

• Fats and Oils

CHROMATOGRAPHIC ANALYSIS OF TALL OIL. A.S. Fomin et al. *Lakokras. Mat.* 1974, No 1, 38-40. Accurate chromatographic analysis of tall oil was carried out by using chromatograph LKhM-8M, thermal conductivity detector and a column loaded with Celite C-22 supporting diethylene glycol maleopimarate. Interpretation of the various peaks and indication of tall oil composition are presented. (World Surface Coatings Abs. No. 387)

PENTANE FORMATION AND RANCIDITY IN VEGETABLE OILS AND IN POTATO CHIPS. K. Warner, C.D. Evans, G.R. List, B.K. Boundy and W.F. Kwolek (USDA Northern Regional Res. Lab., ARS, Peoria, IL 61604). *J. Food Sci.* 39, 761-5 (1974). Pentane formation during initial stages of autooxidation is indicative of rancidity in aged vegetable oils and potato chips. Pentane in the headspace gas of oils and chips was measured by gas chromatography. An 18-member taste panel evaluated the samples for development of rancid odors and flavors. Significant linear correlations were obtained between the amount of pentane developed and the number of rancid descriptions. Samples needed only 0.08 ppm pentane in the headspace to be described as rancid by 90% of the panel. Both oils and chips were more stable to the development of pentane as the linoleate content and the iodine value decreased.

MODIFICATION OF THE AOAC QUALITATIVE TEST FOR THE DETERMINATION OF FISH OILS AND MARINE OILS. W.G. Schwien (Food and Drug Admin., 1009 Cherry St., Kansas City, MO 64106). *J. Assn. Off. Anal. Chem.* 57, 1005 (1974). The current AOAC test for the detection of fish oil in vegetable oils, 28.098, can be readily modified through the use of diluted bromine solution and Wijs solution. This modification allows the precipitation of octabromides, which is indicative of the presence of fish oils, to be observed more clearly. Since the precipitate can be measured, the test can be semiquantitative when known fish oil mixtures are treated in a like manner. This modification has been adopted to replace 28.098.

GAS-LIQUID CHROMATOGRAPHIC METHOD FOR THE DETERMINATION OF MILK FAT IN COCOA BUTTER EXTRACTED FROM MILK CHOCOLATE. J.L. Iverson (Div. of Food Technol., Food and Drug Admin., Washington, D.C. 20204). *J. Assn. Off. Anal. Chem.* 57, 934 9 (1974). A rapid extraction procedure was selected for obtaining lipids from milk chocolate and a gas-liquid chromatographic method was developed for estimating milk fat in chocolate. The milk fat content of milk chocolate can be estimated on the basis of the lauric acid content of the lipid extract of milk chocolate. The method is accurate in detecting 10-30% milk fat, the range of practical interest in cocoa butter. Data were obtained on the fatty acid composition of 8 authentic cocoa butter-milk fat samples, 11 domestic milk chocolates, 11 imported milk chocolates and 9 miscellaneous chocolates.

COMPARISON OF PYRROLIDIDES WITH OTHER AMIDES FOR MASS SPECTRAL DETERMINATION OF STRUCTURE OF UNSATURATED FATTY ACIDS. B.A. Anderson, W.H. Heimermann and R.T. Holman (Hormel Inst., Univ. Minn., Austin, Minn. 55912). *Lipids* 9, 443 49 (1974). Amides of unsaturated fatty acids give mass spectra which indicate the locations of the double bonds. A survey of several amides of oleic acid was made to evaluate which amide might be most suitable for routine use in elucidation of structure. Methods of preparation of several amides of oleic acid are presented with mass spectral and gas chromatographic data. Tertiary amides have most easily interpretable mass spectra which indicate the position of the double bond in the fatty acid chain. The pyrrolidide is advantageous for fatty acid gas liquid chromatography and mass spectrometry analyses because it can be prepared easily in high yield on a microscale, it is volatile enough for gas liquid chromatographic separations and it has a simple and easily interpretable mass spectrum which indicates the structure.

STEROL LIPIDS ISOLATED FROM PEA SEEDS (*PISUM SATIVUM*). T. Miyazawa, S. Ito and Y. Fujino (Dept. Agr. Chem., Obihiro Chikusan (Zootechnical) Univ., Obihiro, Hokkaido, Japan). *Cereal Chem.* 51, 623 9 (1974). Four classes of sterol lipids—

free sterols, sterol esters, sterylglucosides, and acylsterylglucosides—were isolated from pea seeds, identified and examined for fatty acid and sterol composition as well as sugar constituents. β -Sitosterol was the principal free sterol. The sterol esters were mainly composed of linoleic acid and β -sitosterol, sterylglucosides of glucose and β -sitosterol, and acylsterylglucosides of palmitic acid, glucose and β -sitosterol.

HEMOGLOBIN PEROXIDATION TEST SCREENS ANTIOXIDANTS. W.M. Cort (Food and Agr. Prod., Prod. Dev. Dept., Hoffmann-LaRoche Inc., Nutley, N.J. 07110). *Food Technol.* 28(10), 60-6 (1974). An antioxidant screening test has been developed that indicates within 5 minutes whether a compound has antioxidant activity. Hemoglobin is used to peroxidize a 10% safflower oil emulsion, and oxygen removal from the emulsion is measured with an oxygen analyzer. This article describes the test procedure and presents results on various chemicals and natural materials such as spices.

