ABSTRACTS

R. A. REINERS, Editor. ABSTRACTORS: J. G. Endres, Kazuo Fukuzumi, J. Iavicoli, K. Kitsuta, F. A. Kummerow, C. C. Litchfield, Gladys Macy, Louise R. Morrow, E. G. Perkins, and T. H. Smouse

• Fats and Oils

Foaming tendancies of frying oils. IV. Physical properties of foaming oil. Shizuvuki Ota (Ajinomoto Co., Tokyo). *Yukagaku* 13, 269–74 (1964). Surface tension had no relation with the foaming of oil but surface viscosity and viscosity seemed to be related to it.

Loss of oil during the deep fat frying. Shizuyuki Ota and Yasuko Izuyama (Ajinomoto Co., Tokyo). *Yukagaku* 13, 328–34 (1964). Quantitative determination of loss of oil during deep fat frying of soybean curd (Tofu) has been investigated. The possibilities for the loss of oil during the deep fat frying are the formation of volatile product of fat degradation and adsorption of oil in fried foods. The chief factors governing the amount of loss of oil during the deep fat frying were the time of cooking, the temperature of frying and the composition of the food. In the frying of 15 cakes of Tofu in 1 kg oil, they were fried for a minute one after another for 30 seconds each on both sides. The loss of oil and the weight of fried Tofu were measured and the vaporized water was calculated from these data. The fried Tofus were wiped with dried filter paper and the amount of oil adhered on the food surface was measured. These frying tests did not reveal any significant differences in oil absorptions among soybean oil, cottonseed oil and rapeseed oil. In the cooking for 1 minute at 240, 220 and 200C, there were significant differences corresponding to the temperature of oil. The longer the time of frying, the more was the loss of oil. Using of deteriorated oil or foaming oil, there was significant loss of oil. The cause of loss of oil was mostly the adherence of oil on the surface of food to be fried, although the amount of oil absorbed was rather small.

Isolation of β-sitosteryl ferulate from rice bran oil. Akio Tanaka, Akio Kato and Tomotaro Tsuchiya (Gov. Chem. Ind. Research Inst., Tokyo). *Yukagaku* 13, 260–3 (1964). Rice bran oil (25 kg) (with acid no. 14.5, saponification no. 187, iodine no. 106.5 and unsaponifiable matter 6.32%) was extracted with 25% hexane at −20C, chromatographed through Al_2O_3 and the ether effluent concentrated to give 32 g β-sitosteryl ferulate, $C_{39}H_{58}O_4$, mp 131–1.5C, $[\alpha]_{559}$ 10.9°, λ_{max} 215, 231, 291 and 315 mμ. The ferulate gave an acetate, $C_{41}H_{60}O_5$, mp 165–6°, $[\alpha]_{589}$ 3.7°, λ_{max} 225, 227 and 307.5 mμ. Hydrolysis of the ferulate gave β-sitosterol and ferulic acid, $C_{10}H_{10}O_4$, mp 169.5–70C.

Hydrocarbons in the deodorizer scum of soybean oil. Tomishi Yamada (Kanagawa Univ., Yokohama). *Yukagaku* 13, 321–4 (1964). A hydrocarbon with mp 68–8.5C and molecular weight 435 was obtained from the crude sterols of soybean oil deodorizer scum by extraction with *n*-hexane and column chromatography. Infrared spectrum, gas chromatography and NMR spectrum showed that this hydrocarbon was composed of n-$C_{32}H_{66}$, n-$C_{30}H_{62}$ and a small amount of n-$C_{28}H_{58}$ and n-$C_{31}H_{64}$. Thus extraction with paraffinic hydrocarbon solvent is necessary when soysterol of deodorizer scum is used for the synthesis of steroidal hormones.

Studies on soysterol. V. Chromic anhydride oxidation of sitosterols and stigmasterol. Tomishi Yamada, Saburo Gomi, Yoshimasa Noma and Teruzo Asahara. *Yukagaku* 13, 382–4 (1964). The reactivities of sitosteryl acetate dibromide and of stigmasteryl acetate tetrabromide with chromic anhydride were compared. Stigmasteryl acetate tetrabromide was oxidized but chromic anhydride consumption of the tetrabromide was less than that of sitosteryl acetate dibromide. Diatomaceous earth caused the lowering of adsorption of chromic anhydride and decreased its concentration in the reaction mixture.

General tendencies of researches on fatty oil industry. Saburo Komori (Osaka Univ.). *Yukagaku* 13, 335–41 (1964). A review with 74 references.

Studies on the Japan wax by gas-liquid chromatography using a hydrogen flame ionization detector. Yoshio Sano, Dainosuke Aikawa and Kimiko Murase (Taiyo-Miyoshi Research Inst. of Whale Oils, Tokyo). *Yukagaku* 13, 324–8 (1964). The contents of lower dibasic acids in Japan wax have been investigated. Gas chromatographic analyses were performed by use of a flame ionization detector. The result indicated the presence of dibasic acids containing 5, 6, 7, 8, 9, 10, 11, 12, 14, 16, 18, 20 and 22 carbon atoms, various fatty acid containing 10, 14, 15, 16, 17, 18, 20 and 22 carbon atoms and considerable amount of unknown substances were found in the free acids responsible for the high acid value (21.5) of the wax. In addition, methyl esters, obtained by direct methanolysis of the wax and subsequent removal of the free acids, gave a small amount of dibasic acids containing 14, 16, 18, 20 and 22 carbon atoms. Thin layer chromatography on silica gel was also applied to the analysis of the wax and it was found that the dibasic acids were separable from fatty acids and others and each component was examined in detail.

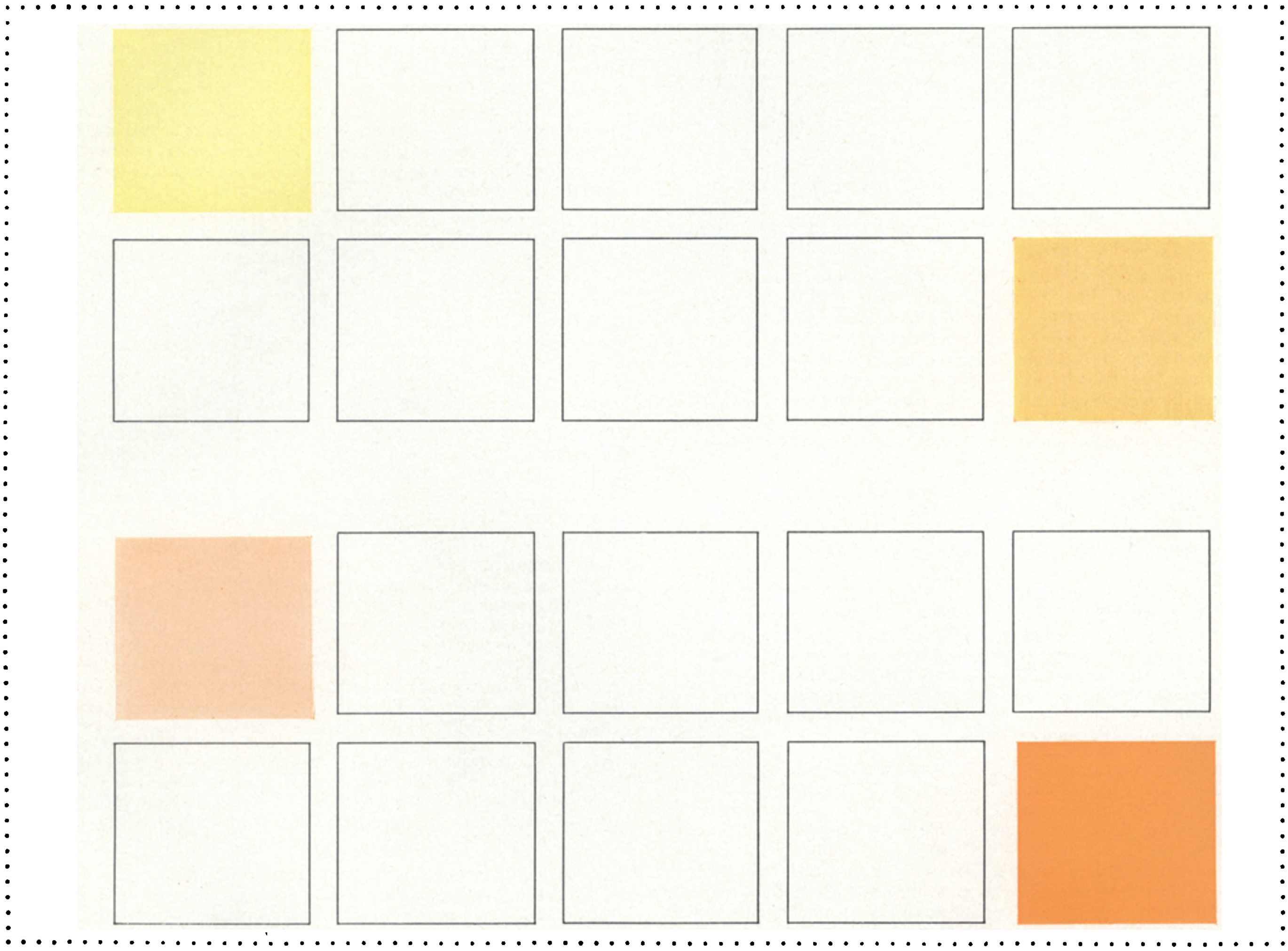
The methods of determination of oxirane oxygen. Yasuo Suhara (Gov. Chem. Ind. Research Inst., Tokyo). *Yukagaku* 13, 378–81 (1964). Oxirane oxygen of non-terminal epoxy compounds was determined by each method, hydrogen chloride-ether, dioxane- and acetic acid and hydrogen bromide-acetic acid solution. Water in the sample caused a decrease of accuracy of determination. Hydrogen bromide-acetic acid solution method was more accurate in the ether method. Anhydrous hydrogen bromide-acetic acid solution was prepared easily by means of a procedure of dissolving hydrobromic acid in acetic acid and then adding an equivalent amount of acetic anhydride to water. For increasing the accuracy of determination, hydrogen bromide-acetic acid solution was modified as follows: In case of standardization of hydrogen bromide-acetic acid solution, sodium carbonate was dissolved with 10 cc of acetic acid and then 10 ml of benzene was added into the solution. The sample was dissolved with a mixture of 10 cc benzene and 5 cc acetic acid.

in-situ Epoxidation of oleic acid, butyl oleate and butyl ester of tall oil fatty acid. Yasuo Suhara (Gov. Chem. Ind. Research Inst., Tokyo). *Yukagaku* 13, 279–80 (1964). Epoxide was obtained by the following procedure in 99% yield. A 95% hydrogen peroxide (7.2 g or 0.2 mole) was added dropwise in 1 hour to a mixture of 36.4 g (0.1 mole) butyl oleate, 17.6 g benzene, 6 g acetic acid and 1.9 g of 50% sulfuric acid at 50–60C. Immediately after addition of all of the hydrogen peroxide, the reaction mixture was poured in water and the epoxide was extracted with petroleum ether. The product was colorless and its purity was 92.7%. The purity of product was better than that of the product obtained by use of peroxyacetic acid. Epoxides of oleic acid and butyl ester of tall oil fatty acid were also obtained by the above procedure. The latter had a purity of 90.8% while that of former was only 60.4%.

