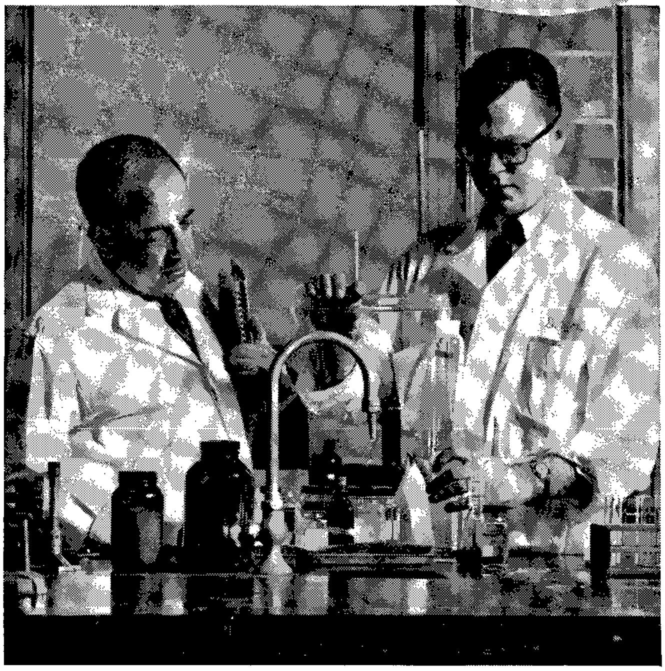
When industry has a problem— Beacon Chemical finds the answer

BEACON CHEMICAL



Chemical research staff at Beacon Chemical Industries, Inc.

THE PROBLEM: Crystal formation in edible and non-edible oils under cold conditions

THE ANSWER: CLARICOL—the new improved crystal inhibitor

Another triumph from Beacon Research. CLARICOL — the new unique crystal inhibitor for use as a winterizing aid in producing edible and non-edible oils and for improving the Cold Test in these products. Salad oils, cooking oils, mayonnaise, salad dressings, remain *crystal-free longer under colder conditions with* CLARICOL.

Get dramatic results at low cost! The addition of very small quantities of CLARICOL inhibits the precipitation of solid fat crystals at low temperatures. As little as 0.03 to 0.04% CLARICOL added to cottonseed or soybean salad oil quadruples the AOCS Cold Test on a typical oil. Cold Tests of 50 to 100 hours are common when CLARICOL is used. CLARICOL's inhibiting power limits eventual crystals to imperceptible size — no *heavy floc.*

When added to refined cottonseed oil, prior to winterizing, as little as 0.02 to 0.04% CLARICOL speeds up crystal precipitation and overall production. Oils containing CLARICOL are more easily and rapidly refined. Yield of winterized oil is increased and the Cold Test of the resulting oil is improved. CLARICOL is semi-fluid, easy to handle, economical to use! It is a Food Additive (21 CFR. Subpart D, Section 121-1016).

BEACON CHEMICAL

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33 Richdale Ave.,
Cambridge 40, Mass.
Manufacturers of Organic Intermediates.

BEACON CHEMICAL—WHERE INDUSTRIAL PROBLEMS ARE SOLVED THROUGH CHEMICAL RESEARCH

• *Report on Fats and Oils*

Lost and Found

THE BEAN MARKET is an excitable, volatile, emotional place. This is due to a number of factors.

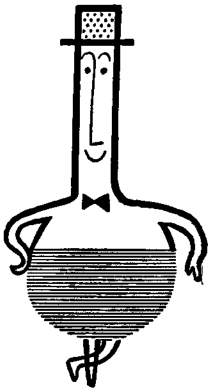
1. The people who trade beans are prone to get on one side of the market and stay there.
2. In past years, the bean market has had big moves.
3. Certain demand vectors for both beans and products are quite unpredictable.
4. Small carryovers mean high leverage for each bushel change in supply or demand.

As a consequence of No. 4 an inordinate number of trading decisions are based on U.S.D.A. crop estimates and U.S.D.A. stock estimates. It is not surprising to see unpredictable shifts in crop estimates since, presumably, the caprices of weather are responsible for most of the changes. Stock estimates, on the other hand, are another item. Most traders calculate what quarterly bean stocks should be in the obvious manner, that is, subtract reported disappearance from beginning stocks. Unfortunately, this will almost never yield a figure even reasonably close to the actual stocks report. The difference between actual stocks and theoretic stocks is referred to by analysts as the "quarterly residual error." Some quarters of the year beans are apparently lost and then suddenly they turn up again. Consequently, residual errors are sometimes referred to as the "Lost and Found Department." This error in the estimate forms a regular pattern but the magnitude is always uncertain. The pattern is that extra beans are found in the first and fourth quarters of the October–October season but beans are lost in the second and third quarters. On an annual basis, some beans are almost always lost. Where the found beans come from, and where the lost beans go to, is a matter of disagreement in the market. Since all processors and terminal elevators are presumably included and presumably they all keep accurate records, the finger points to the farm position where estimates have to be made and by a small sample. This, however, does not explain why the pattern of error is so regular. The annual loss is probably largely due to moisture shrink and handling loss. I am unable to form a strong view on the individual quarter errors. U.S.D.A. stubbornly refers to the discrepancy as, "*mostly quantity fed* but includes waste loss and statistical errors." (Italics mine) I might be persuaded someday to believe that animals ate the 26.9 million bushel loss error in the third quarter of 1961–1962, but I will never be convinced that they coughed up the 28.1 million bushels found in the fourth quarter of the same season.

In recent weeks, the bean market has once again become obsessed with the Lost and Found Department. Interest in it had been rising ever since December, 1962 when the government lowered the 1961 production from 693 to 679 million bushels. This forced sharp revision of quarterly errors for that season and made the annual "lost" error so low as to imply heavy utilization of new crop beans last September. Then the January stocks report showed lost beans for the October–December quarter which is quite unusual. This implied over-estimation of the current crop (1962–1963) even if say, 13 million new beans were used in September. Depending on what crush and export estimates are used for the full season, if the lost beans in the first quarter should be followed by further losses in the second and third quarters (which would be usual), then the July report is liable to imply insufficient beans to last out the season. After the market had a considerable rally, U.S.D.A. managed to chill most of the up move by forecasting a carryover of 55 million bushels at end season. Since by that time the U.S.D.A. had sold most of their inventory and consequently lost their grip on the market, this was considered to be a bearish forecast.

In view of European weather, the progress of crush and

(Continued on page 37)



Meetings

A.O.C.S. National Meetings

1963—Atlanta, Atlanta Biltmore Hotel, April 22–24
Minneapolis, Radisson Hotel, September 30–October 2
1964—New Orleans, Roosevelt Hotel, April 19–22
Chicago, Pick-Congress Hotel, October 11–14
1965—Houston, Shamrock-Hilton Hotel, April 25–28
Cincinnati, October 11–13
1966—Los Angeles, Statler Hilton Hotel, April 24–27
Philadelphia, Bellevue-Stratford Hotel, October 4–6
1967—New Orleans, Roosevelt Hotel, May 7–10
Chicago
1968—New York

A.O.C.S. Section Meetings

North Central—May 1, 1963, at the Builders' Club, 228 N. LaSalle, Chicago, Ill.

Northeast—April 2, 1963, at the Essex House, Newark, N. J. June 4, 1963, at Whyte's Restaurant, 141 Fulton St., New York

Southwest—March 14, 1963 and May 9, 1963, at Rodger Young Auditorium, Los Angeles, Calif., 6:30 p.m.

A.O.C.S. Short Course, 1963

June 23–26—Advances in Soaps and Detergents, Princeton Inn, Princeton, N. J.

Other Organizations

*April 15–19, 1963—5th Annual Gas Chromatography Institute, Canisius College, Buffalo, N. Y.

*May 8, 1963—Semi Annual Meeting and Special Award Presentation, Society of Cosmetic Chemists, Hotel Biltmore, New York, N. Y.

May 26–29, 1963—23rd Annual Meeting and Industrial Exhibit, Institute of Food Technologists, Cobo Hall, Detroit, Mich.

*June 3–6, 1963—5th Annual Symposium, Coatings Technology Dept., College of Chemical Technology, North Dakota State University, Fargo, N. D.

June 5–6, 1963—Symposium and 46th Meeting of the European Federation of Chemical Engineering, Frankfurt am Main.

*June 6–8, 1963—46th Annual Conference and Exhibition of The Chemical Institute of Canada, Royal York Hotel, Toronto, Ont., Canada.

*June 16–18, 1963—Symposium by the Burnsides Research Laboratory, University of Illinois, Urbana, Ill.

June 23–28, 1963—Chalfonte-Haddon Hall, Atlantic City, N. J. American Society for Testing and Materials Annual Meeting.

*Sept. 24–25, 1963—10th Annual Seminar of the Society of Cosmetic Chemists, Hotel Somerset, Boston, Mass.

Oct. 30–Nov. 2, 1963—41st Annual Meeting, Federation of Societies for Paint Technology, Sheraton Hotel, Philadelphia, Pa.

Oct. 31–Nov. 2, 1963—28th Paint Industries' Show of the Federation of Societies for Paint Technology, Sheraton Hotel, Philadelphia, Pa.

*Dec. 3, 1963—Annual Meeting of the Cosmetic Chemists, Biltmore Hotel, New York, N. Y.

Feb. 3–7, 1964—ASTM National Committee Meeting, at the Sheraton Hotel, Philadelphia, Pa.

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* Additions to previous calendar.

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for temperatures to 300°C

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Principle—Air diffused through the porous bottom of the bath produces a flotation effect upon the sand particles, reducing the relative density. The expanded bed behaves as a fluid; surface bubbles give the appearance of a boiling liquid. Heat is distributed through the bath by air passage and the displacement of sand particles. The fluidized bed exerts a buoyant effect upon immersed objects.

Size—Inside dimensions 7¼ inches diameter × 5¾ inches deep to heater coil. Depth is sufficient to immerse 1000 ml round bottom flasks to the neck, or 2000 ml flasks to over half flask diameter. Overall dimensions, approximately 11 inches high × 9½ inches diameter, exclusive of air valve and safety switch, which extend near base approximately 4 inches to the right and rear respectively.

Wattage—Heater is rated at 750 watts at 115 volts, a.c.

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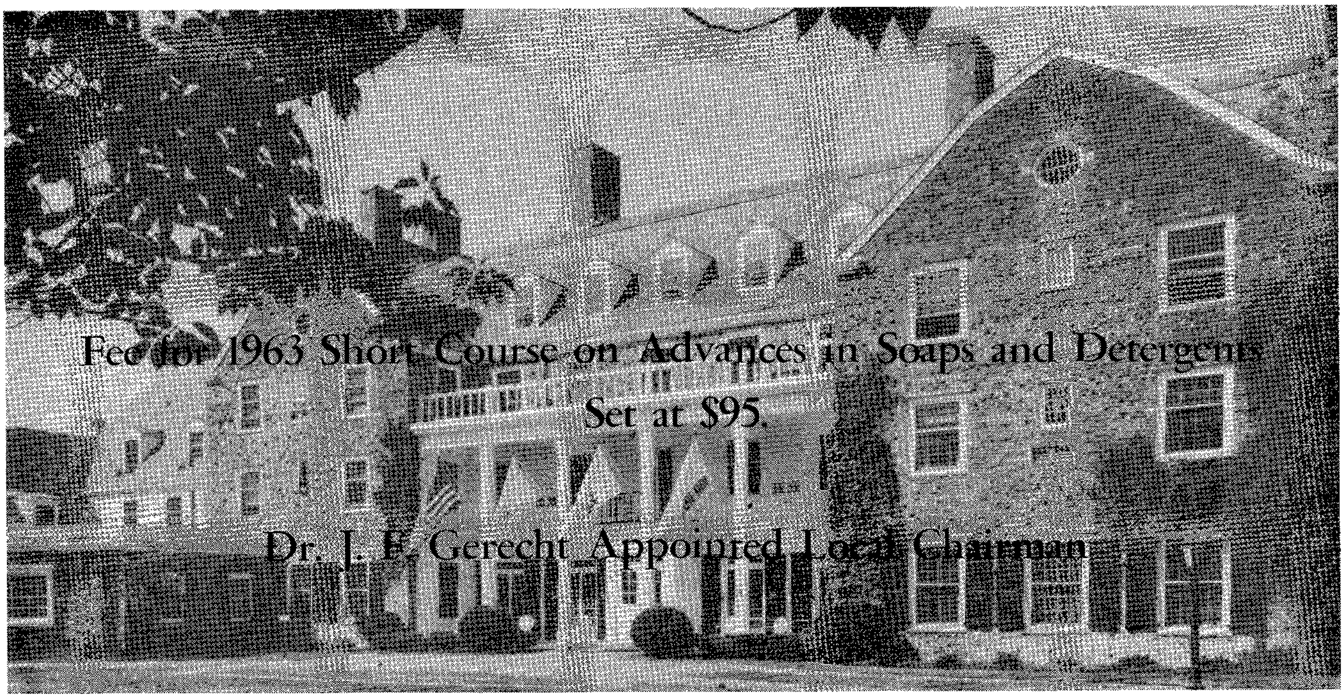


Fee for 1963 Short Course on Advances in Soaps and Detergents Set at $95

Dr. J. F. Gerecht Appointed Local Chairman

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 roommates, 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.