EFFECTS OF GENOTYPE AND PRODUCTION AREA ON THE FATTY ACID COMPOSITION, TOTAL OIL AND TOTAL PROTEIN IN PEANUTS. C.E. Holaday and J.L. Pearson (USDA National Peanut Res. Lab., P.O. Box 637, Dawson, GA 31742). *J. Food Sci.* 39, 1206-9 (1974). A research project to study the effects of peanut genotype and growth habitat on total oil and protein and the fatty acid composition of the oil is reported. Three different varieties or genotypes of each of the three major market types of peanuts (Spanish, Runner and Virginia) were studied. The peanuts were grown in seven locations throughout the three major growing areas of the United States. Genotype differences and location temperatures after pegging significantly affected the total oil content and the fatty acid composition of the peanut oil. Peanuts grown in those locations where the mean temperature was lowest after pegging had significantly higher levels of linoleic acid and lower levels of oleic acid. The Spanish were less affected than either the Runner or Virginia types. Although the total oil tended to decrease with decreasing temperature after pegging the effect was not significant at the 5% level or better. The protein was unaffected by various regimes of temperature.

FATTY ACID COMPOSITION OF MUSCLE PHOSPHOLIPIDS FROM CALVES, AND GROWING AND MATURE STEERS FED PROTECTED SAFFLOWER OIL. W.I. Kimoto, R. Ellis, A.E. Wasserman, R. Oltjen and T.R. Wrenn (USDA Eastern Reg. Res. Ctr., ARS, Philadelphia, Pa 19118). *J. Food Sci.* 39, 999-1001 (1974). Calves and growing and mature steers with increased linoleic acid (18:2) content in the phospholipids were reared. Rump roasts from animals fed protected and unprotected safflower oil showed average 18:2 levels from 38.3-40.3% and from 19.8-29.0%, respectively. Tocopherol levels for the rump roasts varied from 4.2-7.3, 2.7-4.9 and 2.7-3.1 $\mu\text{g/g}$ of sample for the calves, and growing and mature steers, respectively. Lipid oxidation of ground portions from the rump roasts stored at 3C for 3 days was followed by the 2-thiobarbituric acid (TBA) method. Variation in the TBA numbers was minor for mature steers, and not significant for the growing steers fed protected and unprotected safflower oil.

THERMAL INTERACTION OF AMINO ACIDS AND TRIGLYCERIDES. VALINE AND TRICAPROIN. Y.C. Lien and W.W. Nawar (Dept. of Food Sci. Nutr., Univ. Mass., Amherst, MA 01002). *J. Food Sci.* 39, 917-9 (1974). A model system consisting of a mixture of valine and tricaproin was used to study the interaction of fats and amino acids during heat treatment. New decomposition products, not formed originally when each compound was heated alone, were identified from the heated mixture. These were caproic amide, N-isobutylcaproic amide and caproic nitrile. Mechanisms involving the interaction between the amino acid and the triglyceride are introduced to explain the formation of these products. In addition, certain compounds normally produced when the triglyceride or the amino acid are heated separately were absent when the mixture of both was similarly treated. This is explained on the basis of reactions which take place preferentially when both compounds are heated together.

STABILITY OF CANDY FATS. N.E. Harris, A.S. Henick, I. Bloch and D.E. Westcott (Food Lab., U.S. Army Natick Labs., Natick, MA 01760). *J. Food Sci.* 39, 1263-4 (1974). The pur-

pose of this study was to find a way to predict resistance of candy fats to hydrolytic rancidity development. The use of a ratio of free fatty acid after heat treatment divided by the initial free fatty acid of coconut fats proved to be a reliable index for predicting stability in a fat/fondant confection. However, the method cannot be used for predicting the stability of domestic fats where oxidative rancidity was found to be more of a problem.

AN EVALUATION OF A SPECIFIC GRAVITY RAPID FAT ANALYZER. D.C. Egberg, R.H. Potter and G.R. Honold (General Mills, Inc., James Ford Bell Tech. Ctr., Minneapolis, MN 55427). *J. Food Sci.* 39, 1261-2 (1974). A study was conducted comparing the Foss-Let fat analyzer with an AOAC extraction method for a variety of snack products, pie crust mixes, baking mixes, frostings and meat. The rapid method compared favorably with the AOAC technique showing a 0.998 correlation coefficient for the 192 samples studied. Reproducibility data were obtained demonstrating the rapid method to be as reproducible as the AOAC method for the materials studied.

GRAPESEED OIL: A RICH SOURCE OF LINOLEIC ACID. J.E. Kinsella (Dept. of Food Sci., Cornell Univ., Stocking Hall, Ithaca, NY 14850). *Food Technol.* 28(5), 58-60 (1974). Grape seed oil pressed from solid residues from wine manufacture can be used as an edible and cooking oil. Its composition renders it very desirable for inclusion in foods and diets designed for lowering saturated fatty acid intake.

REDUCING THE SURFACE OIL CONTENT OF FRIED PRODUCTS. M. Nonaka, M.L. Weaver and R.N. Sayre (Western Reg. Res. Ctr., USDA, ARS, Berkeley, Calif. 94710). *Food Technol.* 28(5), 50-4, 65 (1974). This article describes a process for reducing the surface oil content of French fried potatoes and various potato and corn snacks. The process involves contacting the fried product with liquid difluorodichloromethane, whereby the excess surface oil and oil emulsified in the surface layers is removed. The procedure obviates some of the nutritive imbalance and the oily appearance of many fried products and in many instances improves the texture and flavor.

CIS AND TRANS ISOMERIZATION IN SOME POLYETHYLENIC C₁₈ FATTY ACIDS: OPEN-TUBULAR GLC DATA FOR THE LIQUID PHASES BUTANEDIOLSUCCINATE, SILAR-5CP, AND APIEZON-L. R.G. Ackman and S.N. Hooper (Dept. of the Environment, Fisheries and Marine Service, Halifax Lab., 1707 Lower Water St., Halifax, N.S., B3J 2R3, Canada). *J. Chromat. Sci.* 12, 131-8 (1974). The GLC behaviour of methyl esters of unsaturated fatty acids representing cis and trans Δ^6 , Δ^{12} and Δ^{18} isomers of 6 mono-, 12 di- and 8 triethylenic acids has been compared on open-tubular columns coated with butanediolsuccinate polyester, or Silar-5CP or Apiezon-L. The parent all-cis triethylenic acid was partially converted to geometrical isomer mixtures and the products reduced to yield mono- and diethylenic products. Under the operating conditions employed, trans unsaturation alone or with cis compounds reduces elution times sufficiently on Silar-5CP phase relative to butanediolsuccinate to permit identification by the approach of small peak shifts on two polar phases of slightly differing polarity. Apiezon-L was less successful with complex mixtures owing to overlap among components of varying degrees of unsaturation.