Separation of unsaturated fatty acids. Toru Takagi (Nagoya Univ.). *Yukagaku* 13, 247–54 (1964). A review with 116 references.

Studies on lower melting hydrogenated oils and fats. I. Effect of the addition of dimethyl polysiloxane on hydrogenation process. Umajiro Shimamura, Kichiji Suzuki, Sadamitsu Maekawa and Shinji Mitsunaga (Nippon Oils & Fats Co., Tokyo). *Yukagaku* 13, 255–9 (1964). Hydrogenation of finback whale oil, cod-liver oil and saury oil were carried out at 200C with 0.2% nickel catalyst and 0.015% dimethyl polysiloxane (MPS). An addition of MPS to the nickel catalyst was effective in slowing down the rate of hydrogenation. Hydrogenated oils with nickel catalyst and MPS showed much better consistency at low temperature, solid fat index and in the softening point than those without MPS. The MPS having viscosity 500 c.s. was superior than that having 100 c.s.

Catalytic action of transition metal carbonyl. II. Separation of a π-diene-iron tricarbonyl complex produced from methyl linolate and iron pentacarbonyl, and the structure and properties. Ikuei Ogata and Akira Misono (Univ. Tokyo). *Yukagaku* 13, 308–13 (1964). In order to confirm the complex formation and further investigation on the catalytic behavior of π-diene-iron tricarbonyl complex a new methyl π-C_{18}-conjugated-diene-carboxylate-iron tricarbonyl was isolated from the reaction product of fatty acid methyl ester of dehydrated castor oil with iron pentacarbonyl. Its structure was discussed based on its infrared, ultraviolet and NMR spectra, and from its ligand exchange reaction with triphenylphosphin.



By treating above its decomposition temperature, the complex polymerized with the coexisting diene carboxylic acid ester mainly into the dimer. By high pressure hydrogen-cracking, it was converted mainly into methyl stearate.

FRACTIONATION OF GLYCERIDES BY CRYSTALLIZATION. E. Sambuc (National Lab. of Fats and Oils (ITERG), Marseille, Fr.). *Rev. Franc. Corps Gras* **11**, 319–25 (1964). Crystal formation and the separation of liquid and solid phases is discussed. The different polymorphic forms, alpha, beta prime and beta are defined and the interconversion of the various crystal forms is discussed. These concepts are applied to glyceride fractionation. Phase diagrams is considered as a graphical representation of solid and liquid phases in balance. The graphs of glyceride fractionation by co- and countercurrent systems is discussed.

THE PARAFFIN HYDROCARBONS OF WOOL WAX. NORMAL, ISO, ANTEISO AND OTHER BRANCHED ISOMERS. J. D. Mold, R. E. Means, R. K. Stevens and J. M. Ruth (Research Dept., Liggett and Myers Tobacco Co., Durham, N.C.). *Biochemistry* **3**, 1293–1298 (1964). The relative abundance of the homologs of the series of normal, iso (2-methyl) and anteiso (3-methyl) paraffin hydrocarbons has been determined for a sample of wool wax. The occurrence of pristan (2,6,10,14-tetramethylpentadecane) has been established for the first time in a wax not of marine origin. The presence of homologous cycloparaffins and at least two other homologous series of branched paraffins has been noted in wool wax.

PROPORTION OF HYDROGENATION AND ISOMERIZATION DURING THE HYDROGENATION OF VEGETABLE OILS. J. Pokorný (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1**, 125–33 (1963). During the hardening of a vegetable oil to a hard fat, hydrogenation and isomerization proceed at approximately the same rate, so that the proportion of the two reaction products formed is nearly constant. In the case of the more unsaturated fats or where hydrogenation is used to produce soft plastic fats, hydrogenation rate exceeds the rate of isomerization.

THE EFFECT OF TEMPERATURE ON THE POLYMERIZATION OF GLYCEROL. J. Zajíc (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1**, 179–89 (1963). Pure redistilled glycerol was heated to temperatures between 140 and 260C under a vacuum of 380 mm Hg. No polyglycerol was formed after 72 hours at 180C. Formation of diglycerol was indicated after 48 hours at 200C. Formation of the higher polymers lagged considerably behind that of the lower ones, for example at a 25–35% concentration of diglycerol no triglycerol had yet formed. The polymerization reaction occurs through the intermediate compound 2,3-epoxypropanol which was found by infrared spectrophotometry. Nevertheless, the possibility of the transient formation of the glycerine radical is not excluded.

THE EFFECT OF ALKALINITY ON THE POLYMERIZATION OF GLYCEROL. *Ibid.*, 191–210. Between 0.05 and 5.0% Na_2O was added to glycerol in order to study its effect on the polymerization reaction conducted at 140 to 240C. While no reaction could be detected at 140C, even with 5.0% Na_2O and after 100 hr, at the higher temperatures an increasingly strong catalytic effect was noted. The reaction is believed to proceed through the intermediate formation of an alcoholate which was detected by paper chromatography. Alkalinity decreases during the course of the reaction due to a partial decomposition of glycerol, with formation of carbon dioxide, acids, etc. The organic impurities present in technical grade glycerol were found to have a positive effect in elevating the catalytic effect of alkalinity.

THE RELATIONSHIP BETWEEN THE PROTEIN AND THE LIPID CONTENT OF OATS. J. Pokorný, J. Hrdlička, V. Pliska and G. Janiček (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1**, 223–30 (1963). Contrary to other cereals, the lipid content of oats is not directly but rather inversely proportional to the protein content. On the basis of this finding, it may be assumed that only a part of the lipids participates in vital cell functions, while the main part has only energetic value.

VOLATILE CARBONYL COMPOUNDS IN AUTOXIDIZED FATS. J. Hrdlička and J. Pokorný (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1**, 113–24 (1963). Ten representatives of different types of fats were autoxidized at 180C by air bubbling and the volatilized carbonyl compounds were separated and identified by paper chromatography and spectral methods. In comparison to low temperature oxidation, more carbonyl compounds with 1 to 4 C atoms were found. Predominantly saturated fats formed mainly saturated compounds and ketones while a greater number of conjugated dienals were produced from highly unsaturated fats. The composition of the oxidation products depends on the total unsaturation and the fatty acid composition but not on the type of the most highly unsaturated acid present. Differences among individual oils are discussed.

THE HYDROGENATION OF SUNFLOWERSEED OIL AT LOW TEMPERATURES. J. Zajíc and J. Preiningerová (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1**, 135–50 (1963). Atmospheric hydrogenation of sunflowerseed oil was carried out at temperatures between 100 and 180C, with 0.3 to 2.0% Ni as the catalyst. *Trans* acid formation was highest (20–30%) at 180C and 0.3 0.5% catalyst and occurred at I.V. 65–70. At the lower temperatures, between 3 and 8% lower levels of *trans* acids were formed. Reaction rates for the various temperatures were calculated and the activation energy for the reaction calculated at 17.4 Kcal.

THE STABILITY OF FATS SUITABLE AS ADDITIVES TO POULTRY FEED. J. Pokorný, J. Zalud and M. Vasáková (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech Prague* **7–1**, 103–12 (1963). Attempts at stabilizing waste and other low quality fats through antioxidant addition, drying and bleaching, have been largely ineffective. Mixing the fats with extracted soybean meal, however, has a favorable effect, especially in the case of solid fats, where a 6- to 11-fold increase in stability is observed. In most cases stability of these mixtures could still be slightly improved by addition of a phenolic antioxidant.

FATTY ACID COMPOSITION OF LIPIDS EXTRACTED FROM OIL SEEDS AND FRUITS. D. Grieco and G. Piepoli (Chem. Lab. Grain Assoc., Milan, Italy). *Riv. Ital. Sostanze Grasse* **41**, 283–7 (1964). The fatty acid composition of 80 oil samples from 36 species of oil seeds and fruits are reported, as well as free acidity and fat content.

PAPER CHROMATOGRAPHY OF HIGHER FATTY ACIDS. J. Sliwiok and Z. Kwapniewski (Inst. Organ. Chem., Katowice, Poland). *Riv. Ital. Sostanze Grasse* **41**, 288–9 (1964). Two experimental dyes (Brilliant Green and Lauth Violet) have been found to be excellent developers in the paper chromatographic separation of fatty acids. A technique using dioctylphthalate as the impregnating substance is also described. By adding bromine to the mobile phase, it is possible to separate critical pairs, such as oleic from palmitic and myristic from linoleic.

ITALIAN EQUIPMENT FOR OLIVE PRODUCTION. G. Jacini and C. Carola (Exper. Sta. Fats and Oils, Milan, Italy). *Riv. Ital. Sostanze Grasse* **41**, 290–308 (1964). A detailed description is given of machinery produced in Italy for the pretreatment and pressing of olives.

