Technical Highlights of the 53rd Spring Meeting . . . See page 9

Highlights of the 53rd Spring Meeting—II

Diversification Keynote of New Orleans Technical Program

(The second and last of a series, beginning in the June Issue, designed to offer the reader a detailed account of the multi-facited 53rd AOCS Spring Meeting)

Symposia Highlight Twelve-Session Series Sixty-Eight Papers Presented

Outstanding quality and diversification were the two basic objectives of R. T. O'Connor (photo above-right) as he prepared the Technical Program for the 53rd Annual Meeting of the American Oil Chemists' Society held at the Roosevelt Hotel, New Orleans, May 6–9. This review will attest to his success. Highlighting the program were two symposia: "Chemical Modification of Fats and Oils" and "Methodology of Fats and Oils." Topics such as new reaction, chromatography, analytical methods, hydrogenation, detergents, coatings, and biochemistry were discussed.

Sixty-eight papers were presented by authors from fifteen states, the District of Columbia, and Canada. These were contributed by eight academic institutions, eleven government agencies, two research institutions, and fifteen industrial laboratories. Presiding at the twelve technical sessions were H. P. DuPuy, W. A. Pons, Jr., J. J. Ganucheau, R. T. O'Connor, R. L. Holmes, R. Reiser, C. L. Hoffpauir, J. G. Hamilton, F. G. Dollear, and J. J. Spadaro.

Chemical Modification of Fats and Oils

A review of what is known concerning chemical reactions used to modify fats and oils for special uses was the subject of the first symposium. Maggiolo, The Welshbach Corporation, reviewed the history and stressed the growing importance of ozonolysis. Three major reactions involving ozonolysis are the splitting of double bonds, as the ozonolysis of oleic acid to give pelargonic and azelaic acids for synthetic fiber production; the reaction with tertiary amines to give amine oxides for the detergent field; and the reaction with aldehydes to produce peracids for use in epoxidation reactions. Ozonolysis is sufficiently refined for determination of ozone numbers on materials, the unsaturation of which cannot be ascertained reliably by bromine or iodine methods. T. W. Findley, Swift and Company, discussed the progress of epoxidation with particular reference to the commercial application of the *in situ* peroxy acid method. Approximately forty-five million pounds of epoxidized materials are used annually as stabilizers in polyvinyl chloride compounds, undoubtedly because of the scavenging effect on hydrochloric acid released during curing. Regular esters, premium grades of epoxidized soybean oil, epoxy fatty acid esters of certain alcohols and high oxirane content oils are used for this purpose. Many new uses for epoxidized oils are envisioned. E. R. Cousins, Southern Utilization Research and Development Division, pointed out that, although widely used commercially, the mechanism of hydrogenation is still a little understood phenomenon in fat chemistry. Variations of temperature, pressure, catalyst concentration and rate of agitation are only partly effective for achieving selectivity and minimizing isomer formation. Although certain solvents have a directional effect, final solution will result from the basic study of the mechanism of catalysis in hydrogenation.

According to N. O. V. Sonntag, National Dairy Products Corporation, halogenation is applied primarily to prepare

CHEMICAL MODIFICATION OF FATS AND OILS SYMPOSIUM, Technical Sessions A and D; H. P. DuPuy presiding. Seated: H. J. Harwood, A. Maggiolo, H. P. DuPuy, and L. A. Goldblatt. Standing: H. M. Teeter, E. R. Cousins, and T. W. Findley. (N. O. V. Sonntag not pictured.)

modified products. Overall halogen costs are a determining factor in the use of halogenation for analyses, modifications, and intermediate products. He visualizes increased use of fluorinated derivatives and the use of halogenation in modi-

fying fats, and continued use of halogenated fats and derivatives as plasticizers, flame-retardants, and fungicides. A promising future for fat and oil products in urethane polymers is forecast by L. A. Goldblatt, Western Utilization Research and Development Division. Although known for more than a hundred years, the urethane reaction has been used to prepare polymers for only a decade. Types of compounds used and new techniques for preparing urethanes were discussed. Applications include foams, elastomers, coatings, and even some medical and art uses. H. M. Teeter, Northern Utilization Research and Development Division, reviewed the preparation and properties of vinyl monomers which are derived from fats and oils and which are capable of polymerization or copolymerization. Included were examples of monomer preparations, applicable polymerization systems, and potential uses of the polymers. Since there are few new-type reactions of fats and fatty acids, H. J. Harwood, Durkee Famous Foods Division, confined his discussion to general reactions of fatty acids, little known reactions, and reactions for which new information might be available. Reactions of the hydrocarbon chain—substitution, addition, elimination, and cleavage—were emphasized.