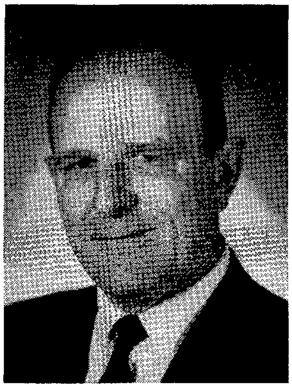
New Firmenich & Co. Film to be Shown

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.

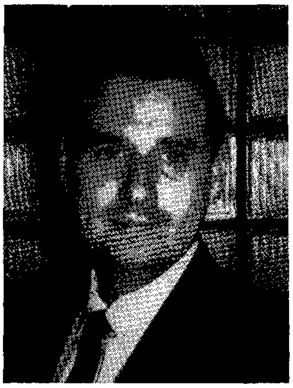
(Continued on page 15)

Eric Jungermann

L. J. Garrison



J. F. Gerecht



M. E. Ginn

Conducting ultraviolet spectrophotometric analysis.

An extremely sensitive system of analysis proves that Esso Hexane has no residue that could possibly contaminate extracted oils. The test procedure, developed by Humble research, can detect as little as one *hundredth* of a part per *million*. Samples of Esso Hexane were analyzed direct from the refinery, from storage, and after transportation to solvent extraction plants. In *no* case could any known polynuclear aromatic carcinogenic materials be found. High-purity Esso Hexane, like the new test procedure, is a development of the world's largest petroleum research organization. For more information about Esso Hexane, contact our sales representative. If you'd like a copy of the technical paper describing the test, write us at Houston, Texas.

IT PAYS TO DO BUSINESS WITH HUMBLE . . . AMERICA'S LEADING ENERGY COMPANY

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 ether 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_2SO_4	CH_2N_2	BF_3	$Pr(OMe)_2$
1	Yield	102.4	94.9	92.3	100.8
	% FFA	0.4	5.9	0.2	3.4
	Time(hr)	1	1	1	1
2	Yield	101.5	101.0	103.0	101.5
	% FFA	0.6	0.4	0.2	51.5
	Time(hr)	3	2½	2	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
	$C_{18}, \Delta 1$	29.45±0.41	28.92±0.39	29.40±0.41	29.25±0.38
	$C_{18}, \Delta 2$	47.29±0.62	47.78±0.65	48.76±0.64	47.71±0.67
	$C_{18}, \Delta 3$	8.09±0.27	7.14±0.24	7.05±0.24	6.53±0.23
	C_{20}	tr.	tr.	tr.	tr.
3	Yield	101.2	98.5	100.0	101.6
	% FFA	1.0	......	0.3	0.2
	Time(hr)	2	2½	1¾	1¾
	C_{16}		(1)		
4	Yield	104.3	103.9	103.6	104.3
	% FFA	0.8	10.2	......	4.2
	Time(hr)	2¼	1¾	1½	2¼
	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
	$C_{18}, \Delta 1$	29.27±0.39	29.53±0.40		29.49±0.41
	$C_{18}, \Delta 2$	47.28±0.66	48.60±0.66		47.89±0.64
	$C_{18}, \Delta 3$	7.02±0.24	6.51±0.22		7.28±0.25
	C_{20}	tr.	tr.		tr.
5	Yield	102.2	101.5	100.0	102.0
	% FFA	0.8	1.6	0.2	2.2
	Time(hr)	2¼	2	1½	2¼
	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
	$C_{18}, \Delta 1$	29.40±0.40	28.46±0.40	29.41±0.40	29.55±0.40
	$C_{18}, \Delta 2$	48.51±0.66	48.74±0.64	48.77±0.65	48.26±0.67
	$C_{18}, \Delta 3$	6.52±0.22	7.34±0.25	6.75±0.23	6.51±0.23
	C_{20}	tr.	tr.		
6	Yield	101.4	101.7	102.8	101.4
	% FFA	0.5	0.2	0.3	0.3
	Time(hr)	2½	2	1½	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 CH_2N_2 and BF_3 methods. For this particular sample only the MEOH-H_2SO_4 method is preferred.

Normally prefer a method using CH_2N_2 in ether.

No. 2—Carbitol carried over during CH_2N_2 procedure. $Pr(OMe)_2$ method gives a product difficult to clear.

No. 3—Difficulties encountered with diazomethane procedure. If CH_2N_2 is to be used, another method would be preferable.

No. 4—None.

No. 5—Equipment for CH_2N_2 method is cumbersome and not satisfactory for a large number of samples.

No. 6—CH_2N_2 procedure not satisfactory because of carry-over of carbitol and water. Method of McKay is better.

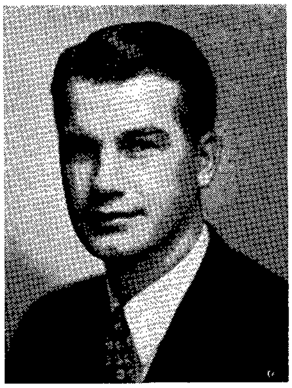
No. 1 and 3 preferred MEOH-H_2SO_4 method.

No. 2, 4, 5, and 6 preferred BF_3 method.

'63 Short Course . . .

(Continued from page 12)


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

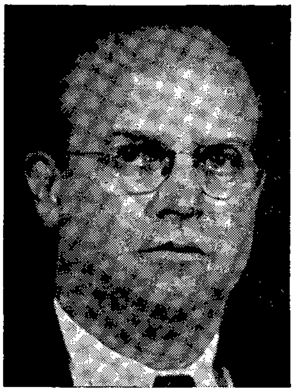
An international note is being added to the Monday session with the participation of Dr. Ballestra of Italy, who will discuss continuous sulfonation processes both in Europe and this country.

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 organophosphorus 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.

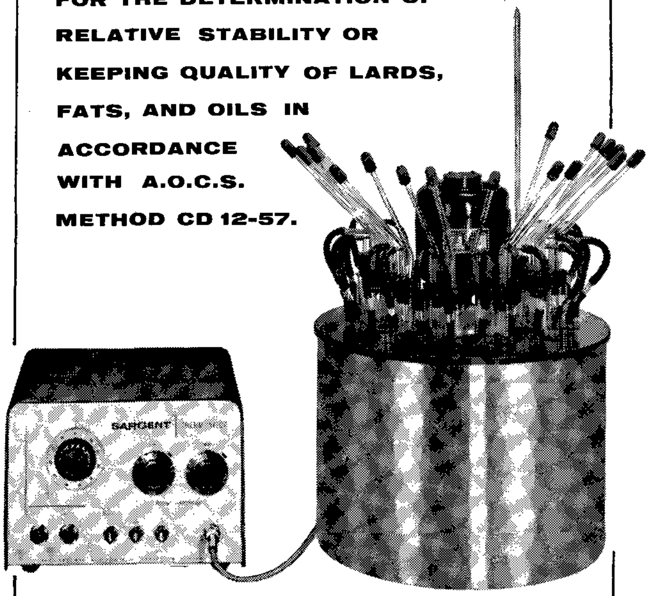
Tuesday: Biodegradability Symposium, Plant Trips

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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Atlanta Abstracts . . .

(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-nitrato-octadecanoate, methyl 12-nitrato-octadecanoate, and 18,19-dinitrato-hexatricontane. In addition, mixed alkyl nitrates and nitrato 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 µc/mg and incorporated 37% of the C^{14} used. The meal (sp. act-0.134 µc/mg) accounted for an additional 16.5%, and the residual roots, leaf, and stem (sp. act-0.05µc/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 desolventization 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 desolventized 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, desolventized 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 pickup 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 180C, 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.

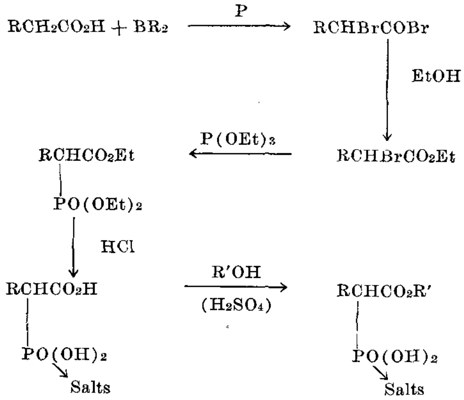
The course of the homogeneous hydrogenation catalyzed by metal carbonyls appears very similar to the heterogeneous catalytic reaction. Metal carbonyls are well known for their isomerizing effects on olefins and for their ability to form stable *pi*-complexes with double bonds of conjugated polyenes and 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 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 $-PO(OH)_2$ group, neutralization of the $-COOH$ group and neutralization of the remaining acidic hydrogen of $-PO(OH)O^-$, 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 alkanolamines.

Methyl, isopropyl and amyl esters $RCH[PO(OH)_2]CO_2R$, 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. Haeberer 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.

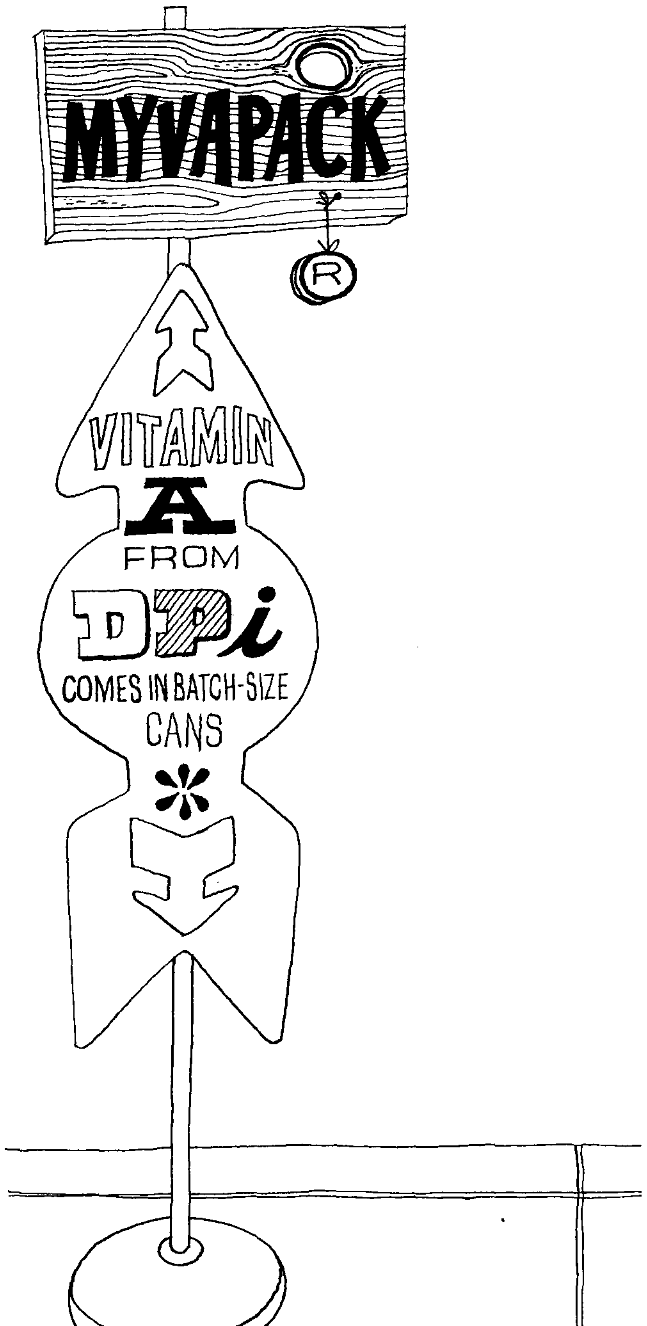
Alkali metal salts of strong acids are not titrated by this method. Disodium or dipotassium salts of α-sulfo fatty acids consume only one equivalent of hydrogen bromide. The method is therefore suitable for determining the amount of alkali metal associated with the carboxylic acid function in salts of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