FATTY-ACID ESTERS OF ALKOXYLATED POLYOL GLYCOSIDES AS EMULSIFIERS IN WHITE LAYER CAKE. L.T. Kissell (Soft Wheat Qual. Lab., Ohio Agr. Res. & Dev. Ctr., Wooster, Ohio 44691), D.K. Mechem and C.L. Mehlretter. *Cereal Chem.* 51, 616-23 (1974). Functional emulsification properties were determined for a series of products prepared by direct reaction of glycerol or propylene glycol with starch or lactose in acid media, followed by alkoxylation with ethylene oxide and propylene oxide, and esterification to introduce one or two fatty acid residues. All materials were tested in white layer cake batters prepared according to AACC Methods. Most effective, and comparing favorably with commercial mono- and diglyceride emulsifiers, were mono- and dipalmitates of moderately ethoxylated propylene glycol and glycerol glycosides. Many of the emulsifiers were too powerful for the high sugar:shortening ratio cake formulation. All surfactants were re-evaluated at reduced levels of total added fat. With the emulsifiers held constant at 2% of the fat, improvement was noted in volume, contour and grain of cakes by reduction of shortening from 50% to the 35-25% range. Preliminary studies to determine the safety of these materials as food additives are underway.

PHYSICAL STUDIES OF EGG PHOSPHATIDYLCHOLINE IN DIETHYL ETHER-WATER SOLUTIONS. P.H. Poon and M.A. Wells (Dept. of Biochem., College of Med., University of Ariz., Tucson,

Ariz. 85724). *Biochem.* 13, 4928-36 (1974). The binding of water to phosphatidylcholine micelles in diethyl ether has been measured by equilibrium dialysis using acetylated dialysis tubing. As the free water concentration of the solvent increases to about 5 mg/ml, the number of water molecules bound per molecule of lipid increases to about 6-7. This water binding appears to follow a simple association equilibrium. As the free water concentration is increased toward the saturation level of the solvent (~9 mg/ml), there is a marked increase in the number of water molecules bound, approaching a limit of approximately 60 molecules/molecule of lipid. This second phase appears to involve cooperative binding of water. Further support for the interaction of water and phosphatidylcholine was derived from density and refractive index increment measurements. Analysis of the activity of *Crotalus adamanteus* phospholipase A₂ shows that the enzyme is active only in the second hydration state.

THE NATURE OF WATER INSIDE PHOSPHATIDYLCHOLINE MICELLES IN DIETHYL ETHER. M.A. Wells (Dept. of Biochem., College of Med., Univ. of Arizona, Tucson, Arizona 86724). *Biochem.* 13, 4937-42 (1974). The properties of water bound phosphatidylcholine micelles in diethyl ether solutions have been investigated by infrared and pmr spectroscopy, by measuring the fluorescence properties of N-dansylphosphatidylethanolamine (dansyl-PE) incorporated into the micelles and by the spectral properties of CoCl₂·6H₂O incorporated into the micelles. Infrared studies of the OH stretching of HOD show the existence of two types of bound water. The first which occurs at low water concentrations is characterized by an absorption band at 3530 cm⁻¹ and a narrow band-width. The second form, which coexists with the first is found at higher concentrations of water, and has infrared properties similar to those of pure water. Pmr studies do not show two forms of bound water; however, the two states can be distinguished by the shorter relaxation times of the water protons bound in the first state. As the amount of water bound to the micelle increases there is a marked increase in the relaxation time of the polar head group of the lipid. In addition the depolarization of dansyl-PE fluorescence decreases. The relationship of the properties of bound water to the activity of *Crotalus adamanteus* phospholipase A₂ shows that the enzyme is only active when dissolved within a micelle, which contains bound water whose properties closely resemble those of pure water.

NUCLEAR MAGNETIC RESONANCE STUDIES OF THE INTERACTION OF ALAMETHICIN WITH LECITHIN BILAYERS. A.I.Y. Lau and S.I. Chan (Arthur Amos Noyes Lab. of Chem. Physics, Calif. Inst. of Technol., Pasadena, Calif. 91125). *Biochem.* 13, 4942-8 (1974). The interaction of alamethicin with both unsonicated lecithin multilayers and sonicated bilayer vesicles has been investigated by nuclear magnetic resonance (nmr) spectroscopy and electron microscopy. It is shown that alamethicin is a surface active agent, which interacts primarily with the polar choline head groups of the lecithins. Alamethicin also induces aggregation and subsequent fusion of small bilayer vesicles ~300Å in diameter, a process which was found to have a profound influence on the nmr spectral properties of these bilayers.

ELUCIDATION OF THE SOLUTION CONFORMATION OF THE A RING IN VITAMIN D USING PROTON COUPLING CONSTANTS AND A SHIFT REAGENT. G.N. LaMar and D.L. Budd (Dept. of Chem., Univ. of Calif., Davis, Calif. 95616). *J. Am. Chem. Soc.* 96, 7317-24 (1974). The proton spin-spin splittings of vitamin D₂, calciferol, have been analyzed in terms of the solution conformation of the A ring. Using the known coupling constants for cyclohexanol, the observed multiplets are shown to be consistent with a dynamic equilibrium between approximately equal amounts of the α and β chair forms for the A ring. Resonances on the A ring not observable in the pure compound were resolved with the aid of the shift reagent Eu(dpm)₃. Analysis of the Eu(dpm)₃ induced dipolar shifts confirms the presence of the 1:1 mixture of conformers. The insensitivity of the proton multiplet structure to the presence of Eu(dpm)₃ suggests that the equilibrium is not perturbed significantly upon coordination. Conformational analysis as a function of temperature, using the induced dipolar shifts, indicates that the β chair is thermodynamically slightly more stable. The merits and problems of quantitative vs. qualitative use of shift reagents in solution conformational analysis of large flexible molecules are discussed.

