 120°. Air-dried specimens retain some 3% H_2O even after extensive drying in vacuo; usually over 0.5% H_2O is present after several hours in vacuo at 50°. Ca stearate shows endothermic effects at 120-35° and 160-70°, and indications of one at 90-110°; after the first effect the salt changes to plastic state capable of being drawn into thin fibers, which explains the utility of such materials as greases. A re-heating, after cooling, shows an endothermic effect at 130-45°. Ba stearate shows endothermic effects at 120-30°, 160-70°, 200°, and 240°; the second is most reproducible, and on cooling curves there appears an exothermic effect at 110-20°. Sr. stearate shows endothermic effects at 110-30° and 160-70°, while on cooling an exothermic effect at 100° appears. All the salts give the plastic modification noted above. (*Chem. Abs.* **46,** 4819)

Solutions of metal soaps in organic solvents. III. The aggregation of metal soaps in toluene, isobutyl alcohol, and pyridine. S. M. Nelson and R. C. Pink. *J. Chem. Soc.* **1952,** 1744-50. The degree of aggregation of a number of metal soaps in toluene, isobutyl alcohol, and pyridine has been investigated by an ebullioscopic method. In toluene, the degree of aggregation is strongly dependent on the length of the hydrocarbon chain and on the polarity of the soap. The more polar soaps form larger micelles or are insoluble, while increase of chain length in soaps of the same metal leads to a decrease in the degree of aggregation. In the polar solvents isobutyl alcohol and pyridine, the associating effect of the soap dipoles is reduced and unimolecular solutions are obtained at low concentrations. Stability of the soap micelles is due to dipole interaction and to the decrease in interfacial energy which occurs on aggregation.

Critical micelle concentrations by a bubble pressure method. A. S. Brown, R. U. Robinson, E. H. Sirois, H. G. Thibault, W. McNeill, and A. Fofias (Colgate U., Hamilton, N. Y.). *J. Phys. Chem.* **56,** 701-5(1952). The bubble pressure method of measuring surface tensions has been modified by using it kinetically. The air pressure needed to maintain a stream of bubbles in a surfactant solution is a complex function of concentration and bubble rate. Plots of bubble pressure against concentration have been obtained for the commercial cationic and anionic materials laurylpyridinium chloride, Santomerse No. 3 and Tergitol TMN-650. The major discontinuities were confirmed as critical micelle concentrations by conductance measurements for the L.P.C., and Santomerse. The utility of the method for solutions containing electrolytes has been examined briefly for Santomerse in 1.84% sulfuric acid.

Determination of anionic synthetic detergents in sewage. H. C. Evans. *J. Soc. Chem. Ind. (London)* **69,** Suppl. No. 2, S76-80 (1950). The concentration of alkyl or aryl sulfates or sulfonates, such as sulfonated castor oil, Na do-decylbenzene-sulfonate, or Na tetradecanesulfonate, in sewage can be determined by measuring the light absorption at 6520 A. of the $CHCl_3$ extract of the methylene blue salts of the detergents at pH 3.25 and 0.7; plotting concentrations against pH units; extrapolating the curve; and reading the concentration indicated at the intersection at pH 2.0. Interference is not produced by $Al_2(SO_4)_3$, $FeCl_3$, soap, CNS, NO_3, Cl or inorganic ions and compounds from urine. (*Chem. Abs.* **46,** 4708[1952])

Analysis of synthetic detergents. G. Gauthier and L. Mazau. *Ann. pharm. franc.* **9,** 678-90(1951). For sulfate esters of aliphatic alcohols it is sufficient to determine the free alcohols. If the total of free alcohols is very low, the mineral salts must be determined. In the case of Sipol wax, determine the H_2SO_4 after hydrolysis. With N-alkylarylsulfonates determine the average molecular weight. The method with p-$H_2NC_6H_4Me$ of Marron and Schifferli is used. (*Chem. Abs.* **46,** 4819)