(Continued from page 7)

2. Good technical programs
3. Committee meetings
4. Opportunity for wide personal contacts
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:

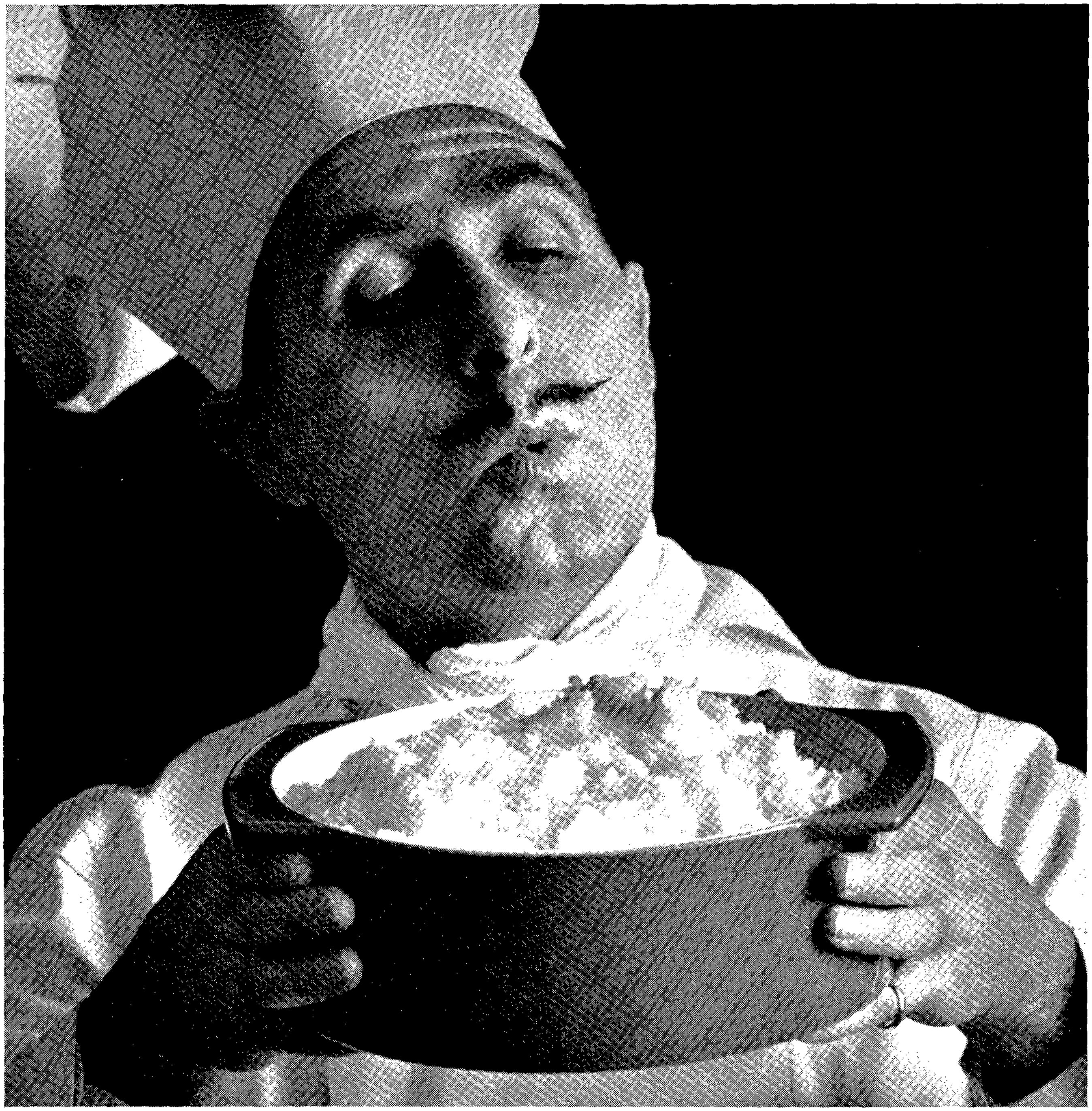
1. Participation of more members
2. Opportunity for authors to present their papers
3. A better chance for more personal contacts among members
4. More opportunity for having meetings in areas such as Dallas, Houston, Memphis, Toronto, St. Louis, Los Angeles, San Francisco, Cincinnati, Minneapolis, Philadelphia 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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Atlanta Abstracts

(Continued from page 17)

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 anl 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-thiobarbituric acid (TBA) procedure for the measurement of oxidative rancidity by the determination of malonaldehyde (MA) as the 535 mμ red pigment has been questioned [JAOCS *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.

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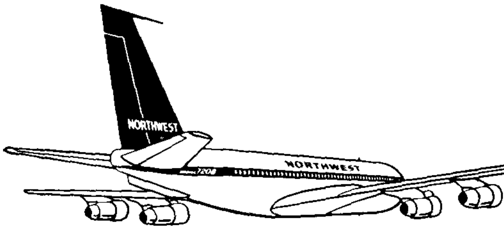

NORTHWEST
ORIENT AIRLINES

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 C_{17} 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 HNO_2 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 HNO_2. 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 —20C.

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

(Continued on page 22)

63 Short Course . . .

(Continued from page 15)

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 Physicochemical 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.

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• *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.

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• *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, Wisc., 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.

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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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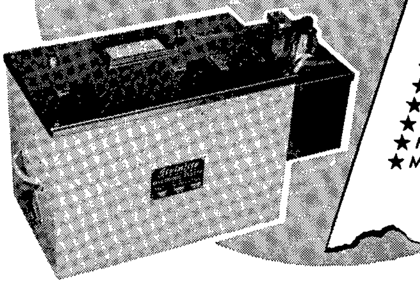

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Atlanta Abstracts . . .

(Continued from page 20)

STUDIES ON THE OZONIZATION OF METHYL OLEATE

O. S. Privett and E. Christense Nickell, University of Minnesota, The Hormel Institute, Austin, Minn.

The ozonides of methyl 9-octadecenoate, 9-octadecene and methyl 9-octadecenedioate, pelargonyl aldehyde, methyl zaelaic semialdehyde and a fraction consisting mainly of hydroperoxyozonides were isolated from ozonizations of methyl oleate in pentane and methylene chloride at —65C. The relative amounts of these compounds were determined in ozonizations of methyl oleate in pentane, methylene chloride and ethyl acetate.

The formation of ozonides by an interreaction of ionic intermediates was demonstrated from studies on the products of the ozonization of mixtures of methyl linoleate with 9-octadecene and with pelargonyl aldehyde.

These and other experiments provided new information on the mechanism of the ozonization of monoethenoid fatty acids and other simple olefins.

VERNONIA ANTHELMINTICA (L.) WILLD. HIGHLY PURIFIED EPOXY FATTY ACID COMPONENTS FROM THE SEED OIL

Charles F. Krewson and Francis E. Luddy, Eastern Regional Research Laboratory, Philadelphia 18, Penn.

In a previous report [52nd Annual Meeting and JAOCS *39*, 334-340 (1962)] conditions for obtaining trivernolin, 1.3-divernolin and vernolic (12,13-epoxyoleic) acid from the oil of mature *Vernonia anthelmintica* seed were described. This report deals with the refinement of these epoxy oil components to obtain chromatographically pure compounds. Some physical properties of these compounds are given.

Trivernolin has been the most difficult of the epoxy components to obtain in a state of high purity. Silicic acid thin-layer chromatography (TLC) monitoring showed the presence of some impurity migrating at a faster rate than trivernolin, several trailing impurities, and an impurity which did not migrate from the base-line. Combinations of low temperature crystallizations, adsorbent treatments and silicic acid column chromatographic techniques were used to produce pure trivernolin.

Although 1,3-divernolin prepared as previously described was of 99% purity based upon oxirane oxygen value, column chromatography removed only the trace of yellow pigment present. Recrystallization from acetone at —20C removed an impurity which trailed the 1,3-divernolin in TLC examination. Recrystallization from petroleum ether at room temperatures, following the acetone crystallization, removed an impurity which did not migrate from the base-line and gave pure 1,3-divernolin.

Vernolic acid, although the last pure (93.5%), was the simplest of the epoxy compounds to refine; a single column chromatographic treatment served the purpose.

Each of the pure compounds when stored in vials at room temperature for a period of 60 days showed evidence of "instability" as demonstrated by thin-layer chromatography.

HYDROGENATION OF LINOLENATE. X. COMPARISON OF PRODUCTS FORMED WITH PLATINUM AND NICKEL CATALYSTS

C. R. Scholfield, R. O. Butterfield, V. L. Davison and E. P. Jones, Northern Regional Research Laboratory, Peoria, Ill.

One mole of hydrogen per mole of ester was added to methyl linolenate over a platinum catalyst at 20C and atmospheric pressure. The product was separated into trienoic, dienoic, and monoenoic esters by countercurrent distribution with acetonitrile and hexane. Each of these ester fractions was further separated by countercurrent distribution with methanolic silver nitrate and hexane. The final fractions were analyzed by gas chromatography, infrared and ultraviolet spectroscopy, alkali and lipoxidase isomerization, and oxidative cleavage, followed by measurement of the dibasic acids.

Comparison with previously described hydrogenations in which a commercial nickel catalyst at 140C and atmospheric pressure was used, shows that with platinum more stearate was formed and more triene remained; that is, the platinum hydrogenation was less selective than the nickel hydrogenation. Also, a smaller amount of *trans* esters was formed with platinum, and there was less shift of double bonds from the original 9, 12, and 15 positions. Other differences and similarities in products from platinum- and nickel-catalyzed reductions are also presented.

A CONVENIENT LABORATORY METHOD FOR PREPARING TRANS,TRANS-9,11-OCTADECADIENOIC ACID

Wilma J. Schneider and L. E. Gast, Northern Regional Research Laboratory, Peoria, Ill.

As a part of our study on Diels-Alder addition reactions with conjugated fatty acids, it was necessary to prepare pure *trans,trans*-9,11-octadecadienoic acid in quantity. The best approach was to dehydrate ricinelaidic acid or its methyl ester. Various dehydration catalysts were tried: sulfonated-polystyrene ion-exchange resin, zinc chloride, sodium bisulfate, boric acid, tetraboric acid, and boric oxide. When these catalysts are used, the yield of *trans,trans*-9,11-octadecadienoic acid ranged from 11–35%; however, thermal pyrolysis of a preformed polyester intermediate gave a higher yield of purer product than previously obtained. Based on this reaction, a convenient laboratory method for the preparation of *trans,trans*-9,11-octadecadienoic acid has been developed.

Ricinelaidic acid obtained by elaidinizing ricinoleic acid was heated to 235C under a vacuum of 5 mm for 3 hr to form a polyester of suitable molecular weight. Pyrolysis of this polyester with simultaneous distillation gave crude dehydrated acids. *Trans,trans*-9,11-octadecadienoic acid was obtained from the crude dehydrated acid mixture in an overall yield of 40–42% by crystallization from 95% ethanol. A study of variables affecting the pyrolysis reaction, i.e., temperature, pressure, time of reaction, and molecular weight of polyester, and leading to the best yields of *trans,trans*-9,11-octadecadienoic acid will be discussed.

Special Methods in Lipid Chemistry (Symposium)

STRUCTURAL INFORMATION ON LONG CHAIN FATTY MATERIAL OBTAINED RAPIDLY AND CONVENIENTLY BY THE USE OF X-RAY DIFFRACTION STUDIES OF SINGLE CRYSTALS OF UREA AND THIOUREA ADDUCTS

N. Nicolaides and Fritz Laves, Dept. of Biochemistry, University of Oregon Medical School, Portland, Ore.

X-ray diffraction patterns of single crystals of urea or thiourea adducts of long chain fatty material (channel type of inclusion compounds) yield diffuse, continuous layer lines instead of the usual spots obtained with ordinary crystals. These lines result from the fact that in adduct crystals there is order in one dimension only (the channel direction), whereas in ordinary crystals there is order in three dimensions, a condition which results in spots.

These continuous layer lines offer unique possibilities for structure determination of the included molecule (guest). From their spacing, i.e. their distance from the equator, one can quickly calculate the length of the guest. This in turn can give information on (1) degree of branching, (2) *cis-trans* isomerism if the substance is unsaturated and (3) molecular weight of the guest. From an evaluation of the intensities of the various orders of the layer lines, one can determine sub-periodicities on the chain of the guest or the position of a substituent group such as a methyl, hydroxyl or keto group.