Methodology of Fats and Oils

The second symposium featured discussions of some methods of analyses which are widely used in fats and oils research. T. K. Miwa, Northern Utilization Research and Development Division, discussed a technique for the determination of equivalent chain length values of fatty derivatives by gas-liquid chromatography. After being determined on known materials, such values are used in conjunction with other analyses to predict chain lengths and structures of unknowns. C. C. Litchfield, A and M College of Texas, reported on the gas-liquid chromatographic determination of relative amounts of *cis* and *trans* isomers in monoenes, dienes, conjugated dienes, and epoxidized materials. Two separate high resolution columns were used on pure *cis* and pure *trans* materials isomerized to various degrees. A "most-likely" configuration was assigned to the various peaks. D. Firestone, Food and Drug Administration, thought that while urea fractionation and chromatographic techniques have broad applications, micro-molecular distillation is still preferred for separating monomers, dimers, and polymers. Several stills used in the work were described.

J. W. Cook, same agency, stated that tolerances in food products had been established for about 125 pesticides and FDA has available methods of assay necessary to enforce these tolerances. Good progress has been made in methodology for chlorinated organic compounds in fat and oil products, but much must be done in developing methods for organophosphorous-type chemicals. Mary Carr Williams, A and M College of Texas, surveyed the various chemical, biological, and physical methods which have been used for the assay of essential fatty acids, pointing out the limitations and precautions to be exercised in applying the methods. Bioassay procedures appear best and simplest for total essential fatty acids in complex mixtures while gas-liquid chromatography (capillary column) appears best for *cis-trans* isomers of linoleic acid. In discussing procedures for the determination of triglyceride types and their isomers, R. J. VanderWal, Armour and Company, favored the oxidation-separation process of Youngs for glyceride structure of synthetic fats. If a 1,3 random 2, random pattern of distribution in natural fats is assumed, the pancreatic lipase method supplemented by gas chromatography shows promise for supplying data for calculation of specific triglycerides. (See this month's cover photograph, also reproduced on page 9, upper left. Caption on page 2.)

Progress in Chromatographic Techniques

The use of thin-layer chromatography plates for the assay of lipids labeled with carbon-14 and with tritium was described by F. L. Snyder, Oak Ridge Institute for Nuclear Studies. Excellent recovery and reliability were obtained and the technique required less than one hour, compared with seven to fifty days for autoradiograms. A comparison

TECHNICAL SESSION E; R. T. O'Connor presiding. Seated: F. R. Earle, R. T. O'Connor, F. L. Snyder, and C. F. Allen. Standing: R. Schofield, L. T. Black, J. C. Hamilton, R. J. Buswell, and E. P. Jones.

of the separations of soybean and brain lipids by column and by thin-layer chromatography was given by C. F. Allen, Pomona College. The latter method produced better resolution and separation. J. G. Hamilton, Tulane University, reported that 1,2 and 1,3-diglycerides appear to interconvert under conditions he employed for silica gel-impregnated glass paper and thin-layer chromatography of diolein and dipalmitic. He thinks that acyl migration is catalyzed by silica gel and that conclusions regarding the structure of glycerides must be drawn with caution. R. J. Buswell, Armour and Company, discussed a liquid-liquid partition chromatographic method for the determination of lactic acid in lactylated glycerides and in shortenings containing lactylated glycerides. The method is claimed to be simpler and less susceptible to interference than a colorimetric method.

