

Committee Meets in N. Y.—Releases Firm Plans

A meeting of the Short Course Committee on "Advances in Soaps and Detergents" under the chairmanship of Eric Jungermann, Armour and Company, was held on January 23, 1963, in New York City. Committee members present were F. D. Snell, F. D. Snell, Inc., L. E. Netherton, Victor Chemical Company, E. Scott Pattison, Soap and Detergent Association, J. F. Gerecht, Colgate-Palmolive Co., R. D. Swisher, representing Ray Liss, Monsanto Chemical Company, and L. Garrison, Jefferson Chemical Company. Unable to attend were A. J. Stirton, Eastern Regional Laboratories, M. E. Ginn, Armour and Company, and J. C. Harris, Monsanto Chemical Company.

Princeton Inn Room and Board Included in \$95 Total

A fee of \$95 was set for the Course to cover registration fee, hotel room and board at the Princeton Inn (pictured above), Princeton, New Jersey. The Course, held under the auspices of the Education Committee of the American Oil Chemists' Society, is scheduled to be held from June 23-26, 1963. The above fee will cover all gratuities and meals, and will include a warm buffet get-together on Sunday evening, June 23.

Gerecht Local Chairman

J. F. Gerecht, Colgate-Palmolive Company, has been appointed local chairman. He will be in charge of making final arrangements and coordination of all activities of the Short Course with the Princeton Inn. The Princeton Inn is a modern, air-conditioned hotel with spacious grounds and a golf course. Twin bedded rooms will be provided,

and registrants are asked to indicate their choice of room-mates, if any. A slightly higher fee will be charged for people requiring private rooms.

Program Broad—Emphasis on Latest Developments

A wide range of topics of general interest to workers in the field has been scheduled for presentation during the Course. The program will stress changes in the Soap and Detergent Industry brought about by new technology and processes. New chemicals and intermediates will be discussed. Emphasis will be away from familiar review-type discussions, and speakers will stress new ideas, new chemicals, new raw materials, and the resultant process and industry problems.

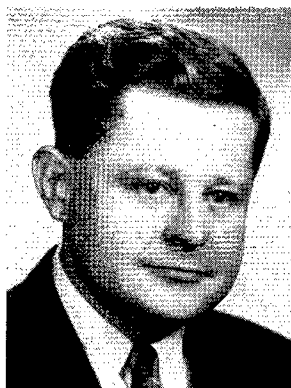
Keller, Pattison to Speak at Dinners

The Committee was fortunate in securing two dynamic after-dinner speakers: G. M. Keller, Vice President, Armour and Company, will give one of the after-dinner talks on "Marketing's Responsibility to Research." E. S. Pattison, Division Manager of the Soap and Detergent Association will be the second after-dinner speaker.

New Firmenich & Co. Film to be Shown

Special arrangements have been made to show a movie produced by Firmenich & Co., called "Challenging Nature's Chemistry." The movie deals with the application of modern research techniques to the elucidation of chemical structures of complicated natural products, such as essential perfume oils.

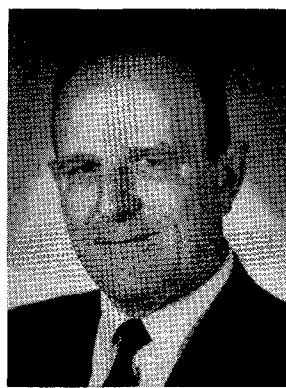
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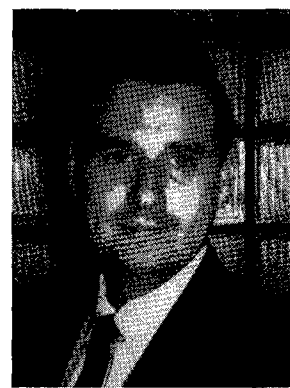
Eric Jungermann



L. J. Garrison



J. F. Gerecht



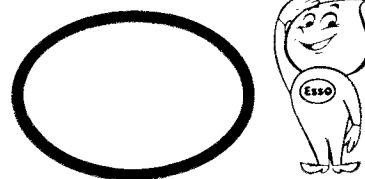
M. E. Ginn



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Report of the Spectroscopy Committee, 1961-62

THE SPECTROSCOPY COMMITTEE held its annual meeting during the American Oil Chemists' Society 53rd Annual Convention at the Roosevelt Hotel, New Orleans, La., May 7-9, 1962. Eight of the twelve members of the Committee were present, and two others were represented by duly accredited alternates at the session held in the Southern Pine Room Monday morning, May 7.

Subcommittee to Investigate Methods for Preparing Methyl Esters from Long-Chain Fatty Acids

The Subcommittee of the Spectroscopy Committee, appointed to study various methods for converting long-chain fatty acids to their methyl esters chiefly for the purposes of spectrophotometric analyses or for gas-liquid phase chromatography, and to recommend a specific procedure for adoption by the Society, met during this Session. This portion of the meeting of the Spectroscopy Committee was presided over by J. R. Chipault, Chairman of the Subcommittee. Results of the first collaborative testing by the Subcommittee were reported briefly. Samples of soybean oil fatty acids were esterified using methanol-sulfuric acid, diazomethane, boron trifluoride, and dimethoxypropane in each of six collaborating laboratories, and the 24 ester samples were examined for yield, free fatty acid content, and composition as determined by gas-liquid chromatography (GLC).

As all GLC analyses have not been completed, the data has not been submitted to detailed statistical analysis. Results of the collaborative work are shown in Table I. cursory examination of these data indicate that from the standpoint of overall yield of esters only, any of the methods appears to be satisfactory. The free fatty acid analysis showed that the boron trifluoride procedure yielded esters with the lowest content of fatty acids and that it was followed closely by the methanol-sulfuric acid method. Of the eleven samples prepared by these two methods, none had a free fatty acid content exceeding 1%. On the other hand, in some laboratories, diazomethane and the dimethoxypropane methods yielded esters with excessively high free fatty acid content. A non-critical examination of the GLC analyses available showed no obvious differences in composition of the esters prepared by the different procedures.