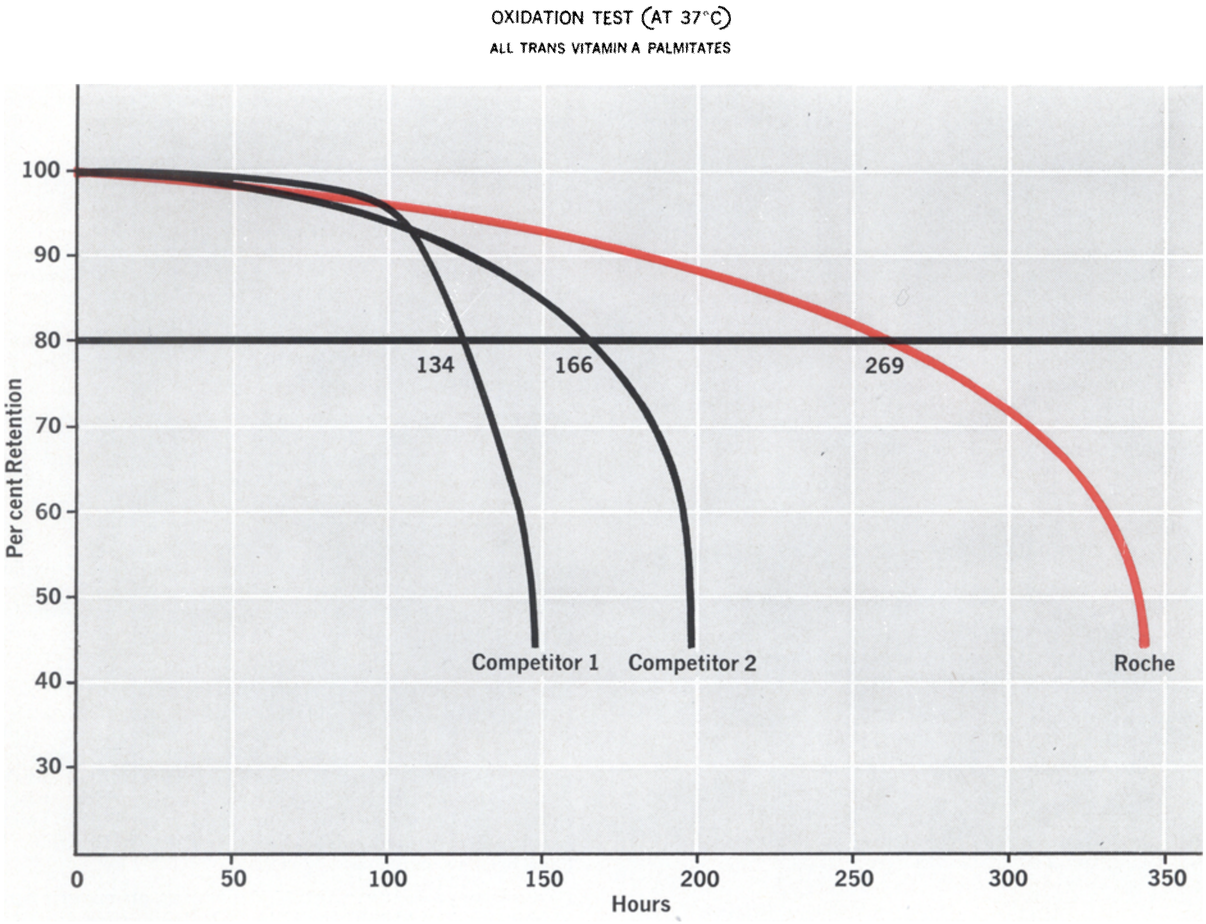
THE STABILITY OF EDIBLE VEGETABLE OILS. P. G. Garoglio (Univ. of Florence, Italy). *Riv. Ital. Sostanze Grasse* **41**, 181–8 (1964). A summary is given of the present knowledge and theories on the mechanisms involved in the autoxidation of fats.

DETERMINATION OF ELAIDINIC ACID BY THIN LAYER CHROMATOGRAPHY. U. Pallotta and L. Matarese (Univ. of Bologna, Italy). *Riv. Ital. Sostanze Grasse* **41**, 210–12 (1964). A thin layer chromatographic method is described which allows detection of as little as 0.25% elaidinic acid. Sensitivity of the method is at least four times greater than that of heretofore available methods.

THE FATTY ACID COMPOSITION OF BUTTER. V. Chioffi and G. Magon (Prov. Chem. Lab., Rovigo, Italy). *Riv. Ital. Sostanze Grasse* **41**, 243–5 (1964). The gas chromatographic fatty acid compositions of 31 butter samples collected over one year's period are reported.

THE BLEACHING OF VEGETABLE OILS. P. Fontana and O. Colagrande (Catholic Univ., Piacenza, Italy). *Riv. Ital. Sostanze Grasse* **41**, 246–50 (1964). The applicability of the Langmuir equation to the bleaching of olive oil has been examined, when either activated earth, coal, or a mixture of the two is used as the bleaching agent. The experimental points fit well on the straight line Langmuir relationship up to a certain value of the color reading, above which they begin to deviate appreciably. The adsorption mechanism in the case of coal is believed to be quite different from that of activated earth, exhibiting

What every margarine manufacturer should know about Roche Vitamin A



It's the most stable

Oxidation tests prove it.

Just as in these tests, Roche vitamin A palmitate provides extra protection in your margarine. That's because Roche vitamin A palmitate is all palmitate—the purest you can get anywhere.

Why is Roche vitamin A palmitate most stable?

Because of our unique, new process of total synthesis and specialized purification techniques. We start with pure, readily available, domestic materials. Throughout the entire process each intermediate is tested for purity before use in the next step.

Then every batch of vitamin A goes through a minimum of 35 separate quality control and stability tests. The oxidation test is just one of them.

Roche vitamin A is also taste and odor free. This makes it much easier for you to work with than the fish oils.

You can buy Roche vitamin A either alone or in batch size cans with Roche Beta Carotene and/or Roche Apo Carotenal.

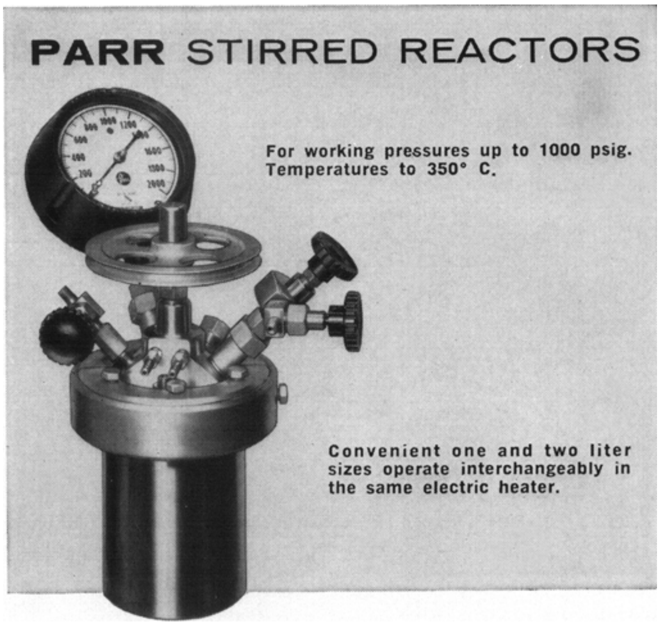
Call your Roche Representative for all the facts. Or call or write Industry Manager, Fine Chemicals Division, Hoffmann-La Roche, Nutley 10, N. J.

ROCHE Vitamin A

negative values for the K_1 and K_2 constants of the equation. However, blends of 20% coal:80% earth behave similarly to earth alone. Previous findings on the advantages of conducting the bleaching operation under a N_2 atmosphere are confirmed.

DEACIDIFICATION OF OILS THROUGH UREA ADDUCTS FORMULATION. J. Zajíc (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* 7–2, 181–92 (1963). Oils of low and medium acidity can be deacidified by urea adducts formation. Optimum results are obtained at 40C, in the presence of water (30%), under agitation and with a reaction time of two hours. An oil with 15.8% free oleic acid was reduced to 1.1% free acidity by use of a 30 molar excess of urea, while the removal of saturated acids proceeds somewhat more easily. However, completely neutral oils cannot be obtained, even if a very large excess of urea (240 mols) is used.

THE EFFECT OF STORAGE CONDITIONS OF RAPESEED ON THE QUALITY OF RAPESEED OIL. G. Janiček and J. Pokorný (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech., Prague* 7–2, 173–9 (1963). The changes taking place during the storage of rapeseed are *mainly* caused by enzymatic processes and only when enzymatic systems are largely deactivated do the purely chemical processes prevail. For this reason, the most unfavorable storage conditions are those that favor the development of active enzymatic systems connected with germination, e.g. storage at 40C in the presence of large amounts of water. However, the lipolytic processes are in no simple relationship to the decrease in stability, even though low stability is found in samples possessing high acidity. Freezing of the seed, leading to damage of the cells and increased enzymatic activity, deteriorates oil quality, while storage at 2–20C gives optimum results.



METHODS OF EVALUATION OF HYDROGENATED FATS. J. Pokorný, E. Mares, I. Zeman and M. Karvánek (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* 7–2, 223–38 (1963). Several margarine oils and other commercial hydrogenated fats have been evaluated with respect to their fatty acid composition, *trans* acids contents, consistency by melting point and differential thermal analysis, keeping quality.

THE RANCIDIFICATION OF OAT FLAKES AND THEIR STABILIZATION. J. Pokorný, F. Ginzelová and G. Janiček (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech., Prague* 7–2, 285–92 (1963). Oat flakes autoxidized rapidly when heated to temperatures of 105C or higher, while their peroxide content rose at the end of the induction period to a flat maximum, then decreased. The extent of oxidation is therefore better determined by following the changes in the refractive index. Moisture has a stabilizing effect, completely dry oat flakes are autoxidized rapidly. Antioxidants have only a moderate effect in slowing down oxidation, better results were obtained through addition of soybean lecithin. Rancidification is always preceded by the appearance of a bitter flavor which again disappeared towards the end of the induction period.

THE FORMATION OF EUTECTICS CAUSED BY THE ADDITION OF SYNTHETIC HARD BUTTERS TO NATURAL COCOA BUTTER. J. Pokorný, E. Mares, I. Zeman and G. Janiček (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* 7–2, 239–48 (1963). Lauric type synthetic hard butters form a eutectic mixture with cocoa butter when added to it in even small amounts. Thus at 30C both cocoa butter and the synthetic product are hard solids, but a blend of 94% cocoa butter and 6% hard butter becomes soft. A *statistically significant* correlation is found between the lauric acid content of the hard butter, or even better between the sum of the C_8 to C_{11} acids and the decrease in dilation. Improved thermal stability is found when the C_{12} content of the hard butter is less than 35% or even more when the hard butter contains only C_{16} and C_{18} fatty acids.