L. T. Black, Northern Utilization Research and Development Division, explained that gas-liquid chromatography is quicker and more accurate than crystallization for determining cyclic fatty acids in cyclic monomers. L. D. Metcalfe, Armour Industrial Chemical Company, described types of columns and conditions used for gas-liquid chromatography of long chain quaternary ammonium compounds. Degradation of quaternaries on a strongly alkaline column produced identifiable tertiary amines. Quaternaries containing up to 38 carbon atoms have been identified. Two papers were presented by W. T. Roubal, Fish and Wildlife Service, on the fatty acid composition and unsaturated isomer structure of tuna oils as determined by gas-liquid chromatography. Oils from light and dark meats as well as raw, precooked and canned meats were analyzed.

New Analytical Methods

F. R. Earle, Northern Utilization Research and Development Division, reported the use of nuclear magnetic resonance for determining the oil content of intact seeds. The method, independent of functional groups, gives excellent correlation with the petroleum ether extraction method, and may be useful for screening new types of oilseeds. R. A. Greff, Jr., Continental Oil Company, stated that high resolution NMR has been used to measure the ratio of hydrophile to hydrophobe for estimating the composition of nonionic surfactants. The method was used to characterize commercial polyethylene oxide condensates and may be applied to formulated detergent products.

In two separate papers, C. R. Schofield and E. P. Jones, Northern Utilization Research and Development Division, reported on counter-current distribution for analyses of commercial hydrogenated soybean oils and fractionation of isomeric esters. Monoenes and trienes are separated by distribution between acetonitrile and petroleum ether and geometric isomer between 0.2M silver nitrate in 90 per cent methanol and petroleum ether. The *trans* content of conventional shortening, containing a comparable solids content, was twice that of a winterized oil, which probably accounts for higher melting characteristics of the shortening.

J. Velasco, Agricultural Marketing Service, proposed a

rapid electrical conductivity method for determining free fatty acid compositions as low as 0.005 per cent in oilseeds. An ammonium-butanol electrolyte reacts with the acids to form stable ammonium salts in solution, the conductance of which is measured.

Physical Properties

V. P. Kuceski, C. P. Hall Company of Illinois, described a new group of disubstituted amides which possess unusual physical and chemical properties. Recently commercially available, these compounds decrease the viscosity of either nonpolar compounds or of compounds in nonpolar solvents. Rubber, cosmetics, petroleum, and even salt water conversion are some areas of use for the N,N-dimethyl fatty acid amides. R. R. Mod, Southern Utilization Research and Development Division, in presenting data on the freezing point behavior of the ternary salt pair system, the morpholine and 2,2′-dipyridylamine salts of stearic and palmitic acids, stated that this fundamental data had practical application in purifying the morpholide derivative of palmitic acid.

TECHNICAL SESSION C; J. J. Ganucheau presiding. Left to right: J. J. Ganucheau, W. M. Linfield, T. H. Liddicoet, K. L. Johnson, and W. C. Powe.

Detergents and Surfactants

The detergent activity and fabric softening properties of amphoteric surfactants were described by W. M. Linfield, Armour and Company. The compounds had a quarternary ammonium group, and anion, and either one or two fat-derived long chains. The effects of the constituents on detergency, fabric softening, and compatibility were investigated. W. C. Powe, Whirlpool Corporation Research Laboratories, outlined a gravimetric method for removing and determining fatty soil in cotton textiles in aqueous detergent solutions.

T. H. Liddicoet, California Research Corporation, pointed out that the method of sulfonation and the quality of the alkylbenzene affect the alkylbenzene sulfonates. Some engineering factors relating to the continuous sulfonation of alkylbenzenes with sulfur trioxide were discussed by J. W. McCutcheon, John W. McCutcheon, Inc. A new class of nonionic detergents is made by reacting epoxidized soybean and linseed oils with polyoxyethylene glycols in the presence of boron trifluoride, according to K. L. Johnson, Swift and Company. The products have molecular weights ranging from 2000 to 6000. Possible applications are as agricultural chemical emulsifiers, in textile processing, as aqueous and nonaqueous lubricants, and as industrial emulsifiers.