These procedures are described.

FRACTIONATION OF LIPIDS BY COUNTERCURRENT DISTRIBUTION

Donald G. Therriault, U. S. Army Research Institute of Environmental Medicine, Natick, Mass.

The general theory of countercurrent distribution is presented. Its application to both analytical and preparative work, as well as the advantages and disadvantages of the method are discussed. Data is presented on the distribution of known lipid classes, the identification of new lipid classes, the use of derivatives as a means of fractionating lipids, and the separation of lipids with particular functions. Other possible applications of countercurrent distribution, such as its use in the study of associations of lipids with other substances, are discussed.

SPECIAL METHODS OF PURIFYING FATTY ACIDS

Nicholas Pelick, Robert S. Henly & Robert F. Sweeney, Applied Science Laboratories, Inc., State College, Penn.

Preparative gas chromatography and thin-layer chromatography have become important tools in purification of certain fatty acids. The most important applications are 1) separation of individual components from a small amount of a complex mixture, 2) any purification when only a very small quantity is available and 3) separation of components which are not amenable to more conventional methods such as distillation and crystallization.

One prime example of isolating small amounts of pure components from a complex mixture is in preparing uniformly tagged carbon-14 acids from milligram quantities of the mixed fatty acids. Although working with such small quantities is usually troublesome, the small quantity is actually an advantage when using gas chromatography.

An example of preparative thin-layer chromatography was encountered in making carboxyl-tagged cholesteryl palmitate. Only a very small quantity was available, and this was precious. After developing the sample on a special plate, two radioactive spots (cholesteryl palmitate and methyl palmitate) were located. The pure cholesteryl palmitate was simply recovered by scraping the spot off and extracting.

Gas chromatography in preparing gram and larger quantities of difficultly purified chemicals is used when more conventional means fail. The method is a last resort because of the many problems encountered. In addition to the usual problems of preventing decomposition, attaining good trapping recovery, etc, the preparation of fatty acid esters often has a peculiar problem of compatibility between the esters and the stationary phase. Many of the favorable separations may be attained only by using polyester phases. These materials allow only relatively small samples to be injected on each pass. In spite of these problems, many rare chemicals (e.g. methyl lauroleate) have been obtained by use of gas chromatography.

ACID-TREATED FLORISIL AS AN ADSORBENT FOR COLUMN CHROMATOGRAPHY

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Florisil, a commercial preparation of activated magnesium silicate, has been used for several years in this laboratory for the separation of neutral lipid classes. The order of elution of neutral lipids is the same as that observed with silicic acid but Florisil is easier to handle and permits faster flow rates because of its coarser mesh size. Free fatty acids and phospholipids are adsorbed more strongly on Florisil and although it is possible to recover fatty acids quantitatively following elution of the neutral lipids, attempts to separate and recover phospholipids were unsuccessful. (K. K. Carroll, J. Lipid Research *2*, 135, 1961).

Subsequent work has shown that Florisil can be modified by treatment with hot concentrated hydrochloric acid so that it can be used for separation and recovery of phospholipids as well as neutral lipids. The acid-treated Florisil separates phospholipids in much the same way as silicic acid and chemically it is probably almost identical to silicic acid but it retains the coarse mesh size and desirable flow-rate properties of Florisil. Although Florisil and acid-treated Florisil both consist of relatively coarse granules, the amount of adsorptive surface appears to be comparable to that of silicic acid in terms of the amount of lipid that can be separated without overloading the columns.

The following procedure is suggested for chromatographic separation of naturally-occurring lipid mixtures. The crude lipid extract is applied to a column of acid-treated Florisil and is eluted first with chloroform to obtain neutral lipid classes and free fatty acids as a group and then with increasing amounts of methanol in chloroform to separate and recover different classes of phospholipids. The chloroform eluent containing the neutral lipid classes and free fatty acids is subsequently separated on a Florisil column by the procedure already described.

(Continued on page 42)

NEW BOOKS

EINFÜHRUNG IN DIE LEBENSMITTELCHEMIE, by A. Beythien and W. Heimann (Theodor Steinkopff Pub., Dresden and Leipzig, *Germany*, 491 pp., 1961). This introduction to food chemistry is the fifth edition of the original introduction by A. Beythien of this subject. The present edition is divided into three main sections: one, the ground work for food chemistry; two, the individual food components; and three, the governmental regulations involved in food production.

The first section is divided into eight chapters which include the chemistry of the proteins, lipids, carbohydrates, minerals, and vitamins, the necessity for the food components, the enzymes involved in fermentation, the proximate composition of animal and plant tissue, the digestion of foodstuffs, and the preparation and preservation of foodstuffs.

The second section is divided into four main chapters. The first involves the individual food components such as meats, meat extracts, sea foods, eggs, dairy products such as milk, cheese, and butter, fats and edible oils, sugar, and products produced with a high proportion of sugar such as marzipan. The second chapter involves artificial sweeteners, alcoholic beverages, spices, essential oils, salt, vinegar, and the acids such as citric and lactic acids which are used in food preparation. The chapter is concluded with a discussion of coffee, tea, chocolate, and cola drinks. The third chapter involves a discussion of potable and mineral water and the fourth chapter a discussion of the materials which are used in cooking utensils.

The third and smallest section is devoted to two chapters on regulations involving food production. The first involves laws regulating food production and the second the analysis of food products for adulterants.

This book covers many fields of interest to food chemists and therefore cannot hope to be comprehensive in all of them. To the reviewer, the book represents a wealth of terminology which cannot be readily found in a standard German dictionary and thus would serve as a valuable reference book for someone who is interested in reading in context the current German literature in the field of food chemistry.

F. A. KUMMEROW,
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Urbana, Illinois

RADIATION CHEMISTRY OF POLYMERIC SYSTEMS. High Polymers, Vol. XV, by Adolphe Chapiro (Interscience Publishers, John Wiley and Sons, 712 pages, 1962, $21.00). From the point of view of the utilization of radiation as a synthetic tool, one area in particular, of radiation research, has commanded the broadest interest. This is the area concerned with the formation and modification of polymeric materials by irradiation. The need clearly existed for a moderately ambitious treatment of this subject, at which this book has been fairly successful.

The greatest virtue of Chapiro's book, to my mind, is that it makes a serious attempt to teach the polymer scientists some radiation chemistry before plunging into the main subject. Thus the first 120 pages, comprising three chapters, deal with the mechanism of energy dissipation, primary processes in radiation chemistry, and chemical effects in simple hydrocarbons, both gaseous and liquid.

This will be very useful to the general reader. But the reader should be forewarned that much of this part is unnecessarily lacking in rigour and detail. To cite a few examples. On page 39 the author, having pointed out that the energy per ion pair in most gases is approximately twice the ionization potential, concludes that "only about one-half of the absorbed energy can be accounted for by the formation of ions." This simple conclusion is based on the unwarranted assumption that one is dealing here only with molecule ions in the ground state of the ion. This brief treatment also neglects the obvious difference between the rare gases and the various polyatomic molecules listed. On page 40, a brief reference to "sub-excitation electrons" fails to explain the very essence of their role in multicomponent systems as enunciated by Platzman, where they are in fact defined in terms of E_2 (or I_2) $< E_e < E_1$, where E_2 is the excitation potential (and I_2 the ionization potential) of the lesser component. In the section on *Chemical Dosimetry*, the discussion of the Fricke dosimeter, now used so extensively, could have been included under *Absolute Dosimetry* and expanded. It would have been useful to point out to the reader that $[Fe^{2+}]$ may be conveniently measured photometrically at 304 $m\mu$ where ϵ is generally agreed to be 2200 M^{-1} cm^{-1}.

The three chapters on polymerization kinetics and mechanism are authoritative and carefully written. Certain sections, e.g., "Evidence of ionic mechanisms," are somewhat obsolete because of recent developments, but not seriously so.

In the three chapters on radiation effects in solid polymers, the various mechanistic concepts which have been developed are quite adequately presented. The important recent work on trapped radicals by ESR measurement deserves a more extensive treatment than is presented.

The two chapters on polymer solutions, particularly the last long chapter on graft copolymerization, are quite up to date and very well done, reflecting the author's own interests. This is a subject to which he has contributed a great deal of fine work.

On the whole, Chapiro has done a good job on this book. It comes fairly close to being worth the price—which is saying a great deal for a technical book these days.

L. M. DORFMAN, Chemistry Division,
Argonne National Laboratory, Argonne, Ill.

HYDROGEN COMPOUNDS OF THE GROUP IV ELEMENTS, by F. G. A. Stone, (Prentice-Hall, Inc., Edglewood Cliffs, N. J., 112 pp., 1962, $5.25). This small volume is divided into four chapters dealing with the hydrides of silicon, germanium, tin, and lead. Each chapter describes the preparation, properties, and derivatives of the particular metal hydride. The author has emphasized the literature of the past twenty years through 1961, but he has included sufficient references on the pioneering work in these areas to provide an adequate historical background. References (346 by number) are found in a separate section at the end of the book, conveniently arranged in alphabetical order according to authors.

The author has not been content to merely bring all the literature on these compounds together in a single volume, although this accomplishment alone would make the book valuable to anyone working in these areas. In addition, he has critically analyzed and correlated the available literature, and this becomes the most useful and valuable aspect of this book. In most instances, facts are accompanied by attempts to show how they are consistent with modern orbital theory, anticipated periodical variations, and, when available, thermochemical and spectral data.

This book will be valuable to the very limited number of people working in these areas and it will provide interesting reading for inorganic chemists in general.

TED J. LOGAN, The Procter &
Gamble Co., Cincinnati, Ohio

• *Obituaries*

H. E. Wilson (1942) Wharton, Texas passed away January 25, 1963. He served as Executive Secretary-Treasurer of International Oil Mill Superintendents' Assn, and as Editor of this organizations' publications, *The Oil Mill Gazetteer,* for approximately 50 years.

A. H. Preston (1947) died as a result of an automobile accident February 8, 1963. Mr. Preston was the President of Houston Laboratories, Houston, Texas.

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

• Fats and Oils

Beta-Apo-8′-carotenal—A new food color. J. C. Bauernfeind and R. H. Bunnell (Hoffmann-La Roche Inc., Nutley, N. J.). *Food Tech.* 6, 76–82 (1962). β-Apo-8′-carotenal, a new carotenoid made available by chemical synthesis, is an aldehydic carotenoid with vitamin A value that occurs widely in nature. Market forms of β-apo-8′-carotenal were prepared similar to those prepared for β-carotene, and included oil solutions, oil suspensions, emulsions and beadlets. These market forms were used to color various foods such as cheese, carbonated beverages, cakes, etc., a light to dark orange shade. Stability of the β-apo-8′-carotenal, in both the market forms and the various foods, was good. β-Apo-8′-carotenal should therefore find use in the food industry for coloring either water- or oil-base food where an orange to reddish-orange shade is desired.

Principles of column performance in large-scale gas chromatography. J. C. Giddings (Dept. of Chemistry, Univ. of Utah). *J. Gas Chromatog.* 1, 12–21 (1963). The loss of efficiency in large preparative columns is a combined result of many detracting influences. These are summarized and their importance is discussed. Particular attention is given to the way in which resolution is lost as column diameter is increased. Simplified theoretical treatments are given to show the variables which must be controlled for optimum performance. In particular the column plate-height contributions are formulated and shown to have altogether different and unusual properties when compared to analytical column characteristics. These results have many practical implications, and on this basis a number of approaches are suggested for improving the performance of large columns.

Gas chromatography of free fatty acids using Golay columns. W. Averill (The Perkin-Elmer Corp., Norwalk, Conn.). *J. Gas Chromatog.* 1, 22 (1963). When using Golay columns for the analysis of free fatty acids with such a liquid phase as Trimer Acid, definite tailing can be observed. In 1961 Averill demonstrated that with the addition of small amounts of highly polar components to the solution used in coating open tubular columns, the adsorption by the column wall can be prevented, resulting in symmetrical peaks. This technique was used in the present case. Best results were obtained by coating the Golay column 200 ft. long, 0.020 in. i.d. with a solution containing 10% Trimer Acid and 0.4% dinonylnaphthalenedisulfonic acid in toluene.