THE EFFECT OF COPPER ON THE STABILITY OF LARD CONTAINING BHT AND CYSTEIN. J. Pokorný and G. Janiček (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* 7–2, 193–8 (1963). The effect of copper in lowering the peroxide value of lard during the autoxidation of lard is caused by the metal's catalytic activity on the decomposition of peroxides. The maximum peroxide concentration in copper-containing samples was only one third as high as in untreated samples. These peroxide values were not affected by such additives as phenolic antioxidants or cystein. The effect of copper is especially remarkable in the elimination of the maximum in the peroxide curve found both in the case of untreated lard and of lard plus antioxidants.

SYNTHESIS OF ODD C-NUMBER UNSATURATED FATTY ACIDS. G. Grimmer and J. Kracht (Univ. of Hamburg, Germany). *Chem. Ber.* 96, 3370–3 (1963). A method for the preparation of unsaturated fatty acids with an odd number of C atoms and completely in the *cis* form is described. The method involves hydrogenation of a triple bond in methanol-pyridine, with Raney catalyst and leads to formation of only negligible amounts of *trans* and saturated acids. Fatty acids with 13 to 19 C atoms have been prepared and identified.

GAS CHROMATOGRAPHIC AND U.V. RESULTS ON PEANUT OIL. G. B. Martinenghi (AITOGA, Milan, Italy). *Olearia* 18, 47–53 (1964). The range of gas chromatographic fatty acid composition of peanut oils of different origins is as follows: lauric: nil to traces; myristic: nil to 0.3; palmitic: 9 to 18%; palmitoleic: traces to 0.7%; heptadecanoic: traces to 0.1%; stearic: 2.5 to 5%; oleic: 43 to 65%; linoleic: 15 to 33%; arachidic: 0.8 to 3%; linolenic: nil to 0.5%; eicosenoic: traces to 1.5%; behenic: 1.5 to 4%; lignoceric: 0.5 to 3%. Erucic acid is consistently absent and its presence up to 0.5% is believed due to contamination at the oil producing site. U.V. spectrophotometric data on peanut oil are also reported.

CARBONYLS IN OXIDIZING FAT. VI. THE GIRARD T REAGENT IN THE ISOLATION AND DETERMINATION OF MICRO AMOUNTS OF N-ALIPHATIC ALDEHYDES AND 2-ALKANONES. A. M. Gaddis, R. Ellis and G. T. Currie (Meat Lab., East. Util. Res. and Dev. Div., Agric. Res. Service, U.S.D.A., Beltsville, Md.). *J. Food Sci.* 29(1), 6–16 (1964). Complete reaction was obtained between Girard T reagent and *n*-alkanals, alk-2-enals, alk-2,4-dienals and 2-alkanones of carbon chain length up to C_{13} when anhydrous tertiary butyl alcohol was the solvent. In the presence of fat the reaction was greatly retarded or inhibited. However, by the use of water in the reaction, quantitative recovery of an aldehyde homologous series from fat was obtained. Optimum reaction occurred at room temperature.

(Continued on page 42)

(Continued from page 34)

PROCESS FOR WRAPPING MARGARINE AND BUTTER. E. L. Josefowicz and D. Melnick (Corn Products Co.). *U.S. 3,148,993.* An edible fat sealant for use of margarine or butter wrappers consists of 100% partially hydrogenated soybean oil, 98% partially hydrogenated soybean oil with 2% completely hydrogenated peanut oil, or 96.5% partially hydrogenated soybean with 3.5% completely hydrogenated cottonseed stearine having a melting point ranging from 105–114F.

ADJUVANT VACCINE WITH ALUMINUM MONOSTEARATE, MANNIDE MONOOLEATE, VEGETABLE OIL AND AN AQUEOUS PHASE IMMUNOLOGICAL AGENT. A. F. Woodhour and T. B. Stim (Merck & Co., Inc.). *U.S. 3,149,036.* A parenteral vaccine preparation in the form of a water-in-oil emulsions comprises: A) an emulsion vehicle which contains, on a volume basis, 0.5–5% aluminum monostearate, 20–75% vegetable oil and 2.5–25% mannide monooleate: B) sufficient aqueous phase to bring the volume to 100%. The aqueous phase has incorporated in it, prior to emulsification, an immunological agent in an amount sufficient to elicit antibody response when parenterally administered to a host.

BATH OIL. M. F. Emory, *U.S. 3,150,049.* A bath oil composition consists of 5–20% by weight of perfume oil; 0.5–3.0% of lecithin; the balance to 100% consists of at least one substance selected from the group consisting of cosmetically applicable vegetable oils and esters of monocarboxylic and dicarboxylic acids having at least 6 carbon atoms in the molecule. The composition is stable on storage and, when brought in contact with excess water, is uniformly dispersed to very fine particles.

STABILITY OF EPOXIDIZED OILS BY OXIDATION AND NATURALIZATION. S. A. Harrison and H. G. Simmerman (General Mills, Inc.). *U.S. 3,150,153.* The process comprises oxidizing an oil consisting of esters of epoxidized fatty acids derived from un saturated fatty acids containing from 8–22 carbon atoms and neutralizing the resulting oxidized oil with an aqueous alkaline material. The oxidizing and subsequent neutralizing are sufficient to improve the heat stability and odor of the oil.

• Fatty Acid Derivatives

SILICON CONTAINING HEAT RESISTANT OILS BASED ON FATTY ACIDS. R. M. Ismail (Res. Lab. of H. T. Zimmer, Frankfurt). *Fette Seifen Anstrichmittel* **66**, 356–358 (1964). New types of organoorganoxy silanes have been prepared in good yield from fatty derivatives and functional silanes. The oils obtained show properties of conventional oils with thermal stability.

SYNTHESIS OF THE PRIMARY ALCOHOLS $C_{19}H_{39}OH$ TO $C_{30}H_{61}OH$. H. Schildknecht and G. Renner (Inst. Org. Chem., Univ. Erlanger). *Fette Seifen Anstrichmittel* **66**, 176–182 (1964). Synthesis of the primary alcohols from nonadecyl alcohol to triacintanol are described. They were prepared from behenic and palmitic acids; stearyl alcohol and *n*-octanol utilizing standard methods of chain extension and degradation. Physical properties and constants of the products and their derivatives are given.

CAFFEOYL GLYCERIDES. H. G. C. King (U.S. of America, Secy. Agr.). *U.S. 3,153,659.* Described are glyceride caffeic acid esters selected from the group consisting of 1-mono-caffeoyl glycerol, dicaffeoyl-monopalmitin and tricaffeoyl glycerol.

• Biochemistry and Nutrition

SYNTHESIS OF α-CEPHALINS BY A NEW PROCEDURE. II. DIOLEOYL AND DILINOLEOYL L-α-CEPHALINS. E. Baer and J. Blackwell (Banting and Best Dept. of Med. Res., Univ. of Toronto, Toronto, Ontario, Canada). *Biochemistry* **3**, 975–6 (1964). The synthesis of L-α-(dioleoyl)cephalin and L-α-(dilinoleoyl)cephalin by a new procedure is described. They were obtained by treating the barium salt of L-α-glycerylphosphoryl-2′-hydroxyethylphthalimide with the chloride of oleic acid or linoleic acid and pyridine and freeing the N-phthaloyl cephalins from their protective phthaloyl group by hydrazinolysis in an atmosphere of nitrogen.

CYCLOPROPANE FATTY ACID SYNTHETASE: PARTIAL PURIFICATION AND PROPERTIES. A. E. Chung and J. H. Law (Dept. of Chemistry, Harvard Univ., Cambridge, Mass.). *Biochemistry* **3**, 967–74 (1964). The cyclopropane fatty acid synthetase from *Clostriduim butyricum* has been partially purified. The enzyme system transfers the methyl group of S-adenosylmethionine to a monounsaturated fatty acid chain of phosphatidylethanolamine to form a cyclopropane-fatty acid chain. The enzyme system is stimulated by anionic surfactants and inhibited by cationic and neutral surfactants. The Michaelis constants for phosphatidylethanolamine and S-adenosylmethionine are 5.3×10^{-4} M and 5.7×10^{-5} M, respectively. The Arrhenius plot for the enzyme reaction is discontinuous over the temperature range 0–40C. The heat of activation for the enzyme reaction is lowered by the addition of the anionic surfactant sodium lauryl sulfate.

CONVERSION OF LINOLEIC-1-C^{14} ACID TO ARACHIDONIC ACID IN THE GERBIL. S. Gordon and J. F. Mead (Dept. of Biophysics and Nuclear Med., Univ. of Calif., Los Angeles, Calif.). *Proc. Soc. Exp. Biol. Med.* **116**, 730–33 (1964). The fatty acid composition of total body fat of the gerbil was determined by gas liquid chromatography and compared to that of the rat. Oleic was the major fatty acid while in the rat linoleic and oleic were the predominant acids. Linoleic-1-C^{14} acid was administered orally as the methyl esters and C^{14} arachidonic acid isolated from total body fat. Stepwise degradation of the product revealed the synthesis of arachidonic acid, as in the rat, by the condensation of linoleate with an acetate moiety derived from linoleic acid.

THE SYNTHESIS OF PHOSPHATIDIC ACID AND PROTEIN-BOUND PHOSPHORYLSERINE IN SALT GLAND HOMOGENATES. M. R. Hokin and L. E. Hokin (Dept. of Physiol. Chem., Univ. of Wisconsin, Madison 6, Wis.). *J. Biol. Chem.* **239**, 2116–22 (1964). The incorporation of P^{32} from adenosine triphosphate-P^{32} into phosphatidic acid and into protein-bound phosphorylserine has been studied in salt gland homogenates under conditions in which $(Na^{+} + K^{+})$-dependent, ouabain-inhibitable adenosine triphosphatase activity occurs. P^{32} was incorporated into both phosphatidic acid and protein-bound phosphorylserine over the time intervals studied (5 seconds to 1 minute). When phosphatidic acid was made radioactive by stimulation of salt gland slices with acetylcholine in the presence of P^{32} and when the tissue was then homogenized, there was a loss of radioactivity from phosphatidic acid. This loss is compatible with a shift of the steady state level of an intermediate from phosphatidic acid to diglyceride at ouabain-sensitive ATPase sites, but it cannot be interpreted as positive evidence that this occurs.