It is the general opinion of the Subcommittee that none of the methods considered should give difficulties with isomerization of polyunsaturated compounds, although this had not been established with certainty for the highly unsaturated fatty acids of fish oils. It is of interest to note when each collaborator was asked to indicate his preference among the four procedures, two selected the methanol-sulfuric acid procedure and four the boron trifluoride technique.

It was pointed out that the particular sample of free fatty acid used, appeared to contain some extraneous materials which interfered with the diazomethane esterification. In many cases, the esters prepared with diazomethane were jelled. Four of the six laboratories preparing the esters indicated dissatisfaction with the particular gaseous diazomethane method used.

Seymore Goldwasser remarked that in his experience, diesters prepared by the diazomethane procedure showed a decrease in polyunsaturates when they were allowed to stand at room temperature for several days while sealed under vacuum. R. R. Allen said that he had observed the same thing with diazomethane esters stored at 40F.

R. T. O'Connor mentioned that one of the objections that has been given to the methanol-sulfuric acid method was that it changed the *trans* content of the acids. Dr. Goldwasser replied that his laboratory had tested the esterification method used here and found that none of them gave rise to isomers of polyunsaturated acids. David Firestone said that he found no increase in isolated *trans* when using the methanol-sulfuric acid method, and that

the method had been used in his laboratory also for direct methanolysis of glycerides by using some benzene or other hydrocarbon solvent to solubilize the triglycerides.

L. A. Goldblatt inquired about the possible fate of hydroxyl and epoxy groups during the esterification procedures. It was agreed that acids containing hydroxyl groups such as ricinoleic acid were difficult to esterify, without also esterifying some of the hydroxyl groups, and that epoxy groups would be destroyed during esterification by acid catalyzed procedures.

It was agreed that the Subcommittee in future cooperative studies should investigate the possible changes in the *trans* content and the hydroxyl content of fatty acids esterified by the methanol-sulfuric acid and the boron trifluoride methods. The methanol-sulfuric acid method would be applied directly to triglycerides as well as to free fatty acids.

Collaborative Testing—Preparation of Secondary Standards

Secondary standards required to calibrate infrared spectrophotometers for the determination of isolated *trans* isomers in methyl esters or triglycerides by the AOCS

(Continued on page 32)

TABLE I
Spectroscopy Committee
Sub-Committee for Preparation of Methyl Esters

Esterification of Soybean Fatty Acids—Trial No. 1					
Laboratory	Analysis	Esterification Procedure			
		MEOH-H ₂ SO ₄	CH ₂ N ₂	BF ₃	Pr(OMe) ₂
1	Yield % FFA Time (hr)	102.4 0.4 1	94.9 5.9 1	92.3 0.2 1	100.8 3.4 1
2	Yield % FFA Time (hr)	101.5 0.6 3	101.0 0.4 2½	103.0 0.2 2	101.5 51.5 3
	C ₁₆	10.58±0.43	11.05±0.45	10.38±0.42	11.76±0.47
	C ₁₈	4.59±0.05	5.12±0.05	4.42±0.05	4.77±0.05
	C ₁₈ , Δ1	29.45±0.41	28.92±0.39	29.40±0.41	29.25±0.38
	C ₁₈ , Δ2	47.29±0.62	47.78±0.65	48.76±0.64	47.71±0.67
	C ₁₈ , Δ3	8.09±0.27	7.14±0.24	7.05±0.24	6.53±0.23
	C ₂₀	tr.	tr.	tr.	tr.
3	Yield % FFA Time (hr)	101.2 1.0 2	98.5 2½	100.0 0.3 1¾	101.6 0.2 1¾
4	Yield % FFA Time (hr)	104.3 0.8 2¾	103.9 10.2 1¾	103.6 1½	104.3 4.2 2¾
	C ₁₆	11.41±0.46	10.85±0.44	(2)	10.99±0.45
	C ₁₈	5.03±0.05	4.52±0.05		4.36±0.04
	C ₁₈ , Δ1	29.27±0.39	29.53±0.40		29.49±0.41
	C ₁₈ , Δ2	47.28±0.66	48.60±0.66		47.89±0.64
	C ₁₈ , Δ3	7.02±0.24	6.51±0.22		7.28±0.25
	C ₂₀	tr.	tr.		tr.
5	Yield % FFA Time (hr)	102.2 0.8 2¾	101.5 1.6 2	100.0 0.2 1½	102.0 2.2 2¾
	C ₁₆	10.74±0.43	10.95±0.45	10.36±0.42	10.15±0.45
	C ₁₈	4.83±0.05	4.52±0.05	4.71±0.04	4.55±0.05
	C ₁₈ , Δ1	29.40±0.40	28.46±0.40	29.41±0.40	29.55±0.40
	C ₁₈ , Δ2	48.51±0.66	48.74±0.64	48.77±0.65	48.26±0.67
	C ₁₈ , Δ3	6.52±0.22	7.34±0.25	6.75±0.23	6.51±0.23
	C ₂₀	tr.	tr.		tr.
6	Yield % FFA Time (hr)	101.4 0.5 2½	101.7 0.2 2	102.8 0.3 1½	101.4 0.3 2½

(1) This sample was gelled and was not analyzed by GLC.

(2) Sample lost through breakage.

Comments of collaborators:

No. 1—This sample of fatty acids appeared to contain extraneous material which gave some difficulty with the CH₂N₂ and BF₃ methods. For this particular sample only the MEOH-H₂SO₄ method is preferred.

Normally prefer a method using CH₂N₂ in ether.

No. 2—Carbitol carried over during CH₂N₂ procedure. Pr(OMe)₂ method gives a product difficult to clear.

No. 3—Difficulties encountered with diazomethane procedure. If CH₂N₂ is to be used, another method would be preferable.