Measurement of fabric soiling. E. A. Leonard (Alexander Smith Carpet Co., Yonkers, N. Y.). *Am. Dyestuff Reptr.* **41,** 322-40(1952). The history of publications on measurements of fabric soiling is reviewed. From these it can be concluded that there are two factors in soiling—the mechanisms of impingement and the mechanisms of retention. Three test methods were developed. The test methods were designed to evaluate in a controlled way the various mechanisms by which soil may impinge upon textiles. The examples used demonstrate the importance in soil retention of fabric construction, type of fiber and type of finish. The three methods were a blower test which causes impingement of soil from an air stream by interception and inertial effect, a tumbler test which causes impingement of soil by deposition and transfer (probably also some direct interception) and a floor test, which produces impingement principally by deposition and transfer. Results show that the tumbler test may prove to be the nearest possible approach to a universal method of soiling evaluation.

Electrocapillary analyses of detergents. W. Kopacz. *Chimie et Industrie* **67,** 761-766(1952). A new classification of synthetic detergents based on the application of electrocapillary analysis to paper chromatography has been devised which differs from the classification based on their chemical composition. In the applications of these substances (detersive, wetting, washing, and dispersive properties) only the physical, chemical, and colloidal states fall in line.

SINITIRO KAWAMURA

Colloid-chemical studies of metal soaps. I. Viscosity of the aluminum stearate organosol. Tomoji Inoue and Yasuji Iida (Aichi Gakugei Univ., Nagoya). *J. Chem. Soc. Japan,* Pure Chem. Sect. **73,** 71-3(1952). Aluminum mono- and distearates in benzene or tolluene gave a max. in the viscosity-temperature curves in some cases. This peculiarity was discussed in relation to the mol. structure.

II. Dielectric studies of aluminum distearate. Tomoji Inoue, Hajime Shiio, Yukio Kurita, and Toshirô Mizuno. *Ibid.* 73-5 (1952). Benzene solution of Al distearate showed very low value of orientation polarization in concentrations higher than 0.6%, and this value increased abruptly when the sol. was diluted. This increase was prominent at higher temperature. Thus Al distearate was associated in very dilute solutions, and dissociated at higher temperatures.

Preparation of propanolamine oleate. Masumi Saitô and Minoru Imoto. *J. Chem. Soc. Japan,* Ind. Chem. Sect. **55,** 22-4 (1952). β-Hydroxy-n-propylamine, dipropanolamine, and tripropanolamine were separately reacted with oleic acid at 120-140° for 100-168 hrs. in a sealed tube. Surface activity of the propanolamine oleate was examined.

PATENTS

Detergent composition. C. B. Durgin (Monsanto Chemical Co.). *U. S. 2,594,257-8.* A non-oily, free flowing detergent composition consisting of the condensation product of a tertiary mercaptan or tall oil with ethylene oxide, tetrasodium pyrophosphate, attapulgite (which reacts with the active ingredient to lower soil redeposition during washing and provide a softer hand of textile products), sodium sesquicarbonate, carboxymethylcellulose and water of crystallization.

Method of spraying freely falling particles. J. J. Mahoney (Colgate-Palmolive-Peet Co.). *U. S. 2,594,469.* Apparatus for forming synthetic detergent particles and coating solid particles with sodium silicate to make them dust-free and free flowing.

Hard toilet soap. J. V. Loon and N. V. Dobbelman. *British 664,484.* In the production of a good toilet soap it is desirable that the soap should possess a fine gloss. Hardness of the soap results in economy in use, while a fine gloss or polish renders the soap more salable. A much harder toilet soap can be produced by superfatting a soap containing at least 25%, and preferably 30% or more, of saponified lower fatty acids, adding relatively high proportions of unsaponified fatty acids, and reducing the water content to less than 10% and preferably less than 9%.

Cleansing compositions. J. J. Ayo, F. J. Gajewski and H. L. Sanders (General Aniline & Film Corp.). *British 671,358.* A detergent composition comprising a highly foaming hydroxy polyalkyleneoxy ether (polyglycol ether), an alkali metal (potassium) soap from palm kernel oil, alkali metal pyrophosphate (tetrapotassium pyrophosphate) and a corrosion inhibiting agent (sodium metasilicate).