THE OCCURRENCE OF α-AMINO-β-PHOSPHONOPROPIONIC ACID IN THE ZOANTHID, ZOANTHUS SOCIATUS AND THE CILIATE, TETRAHYMENA PYRIFORMIS. J. S. Kittredge and R. R. Hughes (Dept. of Biochemistry, City of Hope Med. Center, Duarte, Calif.). *Biochemistry* **3**, 991–6 (1964). A new amino acid, α-amino-β-phosphonopropionic acid, was first detected in aqueous-ethanolic extracts of the zoanthid, *Zoanthus sociatus*, by ion-exchange chromatography and paper electrophoresis. The identity of the natural and the synthetic amino acid was established by isolating the labeled amino acid from the hydrolysate of an aqueous-ethanolic extract of the ciliate, *Tetrahymena pyriformis*, which had been cultured in media containing phosphate-P^{32}, crystallizing it in the presence of the synthetic amino acid and demonstrating a constant specific activity through four serial crystallizations. The labeled and the synthetic compound also exhibited complete coincidence during paper chromatography, paper electrophoresis and ion-exchange chromatography.

ON THE STRUCTURE OF CARDIOLIPIN. J. LeCocq and C. E. Ballou (Dept. of Biochem., University of Calif., Berkeley, Calif.). *Biochemistry* **3**, 976–80 (1964). Cardiolipin (diphosphatidylglycerol) from beef heart and from *Mycobacterium phlei* has been deacylated and degraded to glycerol-1,3-diphosphate by the action of sodium metaperiodate and 1,1-dimethylhydrazine. The results establish that the lipid has the 1,3-diphosphatidylglycerol structure, a point that was still open to question. For references, crystalline cyclohexylamine salts of glycerol-1,3-diphosphate and L-glycerol-1,2-diphosphate have been prepared. The reported optical activity of the triglyceroldiphosphate obtained by deacylation of cardiolipin has been confirmed and is found to be identical with that of synthetic 1,3-di-O-(L-glycerol-3′-phosphoryl)-glycerol.

BIOCHEMICAL CORRELATES OF RESPIRATORY DEFICIENCY. III. THE LEVEL OF SOME UNSAPONIFIABLE LIPIDS IN DIFFERENT STRAINS OF BAKER'S YEAST. H. R. Mahler, G. Neiss, P. P. Slonimski and

(Continued on page 44)

(Continued from page 42)

B. Mackler (Dept. of Chemistry, Indiana Univ., Bloomington). *Biochemistry* 3, 893–5 (1964). Two wild-type (respiratory-sufficient, "grande") and one cytoplasmic-mutant (respiratory-deficient, "cytoplasmic petite") yeasts have been analyzed for their content of unsaponifiable lipids with special emphasis on their content of ubiquinone (coenzyme Q). The methods used involve saponification with methanolic pyrogallol, alumina chromatography and spectrophotometric identification and analysis. Under controlled physiological conditions the differences in content of ubiquinone, tocopherol, vitamin A, ergosterol, total steroid and total unsaponifiable lipids between the wild type and its "petite" mutant are not considered significant.

INTRACELLULAR LOCALIZATION OF LIPOFUSCIN AGE PIGMENTS IN THE NERVOUS SYSTEM. T. Samorajski, J. R. Keefe and J. M. Ordy (Lab. of Neurochemistry, Cleveland Psychiatric Inst., Cleveland, Ohio). *J. Gerontol.* 19, 262–76 (1964). The objectives of the present study were to establish the possible role of lysosomes in the formation of lipofuscin pigment in the nervous system. Histochemical and ultrastructural comparisons were made of intracellular lipofuscin pigments in the nervous system of the mouse, rat and man. In formalin-fixed tissues of the old human and animal Ss, the PAS-straining procedure revealed granular inclusions which appeared in clusters varying in size and shape. They were located mostly in the peripheral portion of the perikarya. Histochemical analyses revealed that these granules contained neutral mucopolysaccharide with lipid and protein fractions which exhibited the physical properties and staining characteristics of lipofuscin-pigment.

INFLUENCE OF GLYCEROL AND SODIUM ACETATE ON THE LIPID COMPOSITION OF STREPTOCOCCUS CREMORIS. J. P. Brown and Patricia MacLeod (Dept. of Animal Industries, Univ. of Conn., Storrs). *J. Dairy Sci.* 47, 831–35 (1964). Cells of *Streptococcus cremoris* were grown in the presence of various levels of glycerol and sodium acetate. The cultures were harvested in the maximum stationary phase and the lipids subsequently extracted and analyzed. The presence of either metabolite resulted in stimulated lipid synthesis. The lipids were fractionated into five major groups by thin layer chromatography. These groups were designated as containing phospholipids, mono-, di- and triglycerides and esterified fatty acids or wax esters. The fatty acid compositions of these fractions in the lipid from the control (no added metabolite) and treated cells were determined. The presence of actate favored the synthesis of phospholipids and influenced the percentage of 18:1 acid(s) incorporated within this fraction.

EFFECT OF LINOLEIC ACID ON NUTRITIONAL MUSCULAR DYSTROPHY IN THE CHICK. C. C. Calvert, I. D. Desai and M. L. Scott (Dept. of Poultry Husbandry and Graduate School of Nutr., Cornell Univ., Ithaca, New York.). *J. Nutr.* 83, 307–13 (1964). Experiments were conducted which showed that in the absence of dietary linoleic acid, muscular dystrophy does not occur in chicks even when the diet is low in both vitamin E and sulfur amino acids. The addition of graded levels of linoleic acid to the diet resulted in proportionate increases in the incidence and severity of muscular dystrophy. The vitamin E requirement for prevention of muscular dystrophy was increased with increasing levels of linoleic acid up to 0.5% linoleic acid. No increase was observed in the vitamin E requirement for prevention of muscular dystrophy when the level of linoleic acid was increased from 0.5 to 2.5% of the diet. Vitamin E at a level which completely prevented muscular dystrophy had no effect on the level of linoleic acid in muscle lipids, which increased in both dystrophic and non-dystrophic chicks in proportion to the amount of linoleic acid in the diet.

INCIDENCE OF ENDOGENOUS LIPOPROTEIN LIPASE ACTIVITY IN HUMAN PLASMA. H. Engelberg (Div. of Lab., Cedars of Lebanon Hosp., Los Angeles, Calif.). *Proc. Soc. Exp. Biol. Med.* 116, 422–5 (1964). Endogenous lipoprotein lipase activity was found in the plasma in 75% of normal subjects.

INFLUENCE OF AMOUNT AND TYPE OF FAT ON METABOLIC EFFICIENCY OF ENERGY UTILIZATION BY CHICK. L. B. Carew, Jr., D. T. Hopkins and M. C. Nesheim (Dept. of Poultry Husbandry and Graduate School of Nutr., Cornell Univ., Ithaca, New York.). *J. Nutr.* 83, 300–06 (1964). Studies were conducted to determine the influence of fats of varying fatty acid composition on the efficiency of energy utilization by chicks. When chicks were fed equal intakes of metabolizable energy from low fat diets or diets containing corn oil, beef tallow, soybean oil, or a lightly hydrogenated olive oil, more energy was deposited in the carcasses of chicks fed diets containing the fats than in the carcasses of chicks fed the low fat diet. However, no increase in energy deposition in the carcass was observed if diets containing hydrogenated coconut fat were fed compared with low fat diets. Ten per cent of corn oil in the diet was nearly as effective in improving efficiency of energy utilization as levels up to 34.9% of the diet.

EFFECT OF CORN OIL ON METABOLIC EFFICIENCY OF ENERGY UTILIZATION BY CHICKS. L. B. Carew, Jr. and F. W. Hill (Dept. of Poultry Husbandry and Graduate School of Nutr., Cornell Univ., Ithaca, N.Y.). *J. Nutr.* 83, 293–99 (1964). Studies were made of the tissue composition and tissue energy gains of chicks receiving equal intake of metabolizable energy and all other nutrients from diets containing 2.5, 12.5 and 22.5% corn oil substituted isocalorically for glucose. Tissue energy gains per unit of metabolizable energy intake increased as the dietary level of corn oil was increased. It is concluded that replacement of dietary carbohydrate with corn oil increases the metabolic efficiency of energy utilization by chicks. Increases in tissue energy were due solely to increased fat content. However, no significant effect of corn oil on growth rate was observed, indicating an inverse correlation between tissue fat deposition and water retention. Based on results reported in the literature for the chicks and other species, it appears most probable that the beneficial effect of corn oil on efficiency of energy metabolism is mediated through the heat increment component rather than the basal component of heat production.

BIOCHEMISTRY OF THE SPHINGOLIPIDS. XVII. THE NATURE OF THE OLIGOSACCHARIDE COMPONENT OF PHYTOGLYCOLIPID. H. E. Carter, B. E. Betts and D. R. Strobach (Div. of Biochem., Noyes Lab. of Chem., Univ. of Ill., Urbana). *Biochemistry* 3, 1103–07 (1964). The oligosaccharide fraction obtained by hydrolysis of phytoglycolipid from corn has been separated on Dowex-2 (HCO_3^-) into a series of oligosaccharides of progressively increasing molecular weight. The first number of the

(Continued on page 48)

(Continued from page 44)

series is the trisaccharide, glucosaminido-glucuranido-inositol. This trisaccharide is obtained by mild acid hydrolysis of all the higher oligosaccharides and accounts essentially quantitatively for their glucosamine and glucuronic acid content. The tetrasaccharide fraction was characterized by preparation of crystalline derivatives and was shown to contain only mannose (in addition to trisaccharide). The higher oligosaccharide fractions contained, in addition, varying amounts of galactose, arabinose and fucose. The penta-, hexa-, hepta- and octasaccharide fractions were in each case mixtures from which no single constituent was separated.

SYNTHESIS IN VITRO OF GLYCERIDE-GLYCEROL BY LIVER OF NORMAL AND PANCREATECTOMIZED RATS. S. S. Chernick and R. O. Scow (Lab. of Nutr. and Endocrinology, National Institute of Arthritis and Metabolic Diseases, National Institutes of Health, Bethesda 14, Maryland). *J. Biol. Chem.* 239, 2416–19 (1964). Incorporation of uniformly labeled fructose-C^{14} and glucose-C^{14} into fatty acid and glyceryl moieties of lipids was studied in liver slices from normal and pancreatectomized rats. Deprivation of food and insulin deficiency markedly reduced fatty acid synthesis *in vitro* but had no effect on glyceride-glycerol synthesis. The amount of glyceride-glycerol formed per hour was equal to 1.5 to 2% of the total glyceride-glycerol content of the liver.