1 α -HYDROXYVITAMIN D₂: A POTENT SYNTHETIC ANALOG OF VITAMIN D₂. H.-Y.P. Lam, H.K. Schnoes, H.F. DeLuca (Dept. of Biochem., College Agr. and Life Sci., Univ. of Wisconsin, Madison 53706). *Science* 186, 1038-40 (1974). A hydroxy analog of vitamin D₂, 1 α -hydroxyvitamin D₂, has been

synthesized and tested for biological activity. This vitamin derivative is active in stimulating intestinal calcium transport and bone calcium mobilization in the rat and exhibits anti-architic activity. Its biopotency is comparable to that of the corresponding vitamin D₃ analog, 1 α -hydroxyvitamin D₃.

THE REACTION OF PHOSPHOGLYCEROLIPIDS AND OTHER LIPIDS WITH HYDROFLUORIC ACID. N. Shaw and A. Stead (Microbiol. Chem. Res. Lab., Univ. of Newcastle upon Tyne, Newcastle upon Tyne NE1 7RU, U.K.). *Biochem. J.* 143, 461-4 (1974). The use of HF as a dephosphorylating reagent for phospholipids was examined. Hydrolysis of phosphatidylethanolamine at 0°C for 24 hr. with 60% HF gives a good yield of diglyceride. Under similar conditions phosphatidylglucosyl diglyceride gives diglyceride and diglucosyl diglyceride. The glycolipid is also obtained from hydrolysis of glycerylphosphoryldiglyceride. No lyso derivative of the glycolipid could be detected and the glycosidic linkage was also stable. Triglycerides, unsaturated and cyclopropane fatty acids were unaffected by the reagent. 1,2-Diglycerides and 1,3-diglycerides were partially isomerized and also gave small amounts of free fatty acid and monoglyceride. Monoglycerides underwent extensive rearrangement to form 1,2- and 1,3-diglycerides. Lyso-phosphatidylethanolamine also gave 1,2- and 1,3-diglycerides as well as monoglycerides. The application of this procedure to the structure determination of various phosphoglycerolipids is discussed.

VITAMIN D IN SOLUTION: CONFORMATIONS OF VITAMIN D₃, 1 α ,25-DIHYDROXYVITAMIN D₃, AND DIHYDROTACHYSTEROLS. R.M. Wing, W.H. Okamura, M.E. Pirio, S.M. Sine and A.W. Norman (Depts. of Chem. and Biochem., Univ. of Calif., Riverside 92509). *Science* 186, 939-41 (1974). Solution conformations of the A and seco B rings of vitamin D₃, 1 α ,25-dihydroxyvitamin D₃, 1 α -hydroxyvitamin D₃, and dihydrotachysterols have been established by high resolution, 300-megahertz proton magnetic resonance spectroscopy. The A ring of these steroids is dynamically equilibrated between two chair conformers. For vitamin D₃, 1 α -hydroxyvitamin D₃, and 1 α ,25-hydroxyvitamin D₃, the relative proportions of the two conformers are 1:1, whereas dihydrotachysterols exists principally as only one conformer. Thus, the substituent groups on the A ring may be either equatorially or axially oriented, and suggests a refinement of the existing topological model for vitamin D hormonal activity.

PROCESS FOR IMPROVING THE THERMAL STABILITY OF SOYBEAN OIL. W.P. Gibble and E.J. Reid (Hunt-Wesson Foods, Inc.). *U.S.* 3,857,866. A method for providing an edible soybean oil which does not develop objectionable odor or flavor for at least 10 minutes when heated to a frying temperature of 325-400F consists of the following steps: (1) deaerating a refined soybean oil to an oxygen content of less than 0.1 ml per 100 ml oil; (2) dissolving carbon dioxide in the deaerated oil; (3) dispersing a minor amount of a treating agent consisting of a finely divided, activated, solid, inorganic metallic salt or oxide adsorbent capable of removing color bodies in the oil; (4) heating the dispersion in a carbon dioxide atmosphere above 212F for 15 minutes to 10 hours; (5) filtering the oil; and (6) vacuum steam deodorizing the oil.

POURABLE LIQUID SHORTENING. E. Reid and P.W. Morgan, Jr. (Hunt-Wesson Foods, Inc.). *U.S.* 3,857,985. The shortening is prepared by the following steps: (1) a minor amount of finely divided, substantially fully saturated, normally solid glycerides, at least 90% of which are convertible to the beta crystal form, is dispersed in a liquid vegetable oil; (2) the mixture is heated to above the melting point of the solid glycerides; (3) the batch is then incrementally cooled as follows: (a) a small amount is continuously removed from the main body of hot oil and cooled to below the crystallization point of the solid triglycerides to form a suspension; (b) the suspension is added back to the main body of oil, thereby melting the crystals and cooling the main body; (4) step (3) is repeated until the crystals do not completely melt and the body of oil is cooled to below the temperature of phase change of the solid glycerides from alpha and beta prime to beta crystals; (5) the suspension is held at the phase change temperature to convert all of the solid glycerides to beta crystals in a stable suspension; and (6) the suspension is deaerated.

PROCESS FOR PREPARING HARD BUTTER. T. Tateishi, K. Marase and Y. Iwanaga (Fuji Oil Co.). *U.S.* 3,856,831. A process for preparing a nontempering hard butter comprises hardening an oil with an I.V. of 60-78 and a total linoleic and linolenic acid content of less than 17% with a nickel catalyst containing 6-21 parts of sulfur per 100 parts of nickel until the absorption of hydrogen has substantially ceased.