No. 4—None.

No. 5—Equipment for CH₂N₂ method is cumbersome and not satisfactory for a large number of samples.

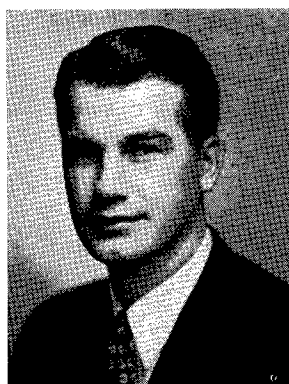
No. 6—CH₂N₂ procedure not satisfactory because of carry-over of carbitol and water. Method of McKay is better.

No. 1 and 3 preferred MEOH-H₂SO₄ method.

No. 2, 4, 5, and 6 preferred BF₃ method.



R. L. Liss



L. E. Netherton

Monday: "New Chemicals and New Processes"

The sessions on Monday will be devoted to "New Chemicals and New Processes." A. L. Schulerud, Colgate-Palmolive, will discuss several new continuous saponification processes now being used in many parts of the world. In particular, he will compare the De Laval Centripure Process, the Mazzoni Process, Meccaniche Moderne Process, and the Sharples Process. Soap making from fatty acids will also be discussed. J. P. Mallee, the Stokes Company, Philadelphia, will discuss modern tableting techniques and the equipment needed for them. Tableted detergents had a phenomenal growth in this country during the last two years, and various aspects of this subject, which is of particular interest to the participants of the Course, will be covered by several speakers.

Italian Scientist to Compare U. S., European Processes

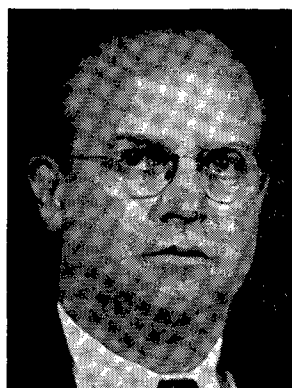
An international note is being added to the Monday session with the participation of Dr. Ballestra of Italy, who will discuss continuous sulfonation processes both in Europe and this country.

Several speakers will discuss some of the important new raw materials now becoming available. T. P. Matson, Continental Oil, will discuss the chemistry and evaluation of long-chain alcohol derivatives. D. B. Lake, du Pont de Nemour Co., will give a comprehensive talk on amine oxides and their uses in detergent products. The field of organo-phosphorus detergents will be covered by R. Cooper, Stauffer Chemical Co., while L. E. Netherton, Victor Chemical Company will discuss inorganic builders for the detergent emphasis with special emphasis on their application in the tableting field.

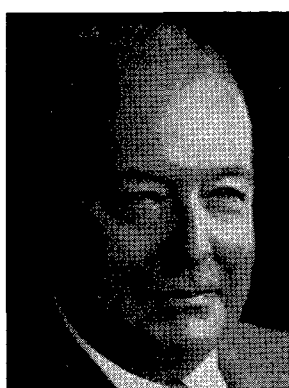
Tuesday: Biodegradability Symposium, Plant Trips

Tuesday morning, June 25, 1963, a special program has been set up to deal with the very important subject of biodegradability. Topics like the nation's water pollution

(Continued on page 21)



A. J. Stirton



F. D. Snell

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(Continued from page 8)

Preliminary studies were made on the rosin acid fraction of tall oil fatty acids using several enrichment techniques such as column chromatography, preferential esterification, amine salt formation and urea adduct techniques. A combination of preferential esterification and silicic acid column chromatography was used to show the effect of maleic anhydride modification on tall oil fatty acids. Quantitative determinations were carried out on various mixtures of tall oil fatty acids and rosin samples which were unmodified and modified with maleic anhydride.

THE REACTION OF ACETYL NITRATE WITH ALCOHOL DERIVATIVES OF FATTY ACIDS

D. C. Malins, J. C. Wekell, and C. R. Houle, Bureau of Commercial Fisheries Technological Laboratory, Seattle 2, Wash.

Interest has increased recently in the preparation and properties of organic nitrates, and the chemistry of these compounds has become an important part of the field of organic chemistry. The use of nitrate esters for the preparation of a wide variety of important derivatives has been the subject of several recent reviews.

In the present work, acetyl nitrate was found to be highly effective for the nitration of various classes of alcohol derivatives. This reagent was shown to react with fatty alcohols, hydroxy esters, and α -glycols to give good yields of nitrate derivatives. The reaction was applied to the synthesis of 1-octadecyl nitrate, methyl 2-nitrate-octadecanoate, methyl 12-nitrate-octadecanoate, and 18,19-dinitrate-hexatriacontane. In addition, mixed alkyl nitrates and nitrate esters were prepared by this method from commercially available marine oils.

The analysis of long-chain nitrate derivatives of fatty acids by infrared spectroscopy and thin-layer chromatography will also be presented.

EFFICIENT PRODUCTION OF BIOSYNTHETICALLY LABELED FATTY ACIDS

H. J. Dutton and T. L. Mounts, Northern Regional Research Laboratory, Peoria, Ill.

Pure fatty acids randomly labeled with C^{14} are in demand for those biomedical, biochemical, and organic chemical experiments in which carboxy-labeled fatty acids are inapplicable or unavailable. Prior methods of fatty acid biosynthesis have frequently involved continuous culture at sublethal levels of radiation. These have yielded low specific activity lipids along with undesired, labeled cell constituents. By growing a plant to seed setting stage under field conditions and then by exposing it to a single large dosage of $C^{14}O_2$, such as would be lethal over extended periods, high specific activities and efficient recovery of C^{14} in the seed lipids and meal are observed. For example, a perilla plant irradiated in the presence of 0.5 mc of $C^{14}O_2$ produced 1 g of seeds containing 371 mg of lipids and 614 mg of meal. The methyl esters had a specific activity of 0.6 μ c/mg and incorporated 37% of the C^{14} used. The meal (sp. act. 0.134 μ c/mg) accounted for an additional 16.5%, and the residual roots, leaf, and stem (sp. act. 0.05 μ c/mg) 34.4%. Reversed-phase chromatographic procedures for separating the 314 mg of methyl esters and methods for establishing the randomness of labeling are described.