THE CONDITIONS OF EXTRACTION OF RAPESEED PROTEINS WITH SODIUM HYDROXIDE. J. Pokorný (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* 7–2, 149–56 (1963). The optimum conditions for the extraction of protein from rapeseed meal differ slightly from those for other oilseed meals. Use of a tenfold amount of solvent, 20 40 min extraction time and 0.1–0.2% aqueous NaOH as the solvent are recommended. The extraction conditions must be mild to avoid excessive contamination of the extract with non-protein substances as well as protein decomposition, so that the yield of protein must not be the only criterion for the selection of the optimum operating conditions.

THE PECTIN CONTENT OF VIRGIN OLIVE HUSKS. A. Corrao (Univ. of Palermo, Italy). *Olearia* 18, 57-60 (1964). The pectin content of samples of virgin olive husks of different origins is rather low, not exceeding 2.5% on a dry, fat-free basis.

FATTY ACID COMPOSITION AS INFLUENCED BY DIETARY FATTY ACIDS AND VITAMIN E STATUS IN THE RABBIT. R. F. Borgman (Clemson College, Clemson, S. Carolina). *J. Food Sci.* 29(1), 20–23 (1964). Weaning New Zealand rabbits received a semipurified diet containing either semipurified oleic acid or semipurified linoleic acid, both with and without vitamin E. Results indicate that vitamin E requirements for skeletal muscle degeneration are very low when oleic acid is in the diet. Vitamin E did not greatly influence the number of gallstones found and those rabbits on the oleic acid diets tended to have more gall stones than those on the linoleic acid diet. The influence of the dietary fatty acid upon the fatty acid composition of the liver, kidneys, heart, adipose deposits, skeletal muscle and thoracic aorta was profound. The liver was less affected than the other organs for those rabbits receiving oleic acid. The heart and adipose deposits of rabbits receiving linoleic acid diets were affected more than the other organs. Minor differences were noted for the vitamin E diets and their deficient counterparts.

A CONTRIBUTION TO THE KNOWLEDGE OF THE STRUCTURE OF TWO HYDROPERICARDIUM—PRODUCING FACTORS FROM A TOXIC FAT. J. C. Wootton and W. L. Courchene (The Procter and Gamble Co., Miami Valley Laboratories, Cincinnati, Ohio). *J. Agr. Food Chem.* 12(1), 94–98 (1964). Mass and other spectral data indicate that the two compounds, previously isolated from a toxic fat and capable of producing hydropericardium in chicks, were isomers with a molecular formula of $C_{14}H_{10}Cl_6$. By reductive dehalogenation of 1 mg quantities of these chlorinated hydrocarbons and gas chromatography of the resulting saturated hydrocarbons, strong evidence was obtained that they are hexachlorohexahydrophenanthrenes.

BIOCHEMICAL PROPERTIES OF PORK AND THEIR RELATIONSHIP TO QUALITY. II. INTRAMUSCULAR FAT. R. G. Kauffman, Z. L. Carpenter, R. W. Bray and W. G. Hoekstra (Depts. of Meat and Animal Sci. and Biochem., Univ. of Wis., Madison, Wis.). *J. Food Sci.* 29(1), 70 75 (1964). Greater quantities of fat were associated with higher flavor, tenderness and especially juiciness. Also as the fat content increased, lower curing and cooking shrinkages resulted.

III. DEGREE OF SATURATION AND MOISTURE CONTENT OF SUBCUTANEOUS FAT. *Ibid.*, 75 79. Degree of unsaturation and per cent moisture of the subcutaneous fatty tissue decreased as the fat content of the young animal increased; with mature sows, this pattern was not evident. Tissue containing a greater amount

(Continued on page 50)

(Continued from page 48)

of moisture and unsaturated fatty acids were related to a softer and leaner pork carcass with less favorable palatability characteristics. Neither extent of unsaturation nor moisture content of the tissue was related to the odor of the heated samples of the fat.

THE FATTY ACID COMPOSITION OF LIVER LIPIDS FROM RATS RAISED ON PORK RATIONS. I. J. Tinsley (Dept. of Agr. Chem., Oregon State Univ., Corvallis, Oregon). *J. Food Sci.* 29(2), 130–136 (1964). The liver lipid fatty acid composition of animals raised on pork rations was determined and compared with that of animals raised on a stock ration. The pork rations contained 25% crude lipid, the proportion of oleic acid being 46–50%. The tissue levels of oleic, linoleic and arachidonic acids provides supporting evidence for the existence of a competitive effect of oleic acid in the conversion of linoleic to arachidonic acid. Results suggest also that the dietary oleate/linoleate ratio is important in essential fatty acid nutrition in rations containing appreciable quantities of oleic acid.

SUSCEPTIBILITY OF ERYTHROCYTES OF VARIOUS ANIMAL SPECIES TO THE HEMOLYTIC AND PHOSPHOLIPID SPLITTING ACTION OF SNAKE VENOM. E. Condrea, Z. Mammon, S. Aloof and A. De Vries (The Rogoff Med. Res. Inst., Dept. of Exp. Biol. of Tel Aviv Univ.). *Biochim. Biophys. Acta* **84**, 365–375 (1964). A parallelism was found between hemolysis and erythrocyte phospholipid splitting induced by the action of cobra venom on washed erythrocytes of various species: guinea-pig, dog, human, rabbit. No significant phospholipid splitting was produced by cobra venom in camel and sheep erythrocytes, which are resistant to hemolytic action of venom. Isolated cobra venom phospholpiase A (phosphatide acylhydrolase) had no/or slight hemolytic and phospholipid splitting action on various erythrocytes, including those of guinea-pig and dog, which are most sensitive to action of the whole venom. Different sensitivity of the various erythrocytes to cobra venom is a reflection of their susceptibility to action of venom direct lytic factor, a basic protein, that has hemolytic but no phospholipase activity. Isolated cobra venom phospholipase readily hydrolyzed the phospholipids of osmotic ghosts derived from both sensitive and resistant erythrocytes.

ON THE OCCURRENCE OF DIPHOSPHOINOSITOL IN THE LIPIDS OF LIVER AND PANCREAS. G. A. Kfoury and S. S. Kerr (Dept. of Biochem., Amer. Univ. of Beirut, Beirut, Lebanon). *Biochim. Biophys. Acta* **84**, 391–403 (1964). The lipids of liver were re-examined for polyphosphoinositide, previously reported absent. The presence of a diphosphoinositide (DPI) was revealed by the isolation of inositol diphosphate (IP_2) from an acid hydrolysate and its glyceryl derivative from an alkaline hydrolysate of the concentrated phospholipid fraction.

THE INTESTINAL ABSORPTION AND METABOLISM OF MICELLAR SOLUTIONS OF LIPIDS. J. M. Johnston and B. Borgstrom (Dept. of Physiological Chem., Univ. of Lund, Lund, Sweden). *Biochim. Biophys. Acta* **84**, 412–423 (1964). The absorption of micellar solutions of conjugated bile salts and labeled fatty acids and/or monoglycerides have been demonstrated employing intestinal slices and brush border preparations. The absorption of the lipid from these solutions appears to be enzymatically and energetically independent and occurs at a faster rate than corresponding solutions bound to albumin or in the emulsified state. The resynthesis of the absorbed micellar lipids into triglycerides has been demonstrated and the relationship of these findings to the mechanism of the absorption of fats *in vivo* discussed.

METHOD OF ADMINISTERING ALDOHEXOSE-UREA HYPOGLYCEMIC COMPOUNDS TO ANIMALS. H. S. Clark (Universal Drug & Res. Labs., Inc.). *U.S. 3,149,033.* A method for lowering the blood sugar in animals consists of orally administering a compound taken from the group consisting of the reaction product of an aldohexose steroisomer and urea and fatty acid esters thereof.

VITAMIN COMPOSITIONS. R. E. Aiello and J. C. Bauernfeind (Hoffmann-La Roche, Inc.). *U.S. 3,149,037.* An emulsifiable vitamin solution comprises 1) up to 70% by weight of a fat-soluble vitamin (vitamin A alcohol, vitamin D, vitamin E and mixtures thereof), 2) at least 0.5% by weight of an emulsifying agent selected from the group consisting of polyethylene glycol esters of fatty acids having 16–18 carbon atoms and polyoxyethylene sorbitan esters of fatty acids having 16–18 carbons, 3) 1.0–10.0% benzyl alcohol, 4) at least 0.1% of an antioxidant selected from the group consisting of butylated hydroxyanisole, butylated hydroxytoluene and mixtures thereof, the remainder of the solution comprising 5) a glyceride selected

(Continued on page 52)

(Continued from page 50)

from the group consisting of a glyceryl triester of a saturated fatty acid having 6–12 carbon atoms, a glyceryl triester of a mixture of saturated fatty acids with 6-12 carbons and mixtures of the glyceryl triesters.

• Detergents

Qualitative and quantitative infrared spectroscopic experiments on ethylene oxide adducts. Chr. Glassmann and K. Maennchen (Phys. Opt. Zehimmer & Schwarz, E. Leitz, Wetzlar). *Fette Seifen Anstrichmittel* **65**, 741–747 (1963). Infrared spectroscopic measurements on ethylene oxide adducts have produced a method for the determination of the amount of hydroxyethylation of various materials. Accurate results can be obtained from fat-alcohol polyglycolethers and alkylphenolpolyglycolethers. Representative infrared spectra are given.

Determination of dimethylformamide in sucrose esters by infrared spectrophotometry. J. Zajíc and M. Bares (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–2,** 215–22 (1963). Very small amounts of dimethylformamide in sucrose esters can be analyzed for by IR in the region of 1650-1690 cm^{-1}, which is the region of amide group vibration. *n*-Butanol is somewhat more suitable as a solvent than chloroform since it gives a more narrow absorption band for the carbonyl group. Sensitivity of the method is about 0.01% dimethylformamide with *n*-butanol and 0.1% with chloroform, with an analytical error of no more than ±10%.

Preparation of mono- and diesters of sucrose with oleic and linoleic acids. J. Zajíc and B. Auerswald (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–2,** 205–13 (1963). The course of the esterification reaction between sucrose and methyl esters of oleic and linoleic acids was investigated. The reaction was carried out in dimethylformamide, with potassium carbonate as catalyst, at 90C and under vacuum. The end product composition could be controlled by a suitable choice of reactants. The course of the reaction can be followed by means of specific rotation, but only when monoesters are being prepared. The prepared sucrose esters had a glassy, resin-like appearance and yellowish color.