INTERESTERIFICATION CATALYST. J.J. Muller and T.J. Koch (Lever Bros. Co.). *U.S.* 3,856,703. The catalyst consists of particles of an alkali metal, at least 90% of which have a size of 0.3-1 mm, dispersed in a solid fat of 25-45 C melting point and having no more than 0.05% water. The proportion of alkali metal in the fat is 5-60%.

NICKEL/COPPER CHROMITE CATALYSTS FOR HYDROGENATING EDIBLE OILS. K.J. Moulton and R.E. Beal (U.S. Secy. of Agriculture). *U.S.* 3,856,710. The composition, for the selective partial hydrogenation of vegetable oils, consists of a mixture of a nickel catalyst, comprising nickel metal and a hardened vegetable oil, and a copper chromite catalyst in proportions such that the weight ratio of nickel metal to copper chromite is 0.125:1000 to 0.50:1000.

ADSORBENT FOR PURIFYING VEGETABLE OILS FROM PHOSPHORUS CONTAINING COMPOUNDS. D.V. Sokolsky, A. Zhubanov, N.F. Shumateva, E.P. Mazin and A.M. Sokolskaya. *U.S.* 3,862,054. The adsorbent for removing phosphorus containing compounds and free fatty acids from vegetable oils is prepared by mixing a 10-20% aqueous solution of a metal salt selected from the group consisting of sulfates, nitrates, and chlorides of metals in Groups II and III of the Periodic Table with a 10-20% aqueous solution of an alkali; depositing the resulting metal carbonates and hydroxides onto a filler consisting of alumina or clay free from iron oxides; separating the filler from the solution to give a paste-like mass; adding a heat resistant cement binder; granulating the mixture; drying the granules; and calcining them at 300-500 C.

TREATMENT OF TALL OIL FATTY ACIDS. B.F. Ward (Westvaco Corp.). *U.S.* 3,860,569. A method of treating the linoleic acid portion of tall oil fatty acids comprises converting the linoleic acid to oleic acid by heating at a temperature of 400-550F in the presence of a bromine-iodine catalyst (1:1-5:1 bromine to iodine) for 10 minutes to 6 hours. The amount of catalyst is 0.06-2.0% of the fatty acids.

STABLE LIQUID EMULSIFIER COMPOSITIONS IN BREAD MAKING. R.K. Langhans (ICI United States Inc.). *U.S.* 3,859,445. The compositions comprise 60-99.3% of a mixture of 10-80% of a fatty acid monoglyceride and a polyoxyethylene-containing compound. The fatty acid moieties of the monoglyceride contain 12-22 carbon atoms, at least 86% of which are unsaturated and 40% of which are substituted in the alpha position. The compositions also contain a clarifier selected from the group consisting of 3-40% propylene glycol, 0.7-40% ethanol, 5-21% water, and 3-25% edible oil which is liquid at 35F. The emulsifier is used in doughs containing not more than 5% shortening and 8% sugar at levels of 0.15-3%.

MARGARINE. B. Sreenivasan (Lever Bros. Co.). *U.S.* 3,859,447. A plastic margarine comprises 80-95% oleaginous phase and 20-5% aqueous phase. The oleaginous phase comprises a non-hydrogenated, directed interesterified triglyceride oil having a linoleic acid content of 50-79%, a linolenic acid content of less than 10%, and a solid fat index of 3-25 at 0C and 2-12 at 21.1C; and minor additives including stabilizers, emulsifiers, preservatives, flavoring substances, vitamins and colorants.

REFINING OF FATTY ACIDS. A. Zvejnieks (AZS Corp.). *U.S.* 3,859,270. A method of refining impure fatty acids containing 6-22 carbon atoms consists of decreasing the volatility of the fatty acids by esterifying them with hydroxyl containing organic compounds, distilling out the unesterified compounds under vacuum, and recovering purified fatty acids from the residue.

HINDERED TRIS-(META-HYDROXYBENZYL) CYANURATE ANTI-OXIDANTS. P.V. Susi (American Cyanamid Co.). *U.S.* 3,862,053. A composition comprising an organic material which tends to deteriorate upon exposure to oxygen contains an antioxidant in an amount sufficient to inhibit oxidative deterioration.

PREPARATION OF UNSATURATED CARBOXYLIC ACIDS. L. Heslinga, H.J.J. Pabon and D.A. van Dorp (Lever Bros Co.). *U.S.* 3,862,972. A process for preparing an acid of the structure

$$RCH_2(CH=CHCH_2)_m(CH_2)_nCH=CHCOOH$$

of 5-22 carbon atoms in the chain, where R is a monovalent aliphatic radical and m is 0 or 1-5, n being 0 when m is 0, comprises contacting a Grignard reagent with an ω -halogeno- α -enoic acid of the structure $BrCH_2(C\equiv CCH_2)_m(CH_2)_nCH=CHCOOH$ in an inert liquid whose temperature is restricted to below -20C if in the reagents selected the α carbon of the group R are all single bonds and m and n are 0.

ADSORBENT FOR OILS. H. Hoshi, K. Murakami, I. Maeda and R. Susuki (Lion Fat & Oil Co.). *U.S. 3,862,963*. An adsorbent for oils comprises a foamed substance having a bulk density of less than 1 and consisting of 5-90% of at least one synthetic resin insoluble in water and oil and selected from the group consisting of polyethylene, polypropylene, and ethylene-vinyl acetate copolymer. The adsorbent also contains 10-95% of an inorganic filler selected from the group consisting of calcium sulfite, gypsum, calcium carbonate, silica, and talc.

COCOA BUTTER COMPOSITION. C.S. Castner (Schuyler Development Corp.). *U.S. 3,862,197*. A method for recovering cocoa butter fatty acids comprises the following steps: (a) mixing cocoa butter in water at elevated temperature with an alkali metal hydroxide to provide a pH of 10.5-11.5, (b) mixing until partial saponification at the desired pH has been complete, (c) adding hydrochloric acid to bring the pH to 4.5-6.0, (d) washing the unreacted cocoa butter and reaction product to remove insoluble and soluble salts, and (e) collecting the washed partially hydrolyzed cocoa butter product.