Some applications of an R.F. oscillator in gas chromatography. T. D. Andrew, C. S. G. Phillips, and J. A. Semlyen (Inorganic Chemistry Lab., Oxford, England). *J. Gas Chromatog.* 1, 27–30 (1963). A simple R.F. oscillator has been used as a flash vaporizer for liquid samples, for the pyrolysis of solid samples, to investigate discharge reactions and as an ionizing source in a modified argon detector. A relatively simple radio frequency oscillator has a number of useful applications in a gas chromatographic laboratory. Details of its construction are given and four examples given, namely (1) for the rapid vaporization of liquid samples on to a gas chromatographic column, (2) for the pyrolysis of solid samples at the inlet of a column, (3) to study the discharge reactions of small quantities of vapors, which may themselves be isolated from a gas chromatogram and (4) as an alternative to the normal radioactive ionizing source in an argon detector.

Gas chromatography of fatty acids and related long-chain compounds on phosphoric acid-treated columns. L. D. Metcalfe (Res. Div., Armour Ind. Chem. Co., McCook, Ill.). *J. Gas Chromatog.* 1, 7–11 (1963). Gas chromatograph columns using 20% diethylene glycol succinate polyester and 3% phosphoric acid as the liquid phase and coated upon 77% (w/w) 60–80 mesh Celite gave well-defined symmetrical peaks with fatty acids when using a thermal conductivity detector and a column temperature of 222C. Quantitative analysis using these columns varies from column to column; a series of calibration factors are required for each individual column. In addition to the analysis of fatty acids, the phosphoric acid-treated columns are useful in the analysis of other long-chain compounds, including nitriles, amides, and sulfonated compounds.

Capillary column gas-liquid chromatography with thermal conductivity detectors. R. D. Schwartz, D. J. Brasseaux, and G. R. Shoemake (Shell Development Co., Houston, Texas). *J. Gas Chromatog.* 1, 32–33 (1963). Capillary gas-liquid chromatographic columns may be used with small volume thermistor or hot-wire filament thermal conductivity detectors. Provided that the columns are not overloaded and that proper flow rates are maintained, the efficiency and resolution obtained are quite satisfactory. Samples of 1–10 microliters may be injected directly without the use of auxiliary splitting devices. Because of the sample size utilized and nondestruction by the detector, it is possible to trap fractions for analysis by mass, ultraviolet, or infrared spectroscopy or for further analysis with chromatographs fitted with more sensitive detectors.

In-place coating of the solid support for gas chromatography. W. Averill (The Perkin-Elmer Corporation, Norwalk, Conn.). *J. Gas Chromatog.* 1, 34–35 1963). In-place coating of the support material is a very practical technique; moreover, it also makes it possible to recoat packed columns in place. A solution of the liquid phase in a suitable solvent is placed in a reservoir and then forced through the column under pressure using an inert gas. After the excess solution is eluted, the column is heated to remove the solvent using an inert carrier gas. The proper concentration of the solution for best results depends on the viscosity of the solution as well as the surface characteristics of the solid support and must be determined by experimentation. The liquid/solid ratio in the column is reproducible on the same solid support material but will vary from support to support depending on its surface area and density. To determine this ratio, it is necessary to weigh the solid support placed in the column and to obtain the weight of liquid phase by extraction from the columns with solvent. The column can then be coated under identical conditions. A column may deteriorate or become contaminated such that it may no longer be useful. With this coating technique, the old coating may be removed with solvent and then be recoated with the same or a completely new liquid phase.

Studies on refining of cottonseed. III. Pilot plant studies on the effect of degumming and addition of chemicals on refining of crude cottonseed oil by wet and dry methods. A. Krishnamurthy and K. S. Murti (Oil Tech. Res. Inst., Anantapur, India). *Indian Oilseeds J.* 6, 14–19 (1962). The relative merits of wet and dry methods of refining crude cottonseed oil, the effect of degumming prior to alkali refining and the effect of certain chemicals on the refining characteristics of cottonseed oil were studied in the pilot plant. The dry method of alkali refining is found to be more advantageous than the wet method for cottonseed oil. Degumming the crude prior to refining results in less total refining loss and easier handling than straight refining of the oil. Addition of salicylic acid and tetrasodium pyrophosphate in small quantities during alkali refining reduces the refining loss significantly. The effect on color reduction of the final bleached oil is slight but not significant.

Lipids of epiphyseal cartilage. V. Zambotti, I. Cescon, B. Banfersoni, and L. Bolognani (Inst. of Chem. Biology, Univ. of Milan, Italy). *Experientia* 18, 318–19 (1962). The analysis of pooled samples of epiphyseal plates of newborn pigs was 6.60% lipid (2% referred to fresh tissue), total phosphorus 3.3%, lipidic phosphorus 0.70%, total nitrogen 8.37%, P/N 0.38%, total cholesterol 0.35%, free cholesterol 0.14%, saturated fatty acids 43.00, unsaturated fatty acids (mono) 47.50, and polyunsaturated fatty acids 9.50%.

Storage studies of cottonseed oil: II. Effect of storage on crude, refined, and bleached cottonseed oils. C. C. Ninan, A. Krishnamurthy, V. V. Krishna Rao, and K. S. Murti (Oil Tech. Res. Inst., Anantapur, India). *Indian Oilseeds J.* 6, 89–94 (1962). Storage studies on crude, refined, and bleached cottonseed oils were carried out for 6 months. Refining loss, color, and free fatty acid content increased during storage but not markedly. Color fixation takes place in crude oil. The extent of color fixation increases as time of storage increases. The bleachability of crude oil decreases correspondingly. Refined and bleached cottonseed oil does not undergo a reduction in color or an increase in free fatty acid content to any marked extent during storage.

Chemical composition of ground nut kernels and changes during storage. K. M. Ramanathan, R. Natarjan, and D. M. Samuel (Agr. Coll. and Res. Inst., Coimbatore, India). *Indian*

Oilseeds J. 6, 95–105 (1962). Three commercial types of peanuts (coromandel, peanut, and red natal) were stored in gunny, gunny lined with polythene, drum, and mud pot. Peanut and red natal types had a low free fatty acid content after storage in gunnies lined with polythene. Even though peanut type had a higher initial moisture content than coromandel, coromandel developed a higher free fatty acid content. A still lower initial moisture content is desirable for coromandel variety.

PLACE OF OILSEEDS IN INDIAN ECONOMY. M. S. Patel, and M. Ramakrishna Rao. *Indian Oilseeds J.* 6, 67–83 (1962). A summary is given of oilseed production and uses of meal and oil in India.

OBTAINING LIGHT-COLORED OIL AND SOAPSTOCK IN REFINING COTTONSEED OIL: II. CONSIDERATIONS IN REGARD TO THE USE OF HYDROGEN PEROXIDE AS ADDITIVE. P. L. Narayana Rao, K. T. Achaya, and S. H. Zaheer (Reg. Res. Lab., Hyderabad, India). *Indian Oilseeds J.* 6, 130–134 (1962). The color of refined and bleached oil is considerably improved by the use of hydrogen peroxide. The yield of such light-colored oil is higher. The color of soapstock is considerably improved. The operation needs no extra equipment beyond standard refining and bleaching units.

THE FORMATION OF CABONYL COMPOUNDS IN FATS AFTER IRRADIATION BY ELECTRON RAYS. H. Luck and R. Kohn (German Forschungsarstalt für Lebensmittelchemie, München, Germany). *Experientia* **18**, 62–63 (1962). Investigations of the formation of carbonyl compounds in fats subjected to electron rays (100 M. rad.) have been reported. More than 20 substances can be determined by GLC of irradiated samples. The splitting of the ester bonds is often accompanied by a fracture of the fatty acid chain and carbonyl compounds with 6–12 carbon atoms are formed (aldehydes mostly up to 9 carbon atoms). A relatively high concentration of unsaturated C_9 ketones and a small amount of ketones with more than 13 carbon atoms have been found in irradiated tripalmitin and palmitic acid. None of these higher compounds could be detected in unsaturated oils, which contained more C_{11} and C_{12} ketones.

THE FATTY ACIDS IN THE CHLOROPLASTS FROM SPINACIA OLERACEA. H. Debuch (Physiological-Chemical Inst. of Kolm Univ., Germany). *Experientia* **18**, 61–62 (1962). The fatty acids of two samples of chloroplasts from *Spinacia oleracaē* have been investigated quantitatively. They contain more than 70% polyenoic fatty acids. A high content of hexadecetrienoic acid was observed. The presence of Δ^3-*trans*-hexadecenoic acid was detected.

QUANTITY AND FATTY ACID COMPOSITION OF LIPID EXTRACTED FROM CELLS OF STREPTOCOCCUS LACTIS. P. Macleod, R. Jensen, G. Gander, J. Sampugna (Univ. of Conn., Storrs, Conn.). *J. Bact.* **83**, 806–10 (1962). A method for extraction of lipid from bacterial cells is described. Lysozyme-treated cells of *S. lactis* yielded 5%, while untreated cells yielded 3% of their dry weight as lipid. More than two-thirds of the bacterial lipid was tentatively classified as phospholipid. There were five major fatty acids present in all lipid fractions, myristic, palmitic, palmitoleic, lactobacillic, and an 18 carbon acid which may be oleic or one of its isomers. Lactobacillic acid accounted for 20% of the fatty acids of *S. lactis* and may prove to be a ready source of this rare acid.

ACTIVATION OF METAL HYDROGENATION CATALYSTS BY IRRADIATION. D. Graham (Jackson Lab., E. I. du Pont de Nemours and Co., Wilmington, Del.). *J. Phys. Chem.* **66**, 510–511 (1962). The catalytic activities of supported metal hydrogenation catalysts (nickel, palladium, and platinum) have been increased by high energy irradiation. The improvement sometimes exceeded 20%. Active centers appeared to be both created and destroyed by irradiation. In general, the energy required for forming new centers and also the stability of these centers vary directly with the temperature required for diffusion of the atoms in the surface of the catalyst metals, which temperature is a function of their melting points.

FAT CONTENT AND FATTY ACIDS IN SOME COMMERCIAL MIXES FOR BAKED PRODUCTS. Rosemarie Ostwald (University of California, Berkeley). *J. Am. Dietet. Assoc.* 42, 32–6 (1963). A number of commercial mixes for baked products have been analyzed for their fat content and for their fatty acid composition. The fat content of most was in the range called for by the respective recipes, from 5% for certain cakes to 34% for pies. A large number of mixes (21 out of 41 analyzed) contained about 25% palmitic, 20% stearic, 40% oleic, and 10% linoleic acids. The proportion of the major fatty acids, palmitic and stearic, to the unsaturated, primarily oleic and linoleic, ranged from a ratio of 20:80 to 80:20, depending on the kind of fat used. Linoleic acid content ranged from 0.8% for a product containing coconut to 55% for a chiffon cake customarily made with oil. Palmitic acid content ranged from 10 to 35% and stearic acid from 3 to 30%.

THE FATTY ACIDS OF NEEM OIL AND THEIR REDUCTION PRODUCTS. J. H. Skellon, S. Thorburn, J. Spence, and S. N. Chatterjee (Brunel College, London). *J. Sci. Food Agr.* **13**, 639–43 (1962). The seed oil of *Melia (Azadirachta) indica* has been re-examined particularly with reference to fatty acid composition, using gas chromatography. Behenic acid has been found to be a component, and the presence of myristic acid and lignoceric acid verified. A suitably purified neem oil has been successfully reduced to the corresponding saturated and unsaturated alcohols by a selective sodium reduction procedure of potential industrial value. The fatty acid composition as determined by gas chromatography is compared with values previously reported in the literature.

METHOD OF PRODUCING A CAKE BATTER. E. Handschumaker and H. G. Hoyer (Textron, Inc.). *U. S. 3,069,270.* A liquid shortening (a normally liquid edible triglyceride oil) and from 0.50 to 1.75% by weight of normally solid monoglycerides of a saturated fatty acid having a chain length greater than 18 carbon atoms are heated to about 140F to melt the solid monoglyceride. The solution is cooled to reprecipitate the monoglycerides in the active form. The cooled liquid shortening containing the reprecipitated monoglyceride is then incorporated into the cake batter within about 45 minutes of reprecipitation.