The effect of partially acetylated mono- and diglycerides on the consistency of cosmetic creams. J. Pokorný, V. Pokorná and I. Tománková (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1,** 167–78 (1963). Partially acetylated mono- and diglycerides, as compared to completely acetylated derivatives, produce cosmetic creams with better thermal resistance, better emulsion stability and better spreadability. Consistency of the creams was judged by the diameter of stripes placed under constant load between two glass plates and was found to increase appreciably during the first 17 days after making.

The preparation of mono- and diesters of sucrose with lauric and stearic acid. J. Zajíc and M. Bares (Inst. Chem. Tech., Prague). *J. Inst. Chem. Tech. Prague* **7–1,** 151–66 (1963). The course of the esterification reaction between sucrose and methyl esters of lauric and stearic acid was studied. Potassium carbonate was used as the catalyst and dimethylformamide as the solvent. Specific rotation was found to be unsuitable as a means of reaction control. It was more convenient to trace the reaction by calculating the molar ratio of the reacting components, after determination of the catalyst and soap contents. Refractive index was found acceptable for a rapid control of the reaction course, reaching a constant value at equilibrium. Measurement of the dielectric constant during the reaction also failed to give reproducible results.

Soap compositions. W. A. Kelly (Lever Brothers Co.). *U.S. 3,150,097.* A soap bar consists of a water-soluble soap which normally forms water-insoluble soap curd in hard water selected from the group consisting of sodium and potassium salts of fatty acids of about 8–18 carbon atoms and from 10–30% of an alkaline earth metal salt of a mixture of sulfonated monophenyl-substituted alkanes having an alkane structure corresponding to a mixture of acrylic polypropylenes. The acid form of the sulfonated mixture has an average molecular weight of between 335 and 370, the alkaline earth metal is selected from the group consisting of calcium and magnesium and the proportions are based on the combined weight of the soap and the sulfonate.

Liquid detergent. J. H. Wilson (Lever Brothers Co.). *U.S. 3,150,098.* A uniform, light duty, liquid detergent composition consists of an aqueous solution of from 40-50% by weight total solids content. The solids content consists of 15–63% by weight of an alkylbenzene sulfonate having an alkyl group averaging from 12–15 carbon atoms, 13–53% of an alkylphenoxypolyoxyethylene sulfate having an alkyl group of 9–12 carbons and containing from 4 6 oxyethylene groups and 17–45% of a short chain alkylaryl sulfonate. The relative amounts of the solids components are adjusted to give the composition uniformity and a viscosity of 150–625 centipoises at 80F. The cationic portions of the three components are selected from the group consisting of Na, K and NH_4.

Solubilizer for synthetic detergent. L. R. Schiltz and R. H. Rogers (Swift and Co.). *U.S. 3,151,084.* A surface active detergent composition, characterized by exhibiting a high degree of water solubility at effective concentration levels contains: a detergent consisting essentially of an alkyl aryl sulfonate and an inorganic detergency builder salt and at least 0.25% by weight of a mixture consisting of 50 95% by weight of ethylene diamine tetraacetic acid tetrasodium and from 5–50% of the sodium salt of N,N-di(2-hydroxyethyl)glycine.

Process of preparing fatty acid esters of hydroxyalkane sulfonic acids. E. Koczorowski, L. Habicht and A. Kluge. *U.S. 3,151,136.* The process of preparing a fatty acid ester of a fatty acid ester of a hydroxyalkane sulfonic acid compound in high yield comprises heating fatty acid an a hydroxyalkane sulfonic acid which is substantially free from its salts at a temperature from 100–150C for a period of time up to about 1 and ½ hours and simultaneously removing the water of reaction.

Resinous compositions as detergent additives. G. P. Touey and H. E. Davis (Eastman Kodak Co.). *U.S. 3,152,993.* A composition soluble in water to form a washing solution which is effective not only to remove soil from textile materials but also to minimize redeposition of removed soil comprises the mixture of an organic anionic detergent and a water soluble sulfonate salt of a condensation product of formaldehyde with either urea or melamine.

◆

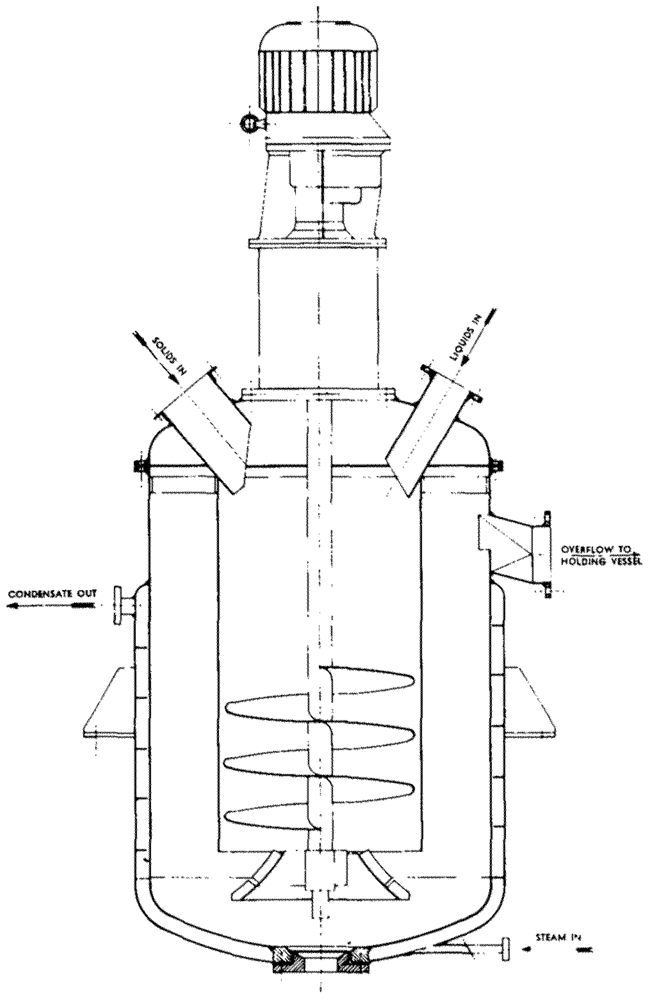


FIG. 3. Continuous mixer.

the world the majority of laundering is still done by hand, so this becomes an extremely important consideration. Removal of lumps and gritty particles is even more important for the steady, reliable operation of the high pressure pump. The high pressure pump is a positive displacement piston pump which relies on inlet and outlet valves seating properly during each stroke. Gritty particles and lumps interfere with the proper seating of these valves and pumping pressures are impossible to maintain.

A steady pumping pressure at the spray nozzle is absolutely essential to the manufacture of quality-sprayed product. Without perfect control over the spraying pressure it is impossible to precisely control the density and moisture of the finished product.

Spray Tower

The spray tower is an evaporator first and formost (Fig. 4). Its specific function is to dry the slurry from 30–35% moisture down to the finished product moisture of 10%. To accomplish this drying, the slurry is atomized into small droplets to provide a very large surface area for efficient heat and mass transfer. Almost without exception pressure atomization is used. Heated air is used to provide the heat for drying. The hot air and the sprayed slurry are contacted in the large cylindrical chamber either counter currently, which is most common, or concurrently.

As soon as the atomized slurry leaves the nozzle and comes into contact with the drying air, heat and mass transfer take place. Because of the temp difference between the drying air and the droplet of slurry, heat is transferred to the droplet at a rate proportional to this temp difference. Simultaneously, heat is being removed from the droplet due to the evaporation of the water from its surface. The rate at which heat is being transferred to the droplet is greater than the rate at which it is being removed by surface vaporization and the water within the droplet vaporizes. This internal vaporization causes the still plastic droplet to puff up.

When the surface of the resulting sphere can be extended no further, it ruptures and the entrapped steam escapes. Thus it is easy to understand why concurrent spraying can yield products of lighter density than can counter-current operation. With concurrent operation the hot drying air enters at the top of the tower along with the atomized slurry. The temp difference between air and slurry is at a maximum and heat is transferred to the slurry droplet at the maximum rate. Internal vaporization and puffing occurs more rapidly while the droplet is still more plastic. The result is a larger volume bead with the same amt of solids—therefore lower density. Depending upon the diam of the spray tower, the spray nozzles are positioned either in the center in the small towers or around the periphery in the larger towers. The number of nozzles also depends upon tower diam and capacity. Most important is to assure that the cross section of the tower is completely covered with the atomized slurry so that there can be intimate contact with the drying air and therefore maximum heat efficiency. Such precise positioning of the nozzles minimizes the possibility of the hot air channeling up through a section of the tower without being effectively used.

The hot air enters through a plenum chamber at the bottom of the cylindrical section of the tower, and is distributed equally around the periphery of the tower. By carefully designed inlets, the precise amt of swirl is induced to provide (1) maximum heat and mass transfer efficiency between drying air and atomized slurry and 2) extended droplet path to permit a maximum drying time so that the particle is dry when it hits the wall of the tower. This minimizes tower wall build-up. Too much swirl would actually result

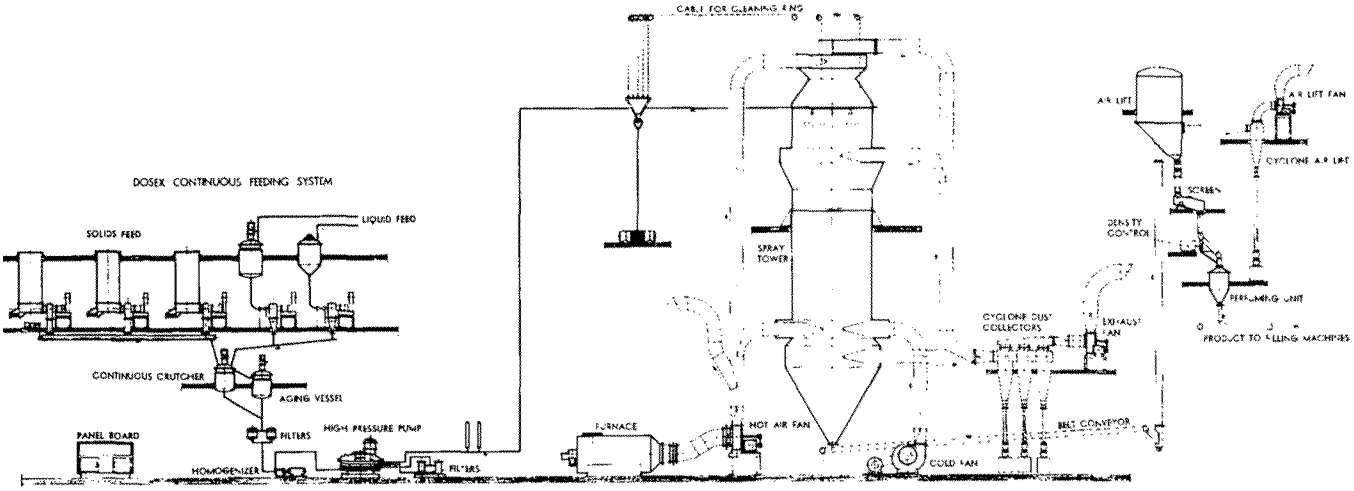


FIG. 4. Spray drying plant.

in throwing the sprayed slurry directly to the walls by centrifugal force, resulting in excessive wet build-up. A haphazard, random flow of air also results in excessive build-up on the walls.