DETERMINATION OF TRIGLYCERIDES. A.W. Wahlefeld, H. Molering, W. Gruber, E. Bernt and P. Roeschlaue (Boehinger Mannheim GmbH). *U.S. 3,862,009*. A method for determining triglycerides by enzymatic saponification comprises saponifying a sample containing triglycerides with a lipase from *Rhizopus arrhizus*, carboxylesterase, and an alkali metal or alkaline earth metal alkyl sulfate with alkyl radicals of 10-15 carbon atoms. The liberated glycerol is measured.

LIPID COMPOSITION OF A FROZEN EGG SUBSTITUTE. D.E. Pratt (Dept. Foods and Nutrition, Purdue Univ.). *J. Am. Dietetic Assoc.* 66, 31-3 (1975). The lipid composition of an egg substitute was compared to that of fresh whole egg. In the substitute, the lipids were derived from corn oil, vegetable lecithin, mono- and diglycerides, and propylene glycol mono-stearate. Following extraction, the total lipid was divided into four fractions (cholesterol esters; triglycerides; free cholesterol, mono- and diglycerides, and free fatty acids; and phospholipids) on a silicic acid column. Each fraction was further separated by TLC, and fatty acids were identified by GLC. The egg substitute was practically devoid of cholesterol and cholesteryl esters. Whole eggs contained considerably higher concentrations of saturated and monounsaturated fatty acids while the polyunsaturated fatty acid content of the substitute was approximately three times that of the eggs. The findings agree reasonably well with those of the manufacturer.

PROCESS OF METHYLATING CEPHALINS. H. Betzing and D. Lekim (Nattermann, A. & Cie GmbH). *U.S. 3,862,968*. A process for methylating a phosphatide of phosphatidylethanolamine or phosphatidylserine comprises mixing the phosphatide with formaldehyde in the presence of formic acid and heating the solution to 100°C or less. At least 2 moles of formaldehyde and 2 moles of formic acid are employed per amino group.

TOXICITY OF PESTICIDE RESIDUE IN FATS AND OILS. G. Viel (Lab. Phytopharmacie -INRA, 78 Versailles). *Rev. Franc. Corps Gras* 21, 155-60 (1974). As the toxicity of fats from pesticide residue is not a special problem, the general aspects of pesticide toxicity are discussed. Some particular aspects regarding pesticide residue in dairy products are mentioned. Physical, chemical, and technological properties are described, then the action on biochemical mechanisms. Toxicological studies involve the study of sharp, short, and medium terms and long term toxicity. Observations on human subjects are recorded. The permissible daily intake is defined.

PESTICIDES USED FOR OLEAGINOUS CULTURE. DETERMINATION OF PESTICIDES OR THEIR METABOLITES IN FATS AND OILS. R. Mestres (Faculté de Pharmacie, Lab. Chim. Montpellier). *Rev. Franc. Corps Gras* 21, 145-53 (1974). The nature of pesticides organochlorinated or organophosphorylated, which are used for oleaginous cultivation, is first indicated in this paper and then the methods used for separating pesticides by total or selective extraction and then by volatilization are described. The elimination of fats from the extract is elaborated in one chapter. The purification may be done through elution chromatography on adsorbant, often after a separation by means of double partition or saponification.

EVOLUTION OF PESTICIDES DURING INDUSTRIAL PROCESSING AND REFINING OF FATS AND OILS. J.P. Wolff (Lab. Wolff, Paris). *Rev. Franc. Corps Gras* 21, 161-4 (1974). The determination of organochlorinated insecticides in the oils at different stages of their production showed that those occurring in oil seeds or in crude oils are totally eliminated during refining. This conclusion is confirmed by laboratory tests on crude oil

voluntarily enriched with organochlorinated insecticides. Meal, theoretically, doesn't contain any insecticide at the end of processing. It can be said that part of contamination inherent to oils and meals in human and animal nutrition is extremely low in regard to organochlorinated insecticides.

SOME ECONOMICAL CONSIDERATIONS ABOUT MARGARINE INDUSTRY IN FRANCE. F. Lopez (Chambre synd. Margarinerie, Paris). *Rev. Franc. Corps Gras* 21, 175-8 (1974). The economical aspects regarding margarine production, consumption, and price are given. Statistical data are used. French margarine production represents about 3% of world production. The production has regularly increased from 1960 to 1971, from 104,000t to 165,000t respectively. Production of margarine for industrial use is increasing faster than margarine production for direct consumption. Per capita consumption of margarine in France is about 3kg; total visible fat consumption is about 25kg. It can be expected that margarine use will increase in the future, especially regarding special kinds of margarine, like margarine with high content of polyunsaturated fatty acids, low calorie margarine, etc.

SOLVENT EMPLOYED IN OIL AND FAT INDUSTRY. INCIDENCE ON SECURITY AND HYGIENE. A. Kreyenbuhl (Caisse Nation. Assurance Maladie, Paris). *Rev. Franc. Corps Gras* 21, 211-4 (1974). Whatever the envisaged or applied safe methods are, the use of benzene for the extraction of vegetable or animal fats presents a potentially serious risk for those working in such an industry. The "Service Prevention des Accidents du Travail" has envisaged to resolve this problem of security by the replacement of benzene by a non-inflammable solvent. The theoretical study on physico-chemical and toxicological properties of such a solvent showed that this replacement seems difficult now notwithstanding the great choice of non-inflammable solvents available in the chemical industry. Besides trichlorethylene, only the 1, 1, 2-trichloro-1, 2, 2-trifluoroethane (R 113) seems to have properties analogous to those of benzene universally employed.