Emulsifiers

F. H. Otey, Northern Utilization Research and Development Division, described the preparation and properties of glycoside polyoxyethylene ethers. The products possess various viscosities and emulsifying action, the unsaturated esters of the polyethers forming insoluble films on drying. J. J. Geminder, Chas. Pfizer and Company, Inc., discussed the use of stearyl monoglyceridyl citrate as an emulsifier enhancer in shortenings, margarines, and whipped toppings. This additive does not appreciably lower smoke point and reduces peroxide formation. J. L. White, Southern Utilization Research and Development Division, described the preparation of stable oil/water suspensions containing a minimum of stabilizer and up to 10 per cent triglyceride. Three dispersants are necessary to prepare suspensions which are stable for at least one month at room temperature.

TECHNICAL SESSION F; R. L. Holmes presiding. Seated: J. L. White, H. Roth, and J. H. Geminder. Standing: R. R. Mod, G. R. Riser, F. H. Otey, and R. L. Holmes.

New Reactions and Derivatives

R. O. Feuge, Southern Utilization Research and Development Division, described a procedure for the direct esterification of mono- and diglycerides with fatty acids by removing water of esterification with vaporizing aliphatic hydrocarbon. Little or no ester-ester interchange occurred; however, intraesterification was extensive. D. E. Anders, Northern Utilization Research and Development Division, reported a minimum of chain degradation and yields of over 90 per cent omega—formyl esters by ozonization of fatty acids containing nine, eleven, and thirteen carbon atoms in a

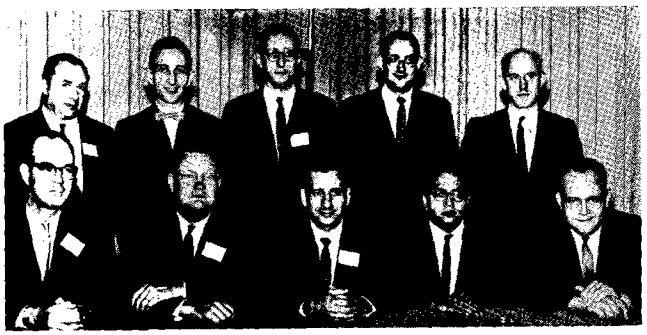
methanol-pyridine solvent. The application of a new phosphorylation reaction to prepare a series of unsaturated fatty derivatives with a phosphorus-carbon bond was discussed by E. Jungermann, Armour and Company. In most cases the phosphorous trichloride-aluminum chloride reagent acts only on the central double bond.

G. Maerker, Eastern Utilization Research and Development Division, presented data on the acid-catalyzed hydration of glycidyl stearate to monostearin in organic solvents under a variety of conditions. Most favorable of these yielded 85 per cent monostearin. The formation of distearin may result from an alcoholysis involving monostearin and glycidyl stearate. T. H. Applewhite, Western Utilization Research and Development Division, described a simple, rapid procedure for synthesizing amides of ricinoleic acid by use of mixed carboxylic-carbonic anhydrides. The procedure uses mild conditions and produces high yields. Compared with conventional ester aminolysis in methanol solution, no appreciable steric hinderance is observed.

Hydrogenation

E. R. Cousins, Southern Utilization Research and Development Division, showed that Raney nickel catalysts deactivated by acetaldehyde can be reactivated to a greater degree of activity than possessed originally by treatment with acetic acid. A. E. Johnson, Northern Utilization Research and Development Division, illustrated the effect of catalyst concentration and temperature on selectivity, *trans* formation and rate of hydrogenation for a commercial electrolytic nickel catalyst. Temperature ranged from 70–230C and nickel concentration from 0.05–10 percent. Rate depends on temperature and concentration, *trans* formation is a function of temperature, and selectivity is influenced by neither.

R. E. Beal, Northern Utilization Research and Development Division, described the design and use of a continuous reactor for the production of cyclic acids from linseed oil. At 240C, twelve minutes are required, compared with ninety minutes for a batch reactor. R. A. Eisenhaur, of the same



TECHNICAL SESSION L; J. J. Spadaro presiding. Seated: E. R. Cousins, A. E. Johnston, J. H. Spadaro, V. V. R. Subrahmanyam, and R. A. Eisenhauer. Standing: J. W. McCutcheon, G. Maerker, F. Scholnick, R. A. Barford, and R. E. Beal.

laboratory, outlined the effect of solvent to fatty acid ratio, excess catalyst, temperature, headspace gas, and gas pressure. Cyclic fatty acid yields in excess of 84 per cent based on linolenic acid are obtained.