Plant Production and Control

ISOMERIZATION DURING HYDROGENATION. V. METHYL CIS 6-, CIS 9-, AND CIS 12-OCTADECANOATES

R. R. Allen, Anderson, Clayton & Co., Foods Division, Sherman, Tex.

It has been generally believed the position of a double bond in relation to the carboxyl group in long chain fatty acids affect its rate of hydrogenation.

By the hydrogenation of a mixture of three isomers, Methyl Cis 6-, Cis 9-, and Cis 12-octadecanoates followed by analysis for the residual double bonds, no appreciable difference in rates of hydrogenation could be demonstrated.

Although in some samples hydrogenated under high isomerization conditions "excess" 9 octadecenoate was found, this can be explained by the shift of both the 6 and 12 monoenes into the 9 position.

No evidence was found for the shift of a double bond into the 17 position indicating the terminal methyl group does not participate in any dehydrogenation reaction with the catalyst.

PILOT PLANT PREPARATION OF DEFATTED PEANUTS

J. Pominski, E. L. Patton, and J. J. Spadaro, Southern Regional Research Laboratory, New Orleans, La.

Interest in defatted peanuts is due to several factors: lower calorie value; possible increase in shelf-life by minimizing oil rancidity, possible use by hemophiliacs to control bleeding; and development of a new product to increase utilization of peanuts. Based on previously conducted laboratory work, pilot plant runs were conducted to prepare large amounts of materials for taste and appearance evaluation, to obtain pilot plant processing data for cost calculations, to investigate practical methods of desolvation of extracted peanuts, to develop a method for salting the defatted peanuts, and to study packaging.

Fully roasted and one-half roasted batches of Virginia peanuts were extracted with hexane at room temperature for various periods of time, and oil losses determined. It was found that fully roasted peanuts with 81% of the oil removed had the best appearance. Low rates of extraction indicate that large scale processing would be a batch method.

The extracted peanuts were desolvated for various periods of time and temperature in both forced draft and vacuum ovens. It was found that drying at a low initial temperature prior to a final high temperature of drying appears to give a better tasting peanut, especially when a forced draft oven is used.

Defatted, desolvated peanuts were salted by either dipping in saturated salt solution at room temperature or preferably by dipping in water and sprinkling with salt. The wet peanuts were oven dried.

Packaging of defatted peanuts (81% oil removed) in metal cans, in either vacuum or in an atmosphere of nitrogen containing less than 2% oxygen proved satisfactory even after one year storage time. In flexible cellophane-type package, defatted peanuts tended to pick up excessive moisture within 30 days.

HOMOGENEOUS CATALYTIC HYDROGENATION OF UNSATURATED FATS: METAL CARBONYLS

E. N. Frankel, Helen VenHorst, E. P. Jones, and H. J. Dutton, Northern Regional Research Laboratory, Peoria, Ill.

Hydrogenation of unsaturated fats with soluble catalysts is being investigated with two objectives: 1) determining whether selectivity toward linolenate can be achieved, and 2) obtaining a better understanding of the mechanism of heterogeneous catalytic hydrogenation.

Iron pentacarbonyl is an effective homogeneous catalyst for the reduction of polyunsaturated fats. Hydrogenation of soybean oil and its methyl esters has been achieved at 180°C, hydrogen pressures of 100 to 1,000 psi, and 0.05 to 0.5 molar concentrations of catalyst. Analyses of partially reduced products showed considerable isomerization of double bonds, reduction with no particular selectivity toward linolenate, accumulation of *cis-trans*- and *trans,trans*-conjugated dienes and isolated *trans* monoenes, and little or no increase in saturates. Reduction and isomerization of double bonds in soybean oil methyl esters were also effectively catalyzed by dimanganese decacarbonyl.

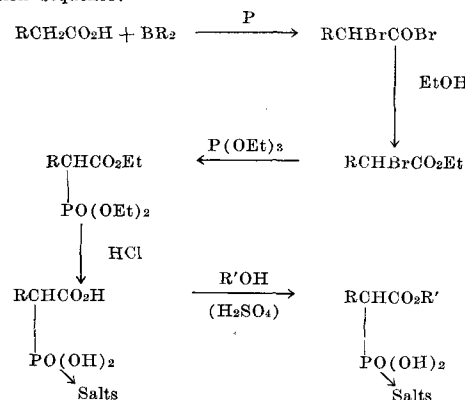
The course of the homogeneous hydrogenation catalyzed by metal carbonyls appears very similar to the heterogeneous catalytic reaction. Metal carbonyls are well known for their isomerizing effects on olefins and for their ability to form stable π -complexes with double bonds of conjugated polyenes and monoenes. These homogeneous complexes provide suitable model systems to study the type of attachment between transition metals and double bonds occurring in chemisorbed fats during heterogeneous catalytic hydrogenation.

Detergents and Surfactants

LONG CHAIN α -PHOSPHONO FATTY ACIDS, SALTS AND ESTERS

E. W. Maurer, A. J. Stirton, Waldo C. Ault and J. K. Weil, Eastern Regional Research Laboratory, Philadelphia 18, Penn.

α -Phosphono fatty acids of 9, 10, 12, 14, 16 and 18 carbon atoms and their salts and esters were prepared from pelargonic, capric, lauric, myristic, palmitic and stearic acids, respectively, in the following reaction sequence:



In comparison to corresponding α -sulfo fatty acids the α -phosphono fatty acids are white solids of higher melting point. They are tribasic, less hygroscopic, have a lower critical micelle concentration and are less resistant to hard water.