SOLVENT IN OIL INDUSTRY. J.M. Klein (Lesieur-Cotelle et Ass., Coudekerque). *Rev. Franc. Corps Gras* 21, 215-8 (1974). Among the solvents which may be employed in oil industry, hexane is the most used. The author discusses the reasons for this choice by the oil processors. The French regulation related to this use is reviewed and the trace solvent authorized in the oils produced is pointed out. In fact, the residual hexane content in the oils sold in France is under 0.1 mg/kg. Studies of different authors show that this hexane content is not dangerous for the consumer. Theoretically, if 20g of oils is consumed daily, the hexane quantity will be 2×10^{-8} mg of hexane per day. This quantity, as shown by the results obtained with experiments done on animals, is not toxic.

REGULATION GOVERNING FATS AND OILS IN FRANCE. J. Castang and R. Souverain (Service Repression des Fraudes et du controle de la qualite, Paris). *Rev. Franc. Corps Gras* 21, 220-31 (1974). After a short historical review of the French regulation, the authors discuss the present rules governing definitions, production and trade of fats and oils. Some specifications for margarine, dietetic products and animal fats are pointed out. After edible fats, industrial fats and their derivatives, such as soaps and oil-meals, are studied. International regulations, EEC's and FAO/HWO's Codex Alimentarius, are described. In conclusion, the authors stress the evolution of the regulation regarding trade, new products and technology, marketing, and the consumer's need for better information.

NEW PROGRESS IN INDUSTRIAL HYDROGENATION AND TRANS-ESTERIFICATION OF EDIBLE FATS. J. Baltes (Deutsche Gesellschaft für Fettwissenschaft, Münster). *Rev. Franc. Corps Gras* 21, 281-93 (1974). The author, laureate of Medaille Chevreul of 1973, describes the progress of two important technological processes: hydrogenation and interesterification. The given results are obtained by the author himself. The batch and continuous industrial plants, the reaction mechanism and kinetics, and the catalysts in heterogeneous and homogeneous phases are reviewed. The author points out that the most scientific methods are applied in hydrogenation and interesterification field and this makes it possible to govern these processes.

WASTEWATERS. B.—INDUSTRIAL SOLUTION FOR WASTEWATER EPURATION AND RECYCLING. G. Choffel (Societe Lesieur-Cotelle et Ass. Boulogne sur Seine). *Rev. Franc. Corps Gras* 21, 301-7 (1974). In the paper, some treatments used today for epuration and recycling of wastewater in oil industries, especially in oil factory Lesieur-Cotelle, are described. The greatest amount of pollution takes place in the refining pro-

cesses. The pollution from oil industry must be regarded as a part of a great problem of environmental pollution and all efforts must be made to reduce this pollution. Some realizations for the treatment of wastewater in the factories of the Lesieur-Cotelle are presented. Some other processes which are in use are also briefly mentioned. The investments and costs of exploitation are mentioned.

SOLID CONTENT DETERMINATION OF FATS AND MARGARINES. E. Sambuc and M. Naudet (Lab. Nationale Matieres Grasses (ITERG), Univ. Provence, Marseille). *Rev. Franc. Corps Gras* 21, 309-12 (1974). The solid and liquid equilibrium determination in fats and in margarines is possible only by wide line NMR spectrometry. The sample must be taken with a sampler and transferred in the analytical tube previously tempered at the sample temperature. For margarines, the water present prohibits the solid content determination in the fatty phase. However, a "solid index" (immobilized protons to total protons ratio) may be calculated. The liquid point determination requires some particular precautions. The studies of the influence of some factors on the solid content and its direct influence on the rheological properties and physical characteristics of fats are also mentioned.

FRACTIONATION AND INTERESTERIFICATION OF FATS IN THE PERSPECTIVE OF THE WORLD MARKET OF RAW MATERIALS AND FINISHED PRODUCTS. I. FRACTIONATION. J.W.E. Coenen (Unilever Research Lab., Vlaardingen). *Rev. Franc. Corps Gras* 21, 343-49 (1974). The object of fat and oil modification: fractionation, interesterification and hydrogenation are pointed out in the introduction of this paper. The principles of the fractionation process are then given. Three procedures of fractionation are described and they are: simple, Lanza (wet method with a wetting surface-active agent), and in solvent phase (hexane + acetone). Data for the products obtained by the application of these techniques are given. Some applications of the products are also given.

SPREADABLE MARGARINE. TECHNOLOGY. G. Bouffard (Raffinerie Maurice Toy-Riont, Marseille). *Rev. Franc. Corps Gras* 21, 351-7 (1974). First, the modern knowledge about spreadable margarines and then the rheological properties (penetrometry and extrusion) are given in the paper. The formulation of the fatty phase (ratio—oil:solid fat), the properties of the aqueous phase, and the phenomena of the differential crystallization are discussed. The three fundamental technological operations are reviewed: emulsification, cooling/crystallization, mechanical work and particularly Unit A and B and the recycling disposal.

ORGANOCHLORINATED PESTICIDES IN FATS AND MEALS FROM DIFFERENT OILSEEDS. EVOLUTION DURING TECHNOLOGICAL TREATMENTS. J.C. Florence et al. (Societe Lesieur-Cotelle et Ass. Coudekerque-Branche). *Rev. Franc. Corps Gras* 21, 359-62 (1974). Organochlorinated pesticides were determined in raw and refined oils and in pressed and extracted meals of different origins: peanut, rapeseed, corn, soya, and sunflower. The technique employed is gas chromatography with electron capture, after extraction, concentration, and purification. No detectable traces of insecticide were found in meals. However, they may be contaminated during storage, transport, etc. Insecticides are easily eliminated from refined oil, especially during deodorization.