Improved Processing Methods

W. A. Pons, Jr., Southern Utilization Research and Development Division, presented an evaluation of the efficiency of modified alumina adsorbents for bleaching cottonseed oils. Sulfurous acid-treated alumina has a pronounced catalytic effect, the reduction of Halphen response being complete after a 30-min bleaching period at 225C. Oil treated with normal alumina for 3 hr was still Halphen positive. Paul H. Eaves, same laboratory, described a pilot plant batch bleach-

TECHNICAL SESSION B; W. A. Pons, Jr. presiding. Left to right: W. A. Pons, Jr., L. D. Metcalfe, R. A. Greff, Jr., and J. Velasco.

ing process which consists of thorough mixing of refined oil and activated alumina, de-aerating under reduced pressure, agitating and heating to 440F for 20–30 min, cooling to 180–200F, and separating and restoring spent alumina. A 20 min treatment with 4 per cent of 0-2 micron alumina has an optimum bleaching effect. K. M. Decossas, same laboratory, gave estimates of the investment and operating cost for bleaching off-colored bleach-resistant cottonseed oils by the described process in new and in converted plants. The most economical alumina bleaching with restoration of spent alumina for reuse should cost 0.4 cent per pound in a large plant.

An extraction process for cottonseed which utilizes a mixture of hexane, acetone and water, either homogeneous or non-homogeneous, was described by E. A. Gastrock, same laboratory. The process was designed to improve the nutri-

TECHNICAL SESSION K; F. G. Dollear presiding. Left to right: J. K. Sikes, W. A. Pons, Jr., K. M. Decossas, P. H. Eaves, F. G. Dollear, E. A. Gastrock, W. T. Roubal, and R. O. Austin.

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tive value of extracted meal by avoiding destruction of *epsilon* -amino-free lysine and by removing most of the gossypol with the oil. J. K. Sikes, Plains Cooperative Oil Mill, reported on studies of the breakdown of urea when in contact with cottonseed, as indicated by the evolution of ammonia. Additional desolventizing toasting before processing the meal minimizes the breakdown with no decrease in nutritive value. The enzyme urease was suggested as the causative agent.

Coatings, Plastics, and Plasticizers

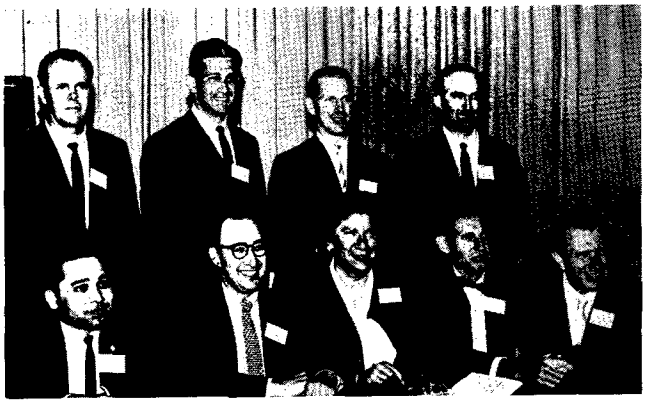
R. O. Austin, Pan American Tung Research and Development League, indicated that benzoyl peroxide is a suitable catalyst for ether cross-linking of triglycerides of tung oil by air blowing at temperatures up to 120C. Reactions occur at the 13–14 position, and some hydroxyl groups and oxirane oxygen are obtained. F. C. Magne, Southern Utilization Research and Development Division, reported on the testing of fatty acid diester-amides containing one long and one short chain fatty acid as plasticizers for vinyl chloride copolymer. Frank Scholnick, Eastern Utilization Research and Development Division, described the testing of phthalic anhydride cured monomers prepared by epoxidizing a series of unsaturated esters. F. W. Quackenbush, Purdue University, compared the rates of *trans*-isomerization and polymerization on selenium-catalyzed isomerization of ethyl linoleate and safflower oil at temperatures from 180 to 235C. G. R. Riser, Eastern Utilization Research and Development Division, outlined the heat and light stabilizing properties of 1–3 per cent of C_{18} acids and their salts in polyvinyl chloride plastic sheets plasticized with dioctyl phthalate. Steric and oleic acids are ineffective, whereas their cadmium and barium salts are. Cadmium epoxyoleate is the best additive.