Potentiometric titration of an aqueous ethanol solution with sodium hydroxide gave equivalence points at pH 5.0, 8.0 and 11.0. This corresponds to neutralization of the first acidic hydrogen of the $-\text{PO(OH)}_2$ group, neutralization of the $-\text{COOH}$ group and neutralization of the remaining acidic hydrogen of $-\text{PO(OH)}_2^-$, respectively.

At 25°, the solubility of the monosodium, disodium and trisodium salts of α -phosphonopalmitic acid is < 1%, 1%, and 1%, respectively. This increase in solubility with successive salt formation is generally true of the alkali metal salts and of salts with ammonia, amines and analcolamines.

Methyl, isopropyl and amyl esters $\text{RCH[PO(OH)}_2\text{]CO}_2\text{R}$, were prepared from the α -phosphono fatty acids. The α -sulfo fatty acids can be directly esterified without the addition of acid catalyst but the lower acidity of the α -phosphono fatty acids required a sulfuric acid catalyst in the case of the lower boiling alcohols.

Wetting, foaming, detergent and other surface active properties of the α -phosphono fatty acids, salts and esters were measured and compared with those of analogous α -sulfo compounds.

ANALYSIS OF SOAPS WITH HYDROGEN BROMIDE IN GLACIAL ACETIC ACID

E. T. Haeblerer and G. Maerker, Eastern Regional Research Laboratory, Philadelphia 18, Penn.

Titration of alkali metal salts of fatty acids with hydrogen bromide in glacial acetic acid and in the presence of crystal violet indicator permits the direct quantitative determination of soaps in the presence of free fatty acids. The analytical method is essentially the same as that used in the determination of oxirane compounds, and the latter interfere in the determination of soaps.

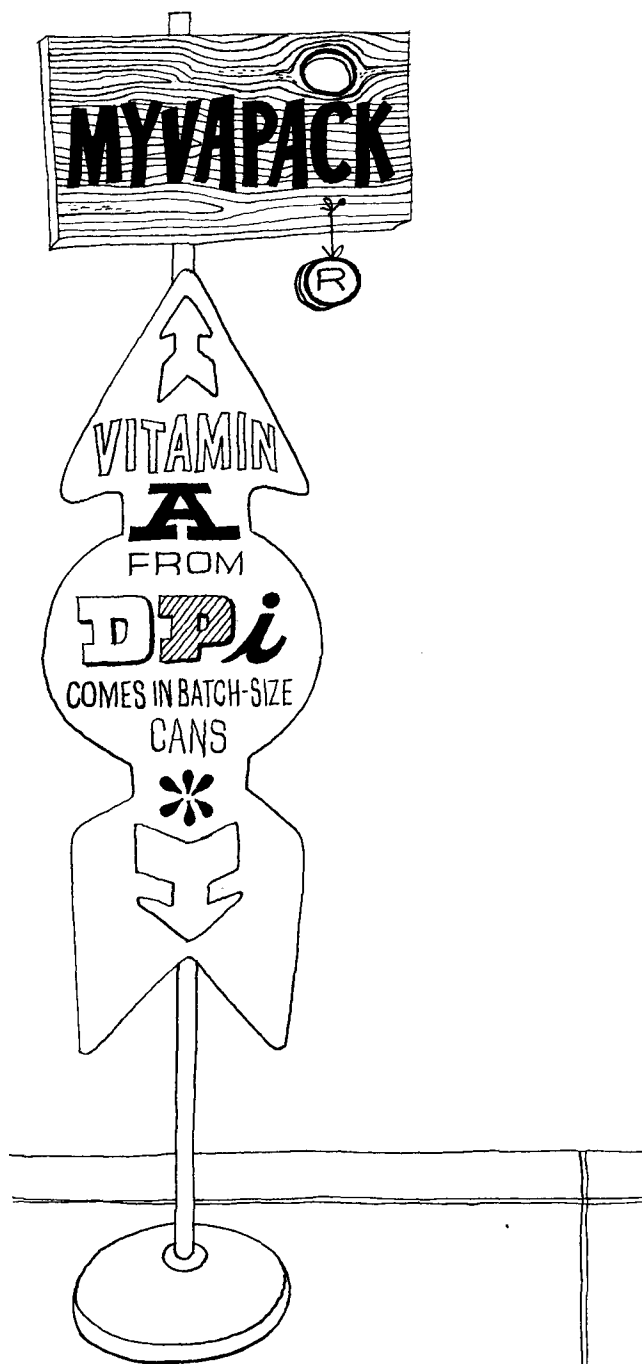
Alkali metal salts of strong acids are not titrated by this method. Disodium or dipotassium salts of α -sulfo fatty acids consume only one equivalent of hydrogen bromide. The method is therefore suitable for determining the amount of alkali metal associated with the carboxylic acid function in salts of α -sulfo fatty acids.

Alkaline earth salts of α -sulfo fatty acids are poorly soluble in the glacial acetic acid solvent and do not give reproducible results.

SODIUM ALKYL PHOSPHATE SURFACTANTS PROPERTIES AND USE IN HEAVY DUTY DETERGENTS

Robert S. Cooper and Allen Urfer, Research Department, Victor Chemical Division, Stauffer Chemical Company, Chicago Heights, Ill.