STUDY OF OXIDIZED FATTY ACIDS OCCURRING IN FATS AND OILS. V.—COMPARATIVE STUDY OF OXIDIZED FATTY ACIDS IN GLYCERIDES, IN FREE FATTY ACIDS, AND IN PHOSPHOLIPIDS. J. Graille and M. Nudet (Lab. National Matieres Grasses (ITERG), Univ. de Provence, Marseille). *Rev. Franc. Corps Gras* 21, 363-9 (1974). Oxidized acids occurring in crude oils are unequally distributed in glycerides, free fatty acids, and phospholipids. Their concentration is higher in non-glycerides than in glycerides, but because of the preponderance of glycerides, the main part of the oxidized acids is in them. The composition of oxidized acids in the glycerides, in the free fatty acids, and in phosphatides indicated that they have the same components, but that they are distributed in a different way in glycerides than in non-glycerides. Studies have been done on crude peanut oil obtained by pressing and extraction. The free fatty acid content of the oil was 3.1% and the oxidized acid content 2.3%. The oxidized acids were examined by thin layer chromatography.

FRACTIONATION AND INTERESTERIFICATION OF FATS IN THE PERSPECTIVE OF THE WORLD MARKET FOR RAW MATERIALS AND FINISHED PRODUCTS. II. INTERESTERIFICATION. J.W.E. Coenen (Unilever Research Laboratories, Vlaardingen). *Rev. Franc. Corps Gras* 21, 403-13 (1974). Two techniques of interesterification: directed and nondirected, are described. The kinetics and reaction mechanism according to the catalysts

and the processing are reviewed. Then the practical point of view is discussed: selection of catalyst, apparatus, batch or continuous process. Some examples are given concerning cocoa butter, palm oil, lard, and fat stock for margarines. The prospect for this modification is full of promise. This process may become more important than hydrogenation in the future.

ENZYMATIC LIPOLYSIS OF COCONUT IN LOW MOISTURE MEDIUM. G. Paulet et al. (Inst. National Sciences Appliquees, 20, av. Albert-Einstein, 69621 Villeurbanne). *Rev. Franc. Corps Gras* 21, 415-22 (1974). A kinetic study of coconut hydrolysis, in low moisture medium, by lipase of white pepper has been made. The main parameters of the reaction: time, weight of pepper and coconut, temperature and hydration level were examined with a reactive model using an inert support (white of an egg). The enzyme is active in a broad zone of temperatures. The releasing of fatty acids is nearly linear with the time and proportional to the quantity of spices. The enzymatic activity was detected for a low water content, contrary to other hydrolases. A very light inhibition of the lipolysis appears after some hydration.

RESEARCH ON SELECTIVE HYDROGENATION BY HOMOGENEOUS CATALYSIS. III. HYDROGENATION OF SOYA METHYL ESTER BY IRON PENTACARBONYL. G. Cecchi and E. Ucciani (Lab. National Matieres Grasses (ITERG), Univ. Provence, 13331 Marseille). *Rev. Franc. Corps Gras* 21, 423-8 (1974). The partial hydrogenation of soya methyl esters by iron pentacarbonyl is studied by the "answer surface method." The analysis of data, furnished by the computer, points out the fact that the variables are not independent. The catalyst is active and selective, but isomerization is extensive. Hydrogenation occurs by the intermediary of a diene-Fe(CO)₅ complex which breaks down to monoenes, conjugated dienes, and degradation products. The latter ones consist of saturated and ethylenic esters with 10, 11, and 12 carbon atoms and they represent 1-5% of the hydrogenation products.

STUDY OF OXIDIZED ACIDS OCCURRING IN FATS AND OILS. VI. STUDY OF EVOLUTION OF OXIDIZED ACIDS DURING THE REFINING. J. Graille and M. Naudet (Lab. Nat. Matieres Grasses—I.T.E.R.G., Univ. Provence, Marseille). *Rev. Franc. Corps Gras* 21, 475-87 (1974). The oxidized fatty acids in crude oil are unequally distributed in the glyceride fraction and non-glyceride (free fatty acids, phosphatides) fraction. Chromatoplates and pondered histograms for oxidized acids from different samples of raw and refined oils and oils taken during industrial refining are compared. It was found that the refining process, as well as phases of the refining process, influence the oxidized acids in three different ways: no modification, more or less important decrease, increase. From the balance of laboratory refining, it can be seen that during each refining phase, the oxidized acids of treated oil are only divided between treated oil and by-products, or disappear partially, or increase.

OBTAINING OF AROMAGRAMS OF FATS. F. Mordret (Institut Corps Gras, Paris). *Rev. Franc. Corps Gras* 21, 489-93 (1974). An original method for separating and analyzing the volatile components which are responsible for the flavor of fats is described. The separational process is called "Aromagrams process": the volatile matter is carried away by a gas flow, trapped out on open circuit, then volatilized straight into a gas-liquid chromatograph and then analyzed. For that an apparatus called "flavor extractor" was used. The best conditions for its utilization are given in the paper with some practical examples dealing with peanut oils freshly refined and of different grades of rancidity.

SAMPLING AND REPRESENTATIVITY OF SAMPLES: THEORY AND PRACTICE FOR VEGETABLE RAW MATERIALS. P. Gy and J.A. Casalis (Service Traitement Minerais, S.M.M. Penarroya, Paris). *Rev. Franc. Corps Gras* 21, 495-500 (1974). The importance of correct sampling of the raw materials, particularly of vegetable origin, is discussed. A bad sampling is a cause of additional error. The authors recall the definitions of these terms and the features of a sampling. They also describe a mathematical model and its errors.

COMPARED OXIDATION OF MARGARINE AND THEIR FATTY BASE. M. Naudet and S. Biasini (Lab. Nat. Matieres Grasses ITERG, Univ. Provence, Marseille). *Rev. Franc. Corps Gras* 21, 501-2 (1974). To examine whether the presence of water, emulsified in the fatty phase, slows down oxidation of margarine, the authors prepared, in laboratory, different samples of margarines and their fatty phases. For these experiments, refined peanut, copra, tallow and hydrogenated fish oils were used.

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