Deterioration

H. Roth, DCA Food Industries, Inc., explained that air contact deteriorates fats held at high temperatures for extended periods of time. Removal of air from contact with fat almost completely inhibits deterioration even at frying temperatures. Silicic acid chromatography was used by R. Sahasrabudhe, Food and Drug Directorate, Canada, to fractionate the degradation products resulting from heating corn oil at 200C. for 48 hours. Nine fractions of varying complexity were observed and the paths of degradation elucidated. R. N. Anderson, General Mills, discussed the oxidation of butylated hydroxy anisole and butylated hydroxy toluene in breakfast cereals under various conditions. Disappearance of the antioxidants was determined by gas liquid chromatography and organoleptic evaluation and correlated with the development of peroxides in the cereal lipids.

Biochemistry

D. Kritchevsky, The Wistar Institute, said that the effects of thyroactive compounds on the oxidation of cholesterol and sodium octanoate are not enhanced by preparations of rat liver mitochrondira. Liver mitochrondria, isolated from rats receiving long term injections of four thyroxine-line compounds, and dinitrophenol indicated little or no alteration in cholesterol oxidation but an enhancement of octanoate oxidation. R. B. Alfin-Slater, University of California, showed that cuttlefish liver oil retards the growth of rats and fails to supply essential fatty acids necessary for regulation of cholesterol metabolism. H. Mohrhauer, The Hormel Institute, presented extensive quantitative data which showed that linoleate, linolenate, and arachidonate fed at different levels affect the composition of rat liver lipids, arachidonate being the most potent. A perfusion technique was used by Murray Heimberg, Vanderbilt University, to study liver damage induced in rats by carbon tetrachloride. Livers from normal rats continuously release triglycerides to the perfusion medium, while those from treated rats do not. Uptake of tripalmitin by the livers of treated rats continues at a normal rate, however. H. Kaunitz, Columbia University, described experiments on rats fed diets containing various coconut oil triglycerides and a polymeric



TECHNICAL SESSION I; J. G. Hamilton presiding. Seated: M. R. Sahasrabudhe, D. Kritchevsky, Roslyn B. Alfin-Slater, J. G. Hamilton, and H. Kaunitz. Standing: R. N. Anderson, M. Heimberg, H. Mohrhauer, and A. J. Sheppard.

fraction from oxidized methyl linolenate. An anti-kidney serum, which induced kidney lesions, was injected. The fatty acid compositions of the kidneys were related to dietary fats but not altered by the disease—evidently a rearrangement of the lipids results from the serum. A. J. Sheppard, Food and Drug Administration, found a modified Bloor method to be superior to the Bailey-Walker method for extracting rat liver lipids for gas-liquid chromatography.

New Oilseed Crops

T. K. Miwa, Northern Utilization Research and Development Division, described the preparation, yield, and physical properties of the C_{20} and C_{22} wax esters obtained from *Limnanthes douglassii, Crambe abyssinica,* and *Lunaria annua.* R. A. Barford, Eastern Division, reported on the

TECHNICAL SESSION H; C. L. Hoffpauir presiding. Seated: Lida L. Placek, T. K. Miwa, C. L. Hoffpauir, E. Jungermann, and R. O. Feuge. Standing: T. H. Applewhite and R. L. Holmes.

preparation and purification of methyl epoxyoleate of 97 per cent purity by transmethylation of *Vernonia anthelmintica* seed oil and subsequent low temperature crystallization. Lida L. Placek, Southern Division, told of the preparation of some nitrogen containing derivatives of petroselinic acid and stated that the derivatives have lower melting points than the corresponding oleic acid product. R. L. Holmes, same Division, outlined a procedure for preparing dodecylamine and 6-aminohexanoic acid from petroselinic acid.

Cite Fine Work of Reporters

Appreciation is expressed to the reporters from the Southern Utilization Research and Development Division who covered the technical sessions; and to Audrey T. Gros, who compiled and produced this review. Reporting the concurrent sessions with accuracy and continuity would have been impossible without their assistance.

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