(Continued on page 20)



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5. Desirable promotion of the national Society
6. Adequate opportunity for authors to present technical papers
7. Maximum participation by the largest number of members
8. Maximum attraction for new members
9. A net return of \$3,000 to \$4,000 per year to cover costs of the national office incidental to the meetings
10. Feasibility of planning the meeting programs

Under the second category, the Committee agreed that the meetings must not create:

1. Too many distractions from the technical programs and committee meetings.
2. Interference of social and commercial activities with the "must" objectives described in 2 (a) above

Under the third category, the Committee agreed that they would like for the meetings to provide:

1. Economy of time and expense for those attending
2. The avoidance of conflicts between technical program, committee meetings, official activities, the golf match, etc.
3. Three-day meetings
4. The maximum stimulation of research and the development of technology
5. Stimulation of enthusiasm of the local groups
6. A good program for the ladies

With the foregoing in mind, the Committee considered alternates of two meetings per year, one meeting per year, one meeting every eight months, one national meeting plus special or regional meetings per year, three meetings per year, and three meetings in two years. After weighing the advantageous and disadvantageous consequences of each, they agreed to recommend to the Governing Board that the Society continue having two meetings per year because the two meetings can better provide:

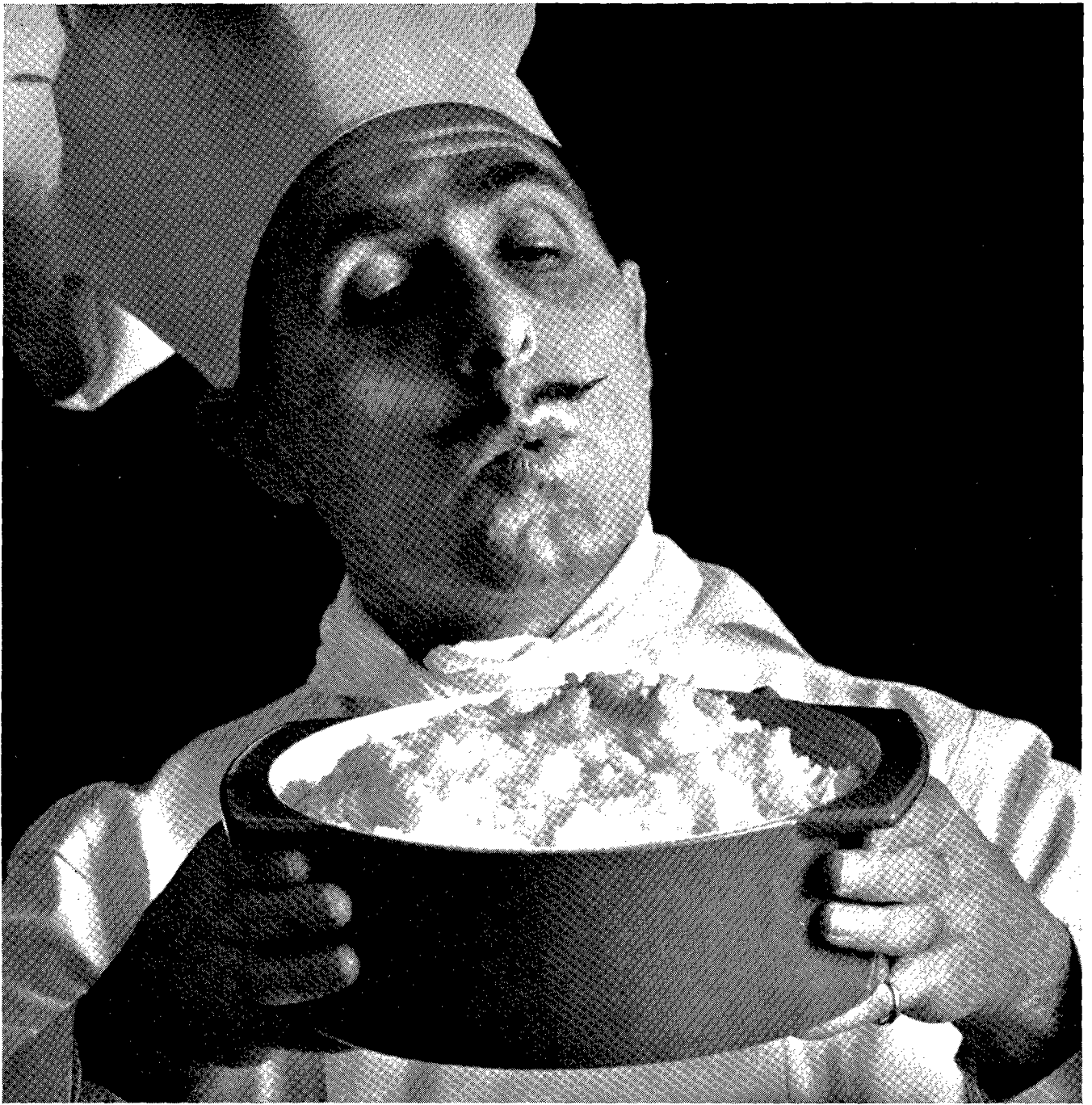
1. Participation of more members
2. Opportunity for authors to present their papers
3. A better chance for more personal contacts among members
4. More opportunity for having meetings in areas such as Dallas, Houston, Memphis, Toronto, St. Louis, Los Angeles, San Francisco, Cincinnati, Minneapolis, Philadelphia and Atlanta where local groups can develop enthusiasm through planning and presentation of the meetings.
5. Attract more new members

At the same time, the Committee thought that consequences of having only one meeting per year would be:

1. It would not attract as many new members
2. It would decrease the total annual attendance at the AOCS meetings
3. It would disappoint authors who would not wish to hold papers for up to a year before presenting them at AOCS meetings
4. It would encourage authors to present their papers in other places because of the time factor.
5. It would lessen the opportunity of personal contacts
6. It would require longer (4 to 5 days) meetings

After lengthy discussion, the Governing Board voted to accept the Special Committee Report, and to continue the Society's activities on the basis of two meetings per year.

(Continued on page 21)



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An extensive study of the physical and surface active properties of sodium salts of monoalkyl dihydrogen phosphates and dialkyl hydrogen phosphates has been made. These products have also been evaluated for use in built detergents and their performance compared with commercial surfactants.

The preparation of pure monoalkyl dihydrogen phosphates and dialkyl hydrogen phosphates is discussed as well as solubility, surface tension, and other properties of the sodium salts.