COACERVATION PROCESS FOR ENCAPSULATION OF LIPOPHILIC MATERIALS. E. H. Jensen and J. G. Wagner (Upjohn Co.). *U. S. 3,069,370.* A process for the encapsulation of finely divided lipophilic material in which the capsule mantle consists of a polymer includes the following steps: (1) preparing a dispersion of lipophilic material such as fats, oils, waxes or wax-like solids, in an aqueous liquid solution of a styrene-maleic anhydride copolymer, the anhydride groups of which are at least 50% hydrolyzed, (2) adding insolubilizing salt until a visible clouding is observed, (3) adjusting the pH of the mixture until an opacity in the mantle is observed, and (4) separating the thus-produced capsules from the aqueous liquid.

METHOD OF TREATING SOYBEAN OIL. N. H. Witte and E. Sipos (Central Soya Co.). *U. S. 3,069,443.* A method of treating soybean oil to improve its quality and to recover a xanthophyll concentrate as a by-product consists of: (1) extracting crude degummed soybean oil containing xanthophyll with methanol at a temperature between 40 and 140F; (2) continuing the extraction until at least a major portion of the xanthophyll has been extracted into the methanol phase while the methanol phase contains less than 2% by weight of the triglycerides of the soybean oil; (3) separating the xanthophyll-containing methanol phase from the oil phase; (4) removing the methanol to give a xanthophyll concentrate; and (5) removing the methanol from the oil phase to obtain a soybean oil product with improved quality.

PROCESS FOR THE PREPARATION OF A COCOA BUTTER SUBSTITUTE AS WELL AS FOR THE PREPARATION OF CHOCOLATE AND THE SHAPED PRODUCTS THUS OBTAINED. Y. A. Sinnema (N. V. Twincon, Koog-Zaandijk). *U. S. 3,070,445.* A cocoa butter substitute from Mowrah fat which, when mixed with cocoa butter in any given proportion gives a mixture with properties (solidification point, dilatations, iodine value) which lie between those of the substitute and those of cocoa butter, comprises a monounsaturated triglyceride fraction of Mowrah fat obtained by removing from the Mowrah fat at least 60% of the weight of the lowest-melting fraction by fractionation with a total quantity of 2 to 20 liters of acetone per kg. of the fat at a temperature between 0 and 12C.

PROCESS OF PREPARING AN HERB-FLAVORED EDIBLE OIL. Edalene Stohr (American Can Co.). *U. S. 3,071,475.* The process of preparing an herb-flavored edible oil in individual consumer-size containers comprises the following steps: forming a mixture of uniformly commingled herb flakes of diverse flavors, separating the mixtures into distinct portions, pulverizing one of the portions to a powder-like consistency, feeding measured quantities of each of the pulverized and the flake portions into each of the containers, and adding to each container a predetermined quantity of an edible oil in a heated state. The heated oil extracts flavor principally from the pulverized portion which becomes substantially imperceptible and suspends the flake portion randomly and perceptibly in it.

Customer Service Report No. 1536-F

The Case of the Hot Spot Gunk

The sample from the rendering plant was labeled *"gunk deposited on tank from 3 to 9 o'clock."*

An accompanying letter explained that this black, gummy material was a wall deposit found in certain areas of a resin-coated iron storage tank for lard. The customer also sent samples of the resin with which the tank was coated.

The customer had contacted us, naturally, because a possibility existed that the trouble was being caused by the Tenox 2 antioxidant used to stabilize the lard. Our laboratory went to work, and this is what they found:

A deposit similar to the black gunk was formed when lard was held in contact with iron at 400°F. for 30 hours. Treating the iron with the coating resin minimized the deposit, as did treating the lard with Tenox 2. When temperatures were maintained at 200°F. for 30 days, no gunk formed under any circumstances. The lab concluded: *"...the gunk is caused by local overheating in the storage tank."*

And so it proved to be. Knowing the cause of the trouble, the customer was able to eliminate the hot spots—and the gunk.

**IT PAYS TO
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• Fatty Acid Derivatives

MONOLAYERS OF MYRISTYL, AND CETYL ESTERS OF OXALIC, MALONIC, SUCCINIC, GLUTARIC, ADIPIC, AND PIMELIC ACIDS. J. L. Shereshefsky, H. T. Carter, E. Nichols, and P. L. Robinson (Chem. Dept. Howard University, Washington, D. C.). *J. Phys. Chem.* **66**, 1846–50 (1962). Monolayers of dicetyl esters of the first six members of the homologous series of dicarboxylic acids were studied, including dimyristyl oxalate, monocetyl oxalate, and monocetyl malonate. The study included measurements of surface pressure vs. area and surface potential vs. area. Monolayers are dependent upon the concentration of the spreading solution, and also on the maximum spreading area. These substances form condensed and liquid-expanded films; vapor-expanded films tend to form when spread from very dilute films. The oxalates tend to form a condensed film of high stability that folds upon itself. Orientations of the head-groups are given. The calculated vertical components of the dipole moments are in good agreement with the measured values.

PREPARATION OF MONOGLYCERIDES FROM CASTOR OIL. I. P. K. Kochhar, S. K. Dey, and P. K. Bhatnagar (Shri Ram Inst. for Ind. Res., Delhi, India). *Indian Oilseeds J.* **6**, 20–23 (1961). Monoglycerides are prepared from castor oil by a unique process. A 2:1 ratio of 98% glycerol and refined castor oil is charged into a autoclave. Carbon dioxide is bubbled through the oil at 50C to remove air. The reaction is carried out at 250C with 200 p.s.i.g. of carbon dioxide for two hours. Gas pressure was then released and the charge cooled to 150C, held for 15 min, then finally cooled to 50C. Residual glycerol was removed by water washing. The composition after removal of glycerol was analyzed to 91% α monoglycerides. The process is applicable to other fats.

II. EFFECT OF TEMPERATURE, TIME AND PRESSURE. P. K. Kochhar, S. K. Dey, and P. K. Bhatnagar. *Ibid.* 144–152. Superatmospheric pressure (50–200 p.s.i.g.) that retains the water normally present in the commercially pure glycerol is highly conducive to the attainment of high monoglyceride concentration at equilibrium. The rate of formation and the concentration of monoglycerides at equilibrium increase rapidly with temperature above 200C. Heating beyond equilibrium at 250C. or over causes low analysis of monoglycerides. Heating for 15 minutes either at 250C or below beyond equilibrium and after the release of pressure is quite sufficient to reduce the free fatty acid content of the product to a low value. Yields over 90% are achieved.

ON THE DAMPING OF WATER WAVES BY MONOMOLECULAR FILMS. F. C. Goodrich (Calif. Res. Corp., Richmond, Calif.). *J. Phys. Chem.* **66**, 1858–1963 (1962). Some features of a generalized theory of monolayer rheology are described, with particular reference to the problem of wave damping by surface-active substances. Experiments on wave attenuation by fatty alcohols are reported and discussed in terms of theory.

SPREADING AND COLLAPSE PHENOMENA IN THE FATTY ALCOHOL SERIES. J. H. Brooks and A. E. Alexander (Dept. Phys. Chem., Univ. of Sydney, N.S.W., Australia). *J. Phys. Chem.* **66**, 1851–53 (1962). The formation of monolayers of pure fatty alcohols by spreading from the crystal and their behavior at the air-water interface was studied. The existence of hydrates of fatty alcohols was shown.

SYNTHESIS OF ESTERS FROM SIMPLE ALKYL HALIDES AND TERTIARY AMINE SALTS OF CARBOXYLIC ACIDS. R. H. Mills, M. W. Farrar, and O. J. Weinkauff (Monsanto Chemical Co.). *Chem. & Ind. (London)* **1962**, 2144. The reaction is carried out by heating approximately stoichiometric amounts of a mixture of the carboxylic acid, alkyl halide, and tertiary aliphatic amine (usually triethylamine) for a few hours after which the ester is washed free of the amine hydrohalide and purified by any convenient method such as distillation, crystallization, etc. Best results are obtained with primary halides; secondary halides give high yields by employing a longer reaction time. Tertiary halides generally give a poor yield. In general, the reactivity of the halogen is in the order: $I > Br > Cl$.

SELECTIVE ALCOHOLYSIS OF EPOXY FATTY ACID ESTERS. F. E. Kuester and J. L. Ohlson (Swift & Co.). *U. S. 3,070,608.* A method for preparation of esters of oxirane containing fatty acids by alcoholysis consists of the following steps: mixing oxirane containing higher fatty acid ester or an unsubstituted aliphatic alcohol selected from the group consisting of mono-, di-, and polyhydric alcohols, the alcohol having from 1–20 carbon atoms and containing no more than 6 hydroxyl groups, *an excess of a material selected from the group consisting of* unsubstituted aliphatic alcohols having from 1–20 carbons and no more than 6 hydroxyl groups, and tertiary amine substituted aliphatic alcohols in which the substituents on the amine are hydroxy alkylene groups, and an alkaline interesterification catalyst at a temperature sufficient to maintain refluxing but not in excess of 125C.

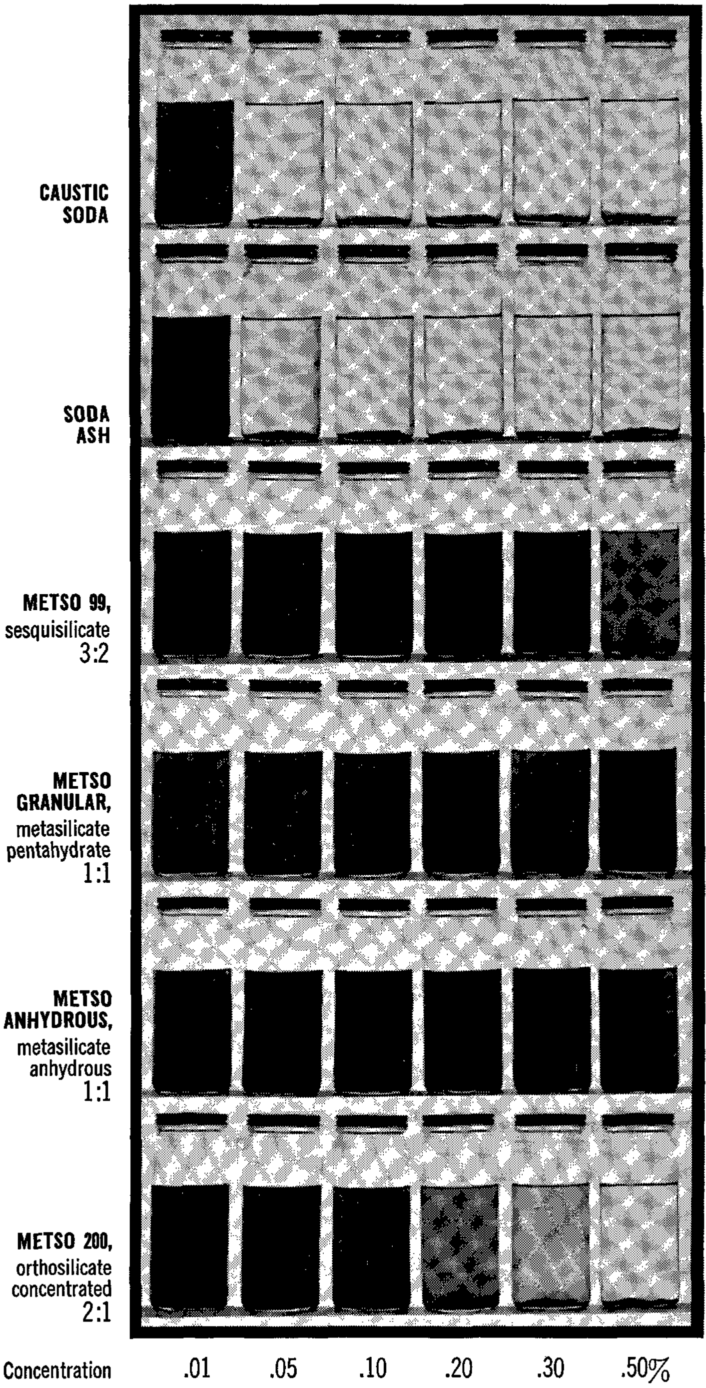
PREPARATION OF LIGHT-COLORED FATTY ACID ESTERS. A. G. Mohan and W. R. Christian (Nopco Chemical Co.). *U. S. 3,071,604.* From 0.05 to 2.00% of hydrophosphorus acid (on weight of fatty acid and alcohol) is added to a mixture of at least 1 fatty acid, at least one member of the group consisting of monohydric and polyhydric aliphatic alcohols having the hydroxyl substituent as the sole reactive substituent, and containing from 1 to 20 carbon atoms, and an esterification catalyst. The mixture is esterified, the acid material contained in the resultant product is neutralized with a base, and a substantially color-free ester is recovered.