In addition to the spray tower providing an efficient and economical means of drying detergent formulations, it also provides the product in a form that has been found highly acceptable to the consumer. The spherical particles have excellent flowability, quick solubility even in cold water, and are esthetically attractive. The product issuing from the bottom of the spray tower needs no further processing other than 1) a simple screening to remove oversize particles, and 2) addition of perfume before it can be placed directly into the carton.

Once all the sound principles for efficient drying have been designed into a spray tower and it is built to evaporate a certain quantity of water, its capacity is fixed and it now becomes the task of that tower to produce a product of specified density and moisture content.

As mentioned earlier, the density is of utmost importance since the finished package has a fixed volume into which must be placed a specified wt. The free volume above the level of powder in the carton must be kept to a minimum, since too much outage always creates an unfavorable reaction by the consumer. If density fluctations are such that low densities make it impossible to put the market wt into the carton, and high densities yield outages that are beyond those considered acceptable, one can appreciate the problems that are prevalent in many plants operating today. The actual dollar losses due to off-spec product, reworking of such material, scrap produced, filling machine inefficiency, etc. are high enough; but since many manufacturers actually overfill purposely to insure that they do not fall below the specified wt, the cost of poor density control becomes enormous. So, excellent density control has real dollar value.

The product moisture is also specified and the specification is generally slightly below the maximum moisture that the particular formula can tolerate. Obviously the closer the plant can maintain the product to the upper limit of the moisture specification the lower is the cost of production. For example, if a tower producing 25,000 lb/hr of powder at 10% moisture ±1.5% limit could raise the average moisture content to 11%, and if we assume 5,000 operating hr/year, and further assume a cost for the solids of \$0.10/lb, then the yearly savings amount to \$125,000. If moisture content goes beyond the upper limit allowable, it is possible that the product will become tacky and wet, causing blinding of the screens, fouling of the filling machines, and possibly caking of the cartoned product on the dealer's shelves. So, excellent moisture control also has real dollar value.

As we pointed out earlier, the preparation of the slurry contributes greatly to the steadiness of operation within the tower. A slurry of constant composition, constant viscosity, constant temp, etc., free of particles than can cause even partial blockage in the spray nozzles, will be atomized steadily and consistently to yield a fixed particle size distribution. With the close control attainable on the drying air rate and temp, product moisture and product density can then be controlled within very narrow limits.

The drying air is taken from the outside, passed through a furnace wherein either oil or gas is burned, and its temp is brought to, let us say, 650F. Automatic controls on the furnace very accurately maintain the desired temp of this drying air stream.

Countercurrent operation within the tower gives the most efficient utilization of the heat available in the drying air. For the heavy duty, high foam products satisfactory densities in the range of 0.25–0.40 can be easily obtained. However, with the heavy duty low foam products on the market today densities in the range of 0.30–0.40 are more difficult to obtain and co-current operation becomes the desirable method. The more rapid puffing of the droplets gives the lower densities.

In countercurrent operation, the inlet air may be at 650F and the temp of the exhaust air leaving at the top of the tower will be at approx 215–220F depending upon the moisture content of the finished product.

The slurry enters at say, 68% solids, and the sprayed powder which falls to the bottom of the tower will be at 10% moisture. As the product falls through the bottom cone of the tower, cool air passing in reduces the temp of the product to approx 140–160F.

The pressure within the tower is maintained slightly negative (ca. 1 in. of water), which eliminates the possibility of any dust getting into the atmosphere and making the area unfit for work. It also permits the in-leakage of cooling air at the bottom cone. This pressure is regulated by a damper on the exhaust fan outlet which is down stream of the dust collector. An endless belt conveys the powder from the botton of the tower and discharges it into the suction leg of an air lift. The product is brought up through the air lift and simultaneously cooled by the lifting air to approx 90–100F. Dropping out of the separator, the powder is screened to remove the oversized particles, passes through a continuous density indicator and controller, then enters a chamber in which perfume at the correct percentage is added, and finally flows into hoppers above the filling machine.

Since the density of the powder is critical, the automatic density controller plays a major role in the steady production of acceptable "on-spec" product. This unique device continuously removes a representative sample of the flowing powder, and leads it into a cylinder of fixed volume which is open on top and bottom. The flow of powder is through the cylinder, but the flow is sufficiently great enough to fill and slightly overflow the cylinder. The cylinder is on a balance and continuously weighed and therefore the density is continuously indicated. This density device controls the speed of the high pressure pump. If the density decreases below that desired, the speed of the high pressure pump is automatically increased, giving higher pumping rates and therefore higher spraying pressures. The higher spraying pressures will give increased product density. The moisture content normally will change negligibly, because the higher spraying pressure gives a finer atomization of the slurry which presents a greater surface area for drying. Thus, increased drying rates counterbalance to a major extent the increased slurry rate to the tower and moisture remains essentially steady.

If moisture content does vary it can be brought under control mainly by increase or decrease of the drying air temp, and increase or decrease of the drying air rate. Since the tower is primarily an evaporator, greater heat input will increase the drying, i.e. decrease the moisture content of the finished product and conversely lower heat input will result in higher product moisture content. The exhaust air from the top of the tower at a temp of approx 215–220F for countercurrent flow is passed through cyclone separators which removes the dust before discharging to the atmosphere. Also, the air used to air lift and cool the product is passed through dust separators prior to its exit from the plant. The total amt of dust collected usually runs ca. 4% of the total solids sprayed into the tower. The fine dust has the same composition as the finished product on a dry basis, of course, but its moisture content is lower, somewhere around 4–5%. The fines are fed back into the mixer at a uniform rate with the corresponding requirement of water to give a 68% solids slurry.

As indicated earlier, sprayed solids do build up on the tower walls. It can be appreciated that this build-up becomes practically bone dry because of its prolonged exposure to the hot drying air. After a time this build-up will intermittently drop off the walls and exit from the tower with the sprayed product. Since this "fall-off" material is of a different moisture content and density than the regular product, and therefore out of specification, it is undesirable and should be minimized. To accomplish this, the Ballestra towers are equipped with a cleaning ring which is permanently installed within the tower and extends around the periphery. At regular intervals this ring is slowly lowered from the top automatically, during tower operation, with its brushes or scrapper blades cleaning the tower walls, and letting the relatively small amt of the "tower wall material" blend in with the sprayed product. After the

(Continued on page 45)

• New Literature

Leeds & Northrup Co. has released a data sheet on Chromax II, which automatically measures the percentage concentration up to three components of a single complex gas stream in one minute. Data Sheet C3.4422. (4901 Stenton Ave., Philadelphia, Pa. 19144)

Beckman Instruments, Inc. describes their DB UV Spectrophotometer for complete color measurement in bulletin 7051. Their Reflectance Accessory converts the DB into a double-beam recording instrument for reflectance determinations, transmittance analyses and tristimulus measurements. (Tech. Information Sec., 2500 Harbor Blvd., Fullerton, Calif.)

Gelman Instrument Co. has issued a basic catalogue of their solutions, "Some Hows and Whys of Fine Filtration." It contains 32 pages of their answer on how to solve your filter problems. (P.O. Box 1448, Ann Arbor, Mich. 48106)

F & M Scientific Corp. has just published a new product bulletin 8100, describing the Series 810 Research Gas Chromatographs. They have also revised and enlarged the catalog of their full line of GC instruments, accessories and services. (41 and Starr Rd., Avondale, Pa. 19311)

New Brunswick Scientific Co., Inc. describes a 10¼ cf "shaker room," designed to fit into laboratories, in their Catalog C26. In only 40 x 20-in. floor space, they claim it is possible to incubate, refrigerate and illuminate a large number of aerobic or anaerobic shake cultures at one time. (P.O. Box 606, New Brunswick, N.J.)

Glycerine Producers Assoc. has issued a booklet, "Uses of Glycerine," second in the series; the 1963 edition being "Physical Properties of Glycerine and Its Solutions." (295 Madison Ave., New York 17, N.Y.)

Jarrell-Ash Co. has released a complete catalog of Chromatographic Supplies, covering columns, supports, phases, coated supports, absorbents, syringes, chart paper, recorders, integrators and miscellaneous supplies (Dept. G-N, 590 Lincoln St., Waltham, Mass.)

Koch Supplies Inc. has issued a new 140-page catalog of equipment and supplies for the meat and food industry—General Catalog No. 182, listing some 3,300 products and engineering services. (1411 West 29th St., Kansas City, Mo. 64108)

◆

Continuous Feeding . . . .

(Continued from page 40)

cleaning ring has reached the bottom of the tower, its direction automatically reverses and it returns to the top. The frequency of this cleaning cycle depends upon the formula being sprayed, and can be as often as 2–4 times shift. Thus the possibility of off-spec product significantly upsetting the main product stream is eliminated.

In many companies several formulas are sprayed, particularly blue and white products, and this cleaning ring permits the rapid and economical cleaning of the tower walls required for the changeover from one product to the other.

Conclusion

To summarize, we have described a manufacturing process for the continuous feeding, mixing and spray drying of detergent formulations.

Its process incorporates all the very latest developments which permit the steady and economical production of these high volume products. Because of the automatic features, labor requirements are minimum, product uniformity is greater and plant efficiencies are at a maximum.

[Received June 1, 1964—Accepted November 3, 1964]