Performance in built detergents is discussed in some detail. Good performance is realized when the number of carbon atoms to phosphorus in the molecule is between 17 and 23 to one. Optimum performance is realized with the symmetrical didecyl hydrogen phosphate salts.

ELECTROPHORETIC PROPERTIES IN AQUEOUS DETERGENT SYSTEMS

M. E. Ginn of Armour and Co., Chicago, Ill., and R. M. Anderson and J. C. Harris, Monsanto Chemical Co., Dayton, O.

Electrophoretic mobilities and zeta potentials were developed for triolein, nujol and glass particles in aqueous detergent solutions. Zeta potentials in deionized water were -79, -69 and -62 mv, respectively, for triolein, nujol and glass. Electrolyte builders and anionic surfactants markedly increase triolein and nujol particle mobilities and negative zeta potentials, and this effect probably contributes to their detersive action. Nonionic surfactants lower mobility of triolein and glass, thus show slight cationic character. Significant inflection points are found for nonionic detergents near cmc. A cationic detergent produced high positive charge and zeta potentials with triolein and glass. Data for surfactant/builder combinations show mobilities are primarily determined by the electrolyte. It was concluded that electrokinetic effects are important for dispersion and emulsification, but are not necessary and sufficient conditions for removing oily soil from glass.

Laboratory Methods of Analysis

FURTHER OBSERVATION ON THE 2-THIOBARBITURIC ACID METHOD FOR THE MEASUREMENT OF OXIDATIVE RANCIDITY

T. C. Yu and Russell O. Sinnhuber, Department of Food Science & Technology, Oregon State University, Corvallis, Ore.

The validity of the 2-thioarbituric acid (TBA) procedure for the measurement of oxidative rancidity by the determination of malonaldehyde (MA) as the 535 m μ red pigment has been questioned [JAQCS 39, 34 (1962)]. Side reactions were reported to occur yielding degradation of TBA which adsorb at same wave length as the TBA-MA complex.

Results reported in the present paper support the TBA procedure as

originally proposed and stress the importance of reagent purification. Little or no decomposition of TBA to produce interfering colors was found after heating with acids, oxidizing agents or hydroperoxides.

GAS CHROMATOGRAPHY OF CIS-TRANS FATTY ACID ISOMERS ON NITRILE SILICONE CAPILLARY COLUMNS

Carter Litchfield, Raymond Reiser, and A. F. Isbell, Dept. of Biochemistry & Nutrition, Texas Agricultural Experiment Station, College Station, Texas; and G. L. Feldman, Institute of Ophthalmology, Baylor University College of Medicine, Houston, Tex.

Three nitrile silicone polymers have been evaluated as gas chromatography liquid phases for separating the geometric isomers of methyl oleate, methyl linoleate, and methyl linolenate on capillary columns. A polymer of β -cyanoethylmethylsiloxane proved the most effective. This liquid phase separated oleate from elaidate, resolved the four geometric isomers of linoleate into three peaks, and divided the eight geometric isomers of linolenate into six peaks. Two other copolymers of dimethylsiloxane and β -cyanoethylmethylsiloxane gave poorer resolution of *cis-trans* isomers, but showed different elution patterns for the geometric isomers of linoleate and linolenate.

A PRELIMINARY STUDY OF THE LENTICULAR LIPIDS OF THE HUMAN EYE

Gerald L. Feldman, Division of Ophthalmology, Baylor University College of Medicine, Houston, Tex.

The lipids of individual human lenses were fractionated by silicic acid column chromatography. Cholesterol was the most abundant neutral lipid followed by triglycerides and cholesterol esters. Gas chromatography studies revealed that both of these lipids contained nearly equal quantities of saturated and unsaturated fatty acids. The lenses contained slightly more phospholipids than neutral lipids and the sphingomyelins predominated. The phospholipids were mostly saturated with palmitate comprising about fifty percent of the total fatty acid. Oleate was the primary unsaturated acid. Cataracts caused a profound change in these parameters. Cholesterol and the phospholipids were greatly increased in diseased lenses. The other neutral lipids were quantitatively unchanged. Slight changes occurred in the fatty acid composition of the neutral lipids, but the phospholipids were markedly altered. This was particularly true in a diabetic cataract in which large amounts of a C₁₇ acid and linolenic acid were observed.

A LABORATORY CENTRIFUGAL REFINING METHOD FOR CONTROL APPLICATION

W. D. Pohle, R. L. Gregory and S. E. Tierney, Research Laboratories, Swift & Company, Chicago, Ill.

A control method is presented for selecting the appropriate processing conditions for alkali refining of crude vegetable oils by the centrifugal process to yield lowest losses with satisfactory color. This technique is sufficiently analogous to actual processing conditions to provide reliable information upon which plant performance can be based.

The classic cup method cannot be used in this manner in that it no longer approximates operating procedures as in the days of kettle refining. The chromatographic neutral oil method on the other hand provides an index of the amount of oil available for recovery without regard to the possibility of attaining such levels.

For these reasons the centrifugal method fills a void of long standing. Other tangible benefits that accrue from this technique are: selection of sources of oil that can be most profitably refined by establishing the relative value of competitive oils, and a means of evaluating plant efficiency.

MEASUREMENT OF SOLIDS IN TRIGLYCERIDES USING NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY

J. R. Taylor, W. D. Pohle, and R. L. Gregory, Research Laboratories, Swift & Company, Chicago, Ill.

A rapid and accurate method is presented for determining the percent of solids in fats and shortenings conditioned at selected temperatures or as received in the laboratory using low resolution nuclear magnetic resonance spectroscopy. This method provides more reliable information on the solids content of fatty materials than the empirical dilatometric solid fat index and is applicable in the range of 50-100% solids which is beyond the limit of the solid fat index.

The relationship between instrument response and actual solids present was determined on known mixtures of liquid and solid triglycerides. Nuclear magnetic resonance and solid fat index measurements were made on a series of commercial margarine oils of varying composition and consistency. Comparisons are presented giving the precision of the two techniques and the relationship between percent solids by the nuclear magnetic resonance technique and the solids fat index.