• Biology and Nutrition

FAT TRANSPORT IN THE LOCUST. A. Tietz (Israel Inst. for Bio. Research, Ness-Ziona, Israel). *J. Lipid Research* **3**, 421–426 (1962). When fat-body tissue from locusts was incubated with palmitate-1-C^{14} in phosphate-saline, the acid was readily taken up by the tissue; 80–90% of that taken up was esterified and recovered in the glyceride fraction. When the prelabeled tissue was incubated in hemolymph, glycerides were released from the tissue into the medium. The effect of hemolymph was specific; glycerides were not released into phosphate-saline, bovine serum, or buffered solutions of bovine serum albumin or egg albumin. The release of glycerides was inhibited by fluoride and by cyanide. The amount of glyceride released was proportional to the amount of hemolymph that was added. Effectiveness of the hemolymph in this regard was not affected by prolonged dialysis, but was destroyed by heating. The specific activity of the glycerides released was at least 10 times higher than the average specific activity of the glycerides inside the tissue. A considerable fraction of the released glycerides was incorporated into the lipoprotein fraction of the hemolymph. Uptake of glycerides by body-fat tissue was also demonstrated.

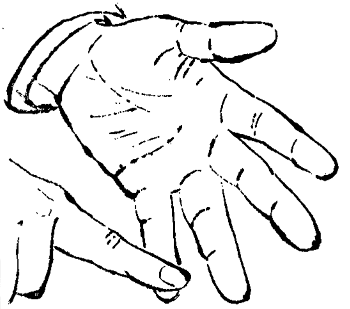
THE INFLUENCE OF EXOGENOUS CHOLESTEROL ON HEPATIC LIPID COMPOSITION OF THE RAT. R. J. Morin, S. Bernick, J. F. Mead, and R. B. Alfin-Slater (Dept. of Physiological Chem., School of Med., Univ. of Calif., Los Angeles). *J. Lipid Research* **3**, 432–438 (1962). Rats were fed diets containing cottonseed oil and/or cholesterol, cholic acid, methyl esters of long-chain fatty acids, and tocopherol. Livers, hearts, and aortas were examined histologically; liver lipids were fractionated by silicic acid chromatography; and the fatty acid composition of the sterol esters, triglycerides, and phospholipids were determined by gas-liquid chromatography. The degree of deposition of liver sterol esters seemed to be related to the availability of dietary fatty acids for esterification with exogenous cholesterol. No differences in sterol ester deposition were noted among the groups in which the dietary cholesterol was supplemented with methyl esters of fatty acids of different degrees of unsaturation. Fatty acid analysis revealed an accentuation by cholesterol feeding of the increase in monoenoic fatty acids and the decrease of linoleic and arachidonic acids characteristic of essential fatty acid deficiency. Two eicosatrienoic acids, occurring primarily in the phospholipids, were identified. The 5,8,11-eicosatrienoic acid appears to be related to essential fatty acid deficiency, whereas the 9,11,14-isomer is probably an intermediate in the conversion of linoleic to arachidonic acid and is increased when exogenous cholesterol induces an increased arachidonic acid synthesis.

TWO-DIMENSIONAL THIN-LAYER CHROMATOGRAPHY OF RAT LIVER PHOSPHATIDES. W. D. Skidmore and C. Entenman (U. S. Naval Radiological Defense Lab., San Francisco, Calif.). *J. Lipid Research* **3**, 471–475 (1962). A system of two-dimensional thin-layer chromatography was developed that separated rat liver phosphatides into several phosphate-positive spots in about 2 hr. developing time. Characteristic hydrolysis products derived from phosphatidyl serine, phosphatidyl ethanolamine, phosphatidyl inositol, phosphatidyl choline, sphingomyelin, and lysophosphatidyl choline were identified. The hydrolytic products of "phosphatidic acid" were not definitely characterized. The application of thin-layer chromatography as described for rat liver phosphatides can be extended to phosphatide extracts of other tissues.



Silicate alkalies of 3:2, 1:1 and 2:1 mol. ratio used in comparison with alkalies that do not contain soluble silica. In 300 ml. of distilled water 0.3 gram of burnt umber was used in each series. Amounts of alkali added to the bottles are noted below the photo. After shaking, bottles stood 19 hours at 140 degrees F.

Both caustic soda and soda ash permitted the soil used in the test to settle out promptly. With the meta and sesqui silicates even after 19 hours soil particles remained suspended. Same is true with orthosilicate over a part of the concentration range.



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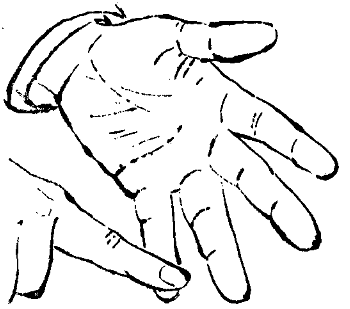
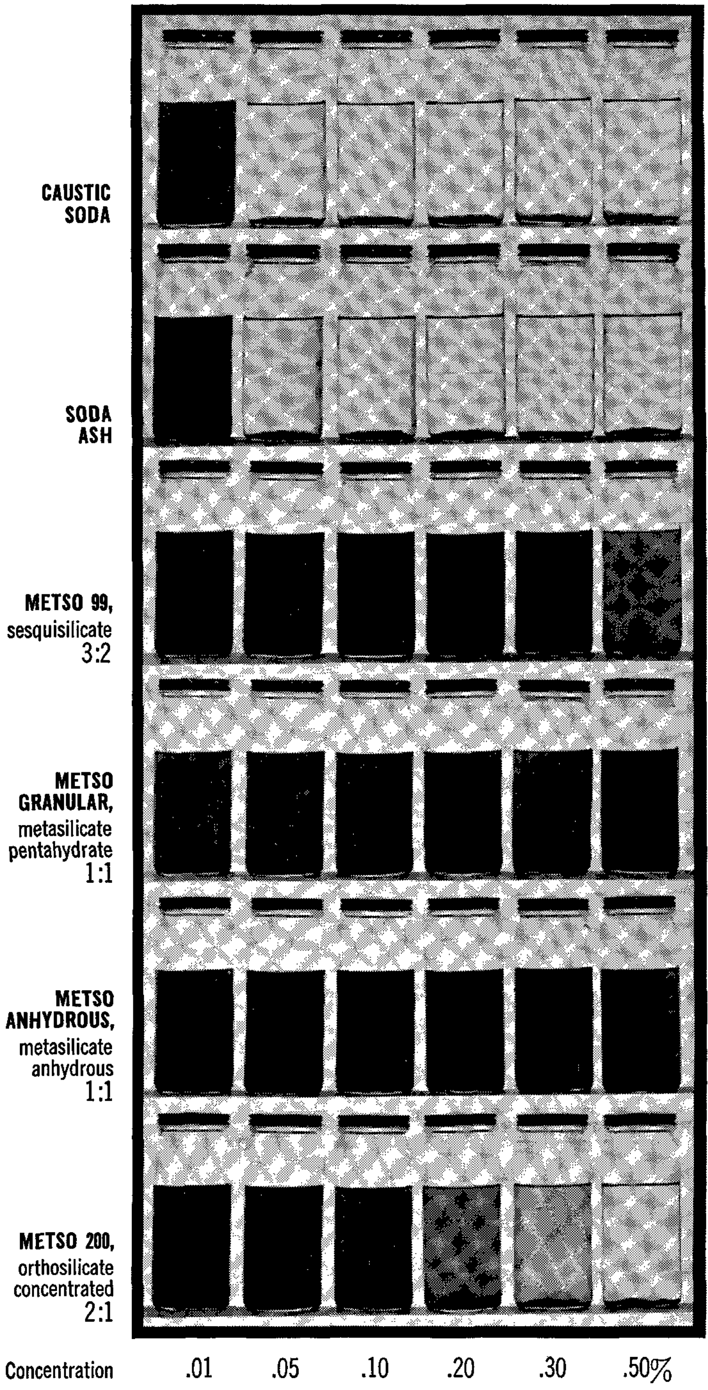


how PQ Silicates aid detergency by SUSPENDING SOIL



Silicate alkalies of 3:2, 1:1 and 2:1 mol. ratio used in comparison with alkalies that do not contain soluble silica. In 300 ml. of distilled water 0.3 gram of burnt umber was used in each series. Amounts of alkali added to the bottles are noted below the photo. After shaking, bottles stood 19 hours at 140 degrees F.

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SEPARATION OF PHOSPHATIDYL ETHANOLAMINE, PHOSPHATIDYL SERINE, AND OTHER PHOSPHOLIPIDS BY THIN-LAYER CHROMATOGRAPHY. V. P. Skipski, R. F. Peterson, and M. Barclay (Div. of Exptl. Chemotherapy, Sloan-Kettering Inst. for Cancer Research, Rye, N.Y.). *J. Lipid Research* 3, 467–470 (1962). Thin-layer chromatography of phospholipids and cerebrosides was performed on Silica Gel G plates using a mixture of chloroform-methanol-acetic acid-water as development solvent. Two types of chromatoplates were used: "neutral" plates, prepared from Silica Gel G slurry made in water, and "basic" plates, prepared from Silica Gel G slurry made in 0.01 M sodium acetate or sodium carbonate solutions. Only chromatograms run on "basic" plates showed good and reproducible separations of phosphatidyl serine from other phospholipids, independent of the amount of phosphatidyl serine present in the sample. However, "neutral" plates gave better separation of cerebrosides from phospholipids. A practical method of applying these systems for separation of phospholipids extracted from rat liver and human serum is presented.

THE DIFFERENTIAL MIGRATION OF STEROL ACETATES ON SILICA GELS AND ITS APPLICATION TO THE FRACTIONATION OF STEROL MIXTURES. P. D. Klein and P. A. Szczpanik (Div. of Biological and Med. Research, Argonne Nat'l Lab., Argonne, Ill.). *J. Lipid Research* 3, 460–466 (1962). The retention volumes of 12 sterol acetates relative to cholesteryl acetate were determined on four well-characterized silica gels of different body structure. These values illustrate the contribution of silica gel structure to the chromatographic process and permit the selection of appropriate gels for a given separation. The application of these values to the fractionation of model and biological mixtures of sterol acetates is illustrated.

THE STRUCTURE OF PLASMALOGENS: VI. CONFIGURATION OF THE DOUBLE BOND IN THE α,β-UNSATURATED ETHER LINKAGE OF PHOSPHATIDAL CHOLINE. W. T. Norton, E. L. Gottfried, and M. M. Rapport (Dept. of Biochem., Neurology, and Med., Albert Einstein College of Med., Yeshiva Univ., N. Y. 61, N.Y.). *J. Lipid Research* 3, 456–459 (1962). The infrared data permit the assignment of the *cis* configuration to the double bond in the α,β-unsaturated ether linkage.

THE LIPOLYTIC ACTIVITY OF RAT KIDNEY CORTEX AND MEDULLA. C. H. Hollenberg and I. Horowitz (Univ. Med. Clinic, The Montreal Gen. Hosp., Montreal 25, Canada). *J. Lipid Research* 3, 445–447 (1962). Slices of rat kidney medulla and cortex were incubated for 30 min. in a triglyceride medium with and without heparin; the slices were then removed and the lipolytic activity of the medium measured over the following 30 min. Heparin markedly increased the activity in the medium when medullary slices were used; it had a much smaller effect with cortical preparations. Protamine sulphate and 1 *M* sodium chloride inhibited the activity released by medullary slices. Homogenates of rat kidney medulla hydrolyzed activated triglyceride to a greater extent than nonactivated substrate. The activity of medullary homogenates was enhanced by heparin and inhibited by protamine and strong salt solutions. Cortical homogenates hydrolyzed activated substrate to only a slightly greater extent than nonactivated substrate, and the activity of these homogenates was not affected by heparin or by protamine sulphate. The results suggest that lipoprotein lipase is present in rat kidney medulla and that a lipase differing from this enzyme in a number of respects is present in rat kidney cortex.