Laboratory Preparation

PREPARATION OF 9-TRANS, 12-TRANS-OCTADECADIENOIC ACID

Robert D. Harlow, Carter Litchfield, and Raymond Reiser, Dept. of Biochemistry & Nutrition, Texas Agricultural Experiment Station, College Station, Tex.

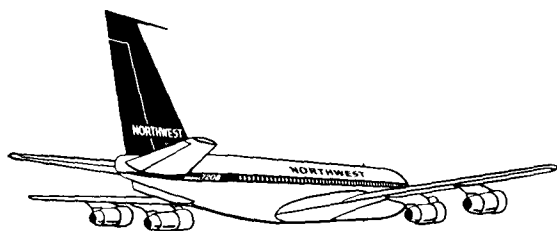
A method for preparing 9-*trans*, 12-*trans*-octadecadienoic acid from safflower oil has been developed. This procedure uses HNO₃ instead of Se as a *cis-trans* isomerization catalyst, since Se produces undesirable side reactions and is difficult to remove from the final product.

Safflower fatty acids were obtained from safflower oil by saponification and acidulation. These acids were then used to produce a linoleic acid concentrate by urea precipitation from methanol solution. Next, the linoleic acid concentrate was converted to its methyl ester and *cis-trans* isomerized with HNO₃. The product was dissolved in petroleum ether and passed through a column of silicic acid to remove nitrogenous reaction by-products. The isomerized methyl linoleate was then converted to fatty acid and recrystallized from acetone at -20°C.

Using this method, 1000 g. of safflower oil yielded 40 g of pure 9-*trans*, 12-*trans*-octadecadienoic acid.

(Continued on page 22)

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J. C. Harris

problems, structural factors affecting the biodegradability of surfactants, development in products and processes as they affect biodegradability development in Europe, are on the agenda. This symposium is under the chairmanship of E. S. Pattison, Manager, Soap and Detergent Association. It is planned to have a European speaker take part in these discussions, giving it added importance and international scope. Details of topics to be covered and speakers taking part in this symposium will be announced in the April issue of this Journal.

Tuesday afternoon will be kept free for plant trips, informal discussions, and rest. A complete schedule of trips will also be given in the next issue.

Wednesday: "Analyses and Properties of Detergents"

The Wednesday session will concentrate on the topics dealing with the "Analyses and Properties of Detergents." M. Schick, Lever Bros., will discuss the physical chemistry of non-ionic detergents. He will cover such areas as preparation of non-ionics and properties such as micellar structures, thermo-dynamics of micell formation, surface films, and detergency and foaming properties. H. B. Trost, Hercules Powder Company, will review the problem of soil redeposition with some emphasis on the application to new fabrics. A. M. Mankowich, U. S. Army Coating and Chemical Laboratory, Aberdeen Proving Ground, will present a paper on the Correlation of Detergents with Physico-chemical Factors, and M. E. Ginn, Armour and Company, will discuss new physical evaluation techniques, pointing out their usefulness in the screening of new detergent raw materials.

The afternoon session will discuss new analytical methods. M. J. Rosen, Brooklyn College, will review the most important developments in the last five years. An additional paper will be given by R. R. Irani, Monsanto Chemical Co., discussing the application of nuclear magnetic resonance to the analysis of detergent products.

Detailed Program in April Issue

A full program for the Short Course will be announced in the April issue of the Journal of the American Oil Chemists' Society and registration forms will be mailed to members. For further information address your inquiries to Dr. E. Jungermann, AOCS, 35 East Wacker Drive, Chicago, Illinois.

• Referee Applications

First Notice. W. D. Simpson of Woodson-Tenent Laboratories, Wilson, Ark., has applied for a Referee Certificate on oilseed meal and vegetable oils. The Chairman of the Examination Board should be contacted by interested parties wishing to comment on this certification. Please write to N. W. Ziels, Chairman of the Examination Board, Lever Brothers Co., 1200 Calumet Ave., Hammond, Ind.

First Notice. J. W. Thomas, of Southern Testing Laboratories, Inc., 5450 River Rd., Westwego, La., has applied for a Referee Certificate on crude vegetable oils and tallows and greases. The Chairman of the Examination Board should be contacted by interested parties wishing to comment on this certification. Please write to N. W. Ziels, Chairman of the Examination Board, Lever Brothers Co., 1200 Calumet Ave., Hammond, Ind.

• Industry Items

DELHI-TAYLOR CORPORATION, New York City, N. Y., has announced that its Charleston, South Carolina terminal now distributes hexane to speed service and delivery to eastern seaboard manufacturers.

UNIVERSAL OIL PRODUCTS Co., Chicago, Ill., recently consolidated two wholly-owned subsidiaries; The Trubek Laboratories, Inc., East Rutherford, N. J., is now The Trubek Chemical Co. and Catalytic Combustion Corp., Detroit, Mich. and Bloomer, Wise., has been designated Catalytic Combustion Co. No changes have been made in the management of the former subsidiaries.

SCIENTIFIC PRODUCTS DIVISION OF AMERICAN HOSPITAL SUPPLY CORPORATION, Evanston, Ill., has been appointed sole distributor of all AOCS Official Supplies. Contact AOCS, 35 East Wacker Drive, Chicago or Scientific Products, 1210 Leon Place, Evanston, Ill., for complete details.

A.O.C.S. Commentary . . .

(Continued from page 18)

From a consideration of the foregoing, I am sure the membership will agree that the Governing Board has been alert to the host of problems connected with the holding of Society Meetings, as well as its growth and service. It is considered that future meetings of the Society will be conducted so as to permit the maximum participation of the members in both the technical and the committee meetings and with the minimum of distraction from these activities by the entertainment features that help so much to round out the meetings and make them universally satisfactory.

A. ERNEST MACGEE, President
American Oil Chemists' Society

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