METABOLISM OF LABELED LINOLEIC-1-C^{14} ACID IN THE SHEEP RUMEN. R. D. Wood, M. C. Bell, R. B. Grainger, and R. A. Teekell (Agr. Res. Lab., Univ. of Tennessee, Oak Ridge). *J. Nutrition* 79, 62–68 (1963). Four western wethers with ligated reticulo-omasular orifices were used to conduct rumenal fat studies using labeled linoleic-1-C^{14} acid. Tagged compounds appeared in the jugular blood about 4 hours after dosing and increased rapidly between the eighth and twelfth hours. Most of the activity appeared in the non-steam distillable fraction, indicating that the compound absorbed from the rumen contained 10 or more carbons. About 0.3 to 0.5% of the total dose appeared in the rumen mucosa and muscularis. Activity also appeared in the liver, lungs, kidneys, spleen, and kidney fat. Of the total dose, from 0.6 to 1.0% was degradated in the rumen. Eighty-five to 96% of the dose was recovered from the rumen of which only 3 to 6% was the original linoleic acid. Approximately 45% was hydrogenated to saturated acids, whereas 33 to 50% had been hydrogenated to oleic or elaidic acids. Unidentified acids accounted for only 5 to 17% of the recovered dose.

DNA SYNTHESIZING CELLS IN RABBIT HEART TISSUE AFTER CHOLESTEROL FEEDING. S. C. Spraragen, V. P. Bond, and L. K. Dahl (Med. Research Center, Brookhaven Nat'l. Lab., Upton, Long Island, N. Y.). *Circulation Res.* 11, 982–986 (1962). Suggestive evidence that cholesterol feeding may increase the number of thymidine-H^3 labeled cells in the heart tissues of rabbits was presented. This increment appeared to be primarily restricted to the interstitial cells and was not related to associated degenerative, necrotic, or inflammatory changes. Evidence of DNA synthesis was also found in capillary endothelial cells and, rarely, in what were considered to be myocardial muscle cells.

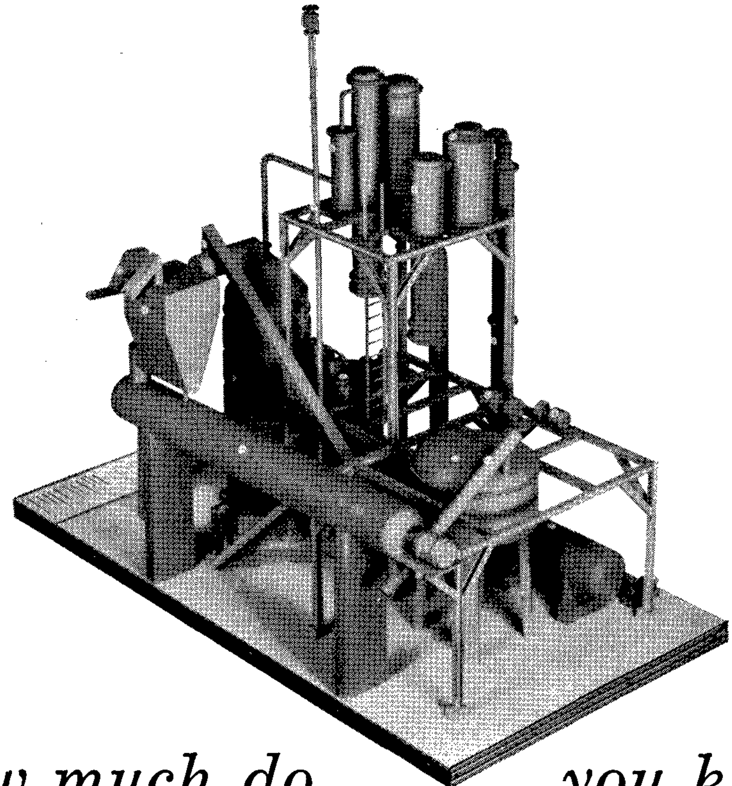
THE SUPPLY OF PRECURSORS FOR THE SYNTHESIS OF FATTY ACIDS. A. F. Spencer and J. M. Lowenstein (Graduate Dept. of Biochem., Brandeis Univ., Waltham 54, Mass.). *J. Biol. Chem.* 237, 3640–3647 (1962). The rapid incorporation of citrate into fatty acids has been demonstrated with high-speed supernatant preparations of lactating mammary gland. The rate of incorporation of citrate is faster than that of acetate in the presence or absence of citrate or malonate. Experiments with asymmetrically labeled citrates show that the incorporation of citrate proceeds chiefly via acetyl coenzyme A produced on the citrate cleavage enzyme reaction, but a small percentage is incorporated via oxaloacetate produced in the same reaction. Malonate is incorporated into fatty acids at an appreciable rate, which is, however, slower than the rates of incorporation of citrate, or of acetate in the presence of citrate or malonate. The rate-limiting step in the incorporation of malonate is the activation of malonate to malonyl coenzyme A.

INFLUENCE OF PYRIDOXINE AND DIETARY FAT ON THE DISTRIBUTION OF SERUM FATTY ACIDS IN DOGS. L. Söderhjelm (Bruce Lyon Memorial Res. Lab. of the Children's Hosp. of the East Bay, Oakland, Calif., and the Dept. of Nutritional Sciences, Univ. of California, Berkeley). *J. Nutrition* 78, 438–444 (1962). Studies were performed concerning the relative distribution of fatty acids in the blood serum of 6 adult Beagle dogs, two German short-hair puppies, and 6 mongrel puppies that received diets deficient in linoleic acid or vitamin B_6 or both. The presence or absence of linoleic acid caused marked changes in the fatty acid spectrum in the serum, whereas the presence or absence of pyridoxine in the diet did not influence the relative content of arachidonic acid in the blood serum. With two rapidly growing young animals (the German short-hair puppies), however, it was noted that arachidonic acid increased somewhat more rapidly in the serum of the animal receiving pyridoxine in the diet than in the littermate fed a diet without pyridoxine, although the magnitude of change was about the same. Apparently the conversion of linoleic acid to arachidonic acid is possible without the presence of vitamin B_6 in the diet.

INFLUENCE OF LINOLEIC ACID CONTENT OF MILK LIPIDS ON OXIDATION OF MILK AND MILK FAT. L. M. Smith, W. L. Dunkley, and M. Ronning (Dept. of Food Science and Tech. and Animal Husbandry, Univ. of Calif, Davis). *J. Dairy Science* 46, 7–10 (1963). The concentration of linoleic acid in milk lipids was increased by infusing two cows with a cottonseed oil emulsion, and changes in the oxidative stability of the milk and milk fat were determined. Infusing 150 g of cottonseed oil (as Lipomul I.V.) tripled the linoleic acid content of the milk fat in the next milking. Linoleic acid in the milk phospholipids increased to a smaller extent, and not until the second milking after the infusion. Little change was noted in the concentration of other fatty acids. No significant change occurred in milk production, fat percentage, or the amounts of copper, tocopherols, and carotenoids. A decrease in oxidative stability of the milk fat appeared to be related to the increase in its linoleic acid content. Furthermore, an increase in susceptibility of the milk to copper-induced oxidized flavor, as measured by the thiobarbituric acid test, appeared to be dependent on increased linoleic acid content of the phospholipids rather than the milk fat.

UTILIZATION OF ALFALFA CAROTENE AND VITAMIN A BY GROWING CHICKS. D. B. Parrish, R. A. Zimmerman P. E. Sanford, and E. Hung (Kansas State Univ., Manhattan). *J. Nutrition* 79, 9–17 (1963). A comparison was made of utilization of vitamin A activity of the ANRC vitamin A standard, USP reference solution and low and high quality alfalfa meals. Effect of furazolidone and ethoxyquin on utilization of vitamin A activity of alfalfa meal also was studied. Furazolidone did not affect utilization of provitamin A of alfalfa meal. Ethoxyquin at 0.02% of diet did not significantly affect gain, or serum and liver vitamin A levels; gains were improved when 0.10% of ethoxyquin was used.

(Continued on page 40)



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Essential Oils . . .

(Continued from page 6)

fume chemicals (compatibility), etc., must be tested repeatedly, and the material should pass routine tests for non-irritation of human skin.

The commercial production may turn out slightly different from the small-scale synthesis, and a typical problem arises; can we establish specifications and can we correlate these with acceptable odor of this new product? Chemical analysis is an inevitable and necessary companion to the odor evaluation of all synthetic perfume chemicals. It is well known, although not always understood by other industries, that the perfume industry emphasizes odor purity above chemical purity in the sense that impurities which do not impair the odor, the effect or the stability of a perfume chemical, may be accepted if the odor of the chemical is accepted by experts in this olfactory evaluation. A product consisting of 99% Octyl aldehyde and 1% Octanoic (caprylic) acid will most conceivably be turned down on olfactory evaluation, while a product consisting of 99% Octyl aldehyde and 1% Octyl alcohol may pass the olfactory test. Analysis will, of course, show the difference, but the manufacturer may be surprised to know that 99% aldehyde is not always a guarantee of perfumery purity.

Thus, to convert the natural raw materials and the basic chemicals into suitable perfumery materials, skill and experience from organic chemists, analytical experts, extraction and distillation specialists are employed by the suppliers of such raw materials, and by the larger manufacturers of finished perfumes. Uniformity and high olfactory quality of raw materials is the beginning and the background for success in the creation of good fragrances.

Among the most important perfume types is the soap perfume. Important not only because of its considerable volume when compared to other perfume types, but particularly in view of the fact that every 2500 pounds of soap perfume helps sell one million bars of soap—or more. The soap perfume may very well mean the difference between a mediocre sales figure from the soap factory—or a mushrooming success in the sale of a soap bar.

The creation of a successful soap perfume may demand as much art and imagination but considerably more technical and scientific skill and experience than is normally required of the perfumer who specializes in handkerchief perfumes. Soap is a chemically active material containing several additives which may further affect the perfume: fillers, stabilizers, rancidity inhibitors, deodorants, etc.

Handkerchief perfume is a solution of perfume oil (the "compound") in Ethylalcohol, and this type of perfume is therefore comparatively stable, provided the original oil is so. Competition within the soap and detergent industry is the main factor deciding the low cost of a soap perfume. However, the soap manufacturers realize that the success of a soap is highly dependent upon the fragrance, and therefore they can see the advantage of having the perfume amount to a considerable percentage of the total cost of the soap.

The soap perfumer is therefore faced with a many-sided problem: odor and color stability of the materials, compatibility with the soap base and its strange, sometimes even unknown components, cost of perfume raw materials, etc. One characteristic about his work is that he cannot judge the effect of his perfume when applied directly on the skin, not even in suitable dilution. He must try it out in soap—preferably in the customer's own soap base. Tables and entire books have been written about the stability of individual materials in soap bases, the amount of discoloration or breakdown of odor to be expected, etc., but the experienced soap perfumer will know that the rules of these books and tables are rules with an unusually high percentage of exceptions. In other words, if he went strictly according to all these more or less theoretical findings, he would eliminate so many good and useful raw materials that he would hardly have any left when he took cost and other factors into consideration.

But the soap perfumer's work is to a certain degree inhibited by the above factors. A successful soap perfume, successful in fragrance and appealing in cost to the manufacturer, is only obtained by many and lengthy experiments, often with numerous disappointments and discouragements. It is true to a certain degree that in perfumery "simplicity above all" is a safe rule to follow, but it is not always possible to arrive at the desired fragrance with 12 or 15 ingredients in the perfume formula. The fewer ingredients the perfumer uses, the more of each ingredient is needed. Certain materials are only available in limited amounts, and this is particularly true about natural perfume materials. This is one reason why soap perfumes often contain a higher proportion of synthetic materials than do the handkerchief perfumes or other cosmetic perfumes. Synthetic materials are, furthermore, easier to control chemically in the sense that we can predict the possible reaction with more accuracy than in the case of the very complex essential oils and other natural materials.

The "construction" of a soap perfume is different from that of other perfume types due to the special virtues demanded for soap perfumes. Power and diffusiveness in the fragrance itself should be balanced and chained properly by the skillful use of fixatives of which there are many types. Some fixatives work simply by the physical lowering of the vapor pressure in the perfume mixture, others have a much more intangible but no less perceptible effect.

Years ago it was fashion to make snow-white soap cakes. This placed very strict limits upon the choice of materials available to the soap perfumer. Today, many successful soaps appear in various lively colors under the protection of which the soap perfumer enjoys the use of a much larger variety of materials. The recent improvement in soap packaging, the aluminum foil wrapper, has been a great help to solving the problem of stability of the perfume in the soap cake, although the foil had other missions, too. The rancidity on long-time shelf storage was greatly reduced, and this fact was in turn a welcome improvement in the stability of the perfume.

The usage of germicides or "deodorants" in soap cakes has been an addition to the many problems of the soap perfumer. The bactericidal ingredients are often of considerable odor themselves, and it may be necessary first to "mask" the odor of the germicide before introducing the soap perfume specially designed for this purpose.

In brief, a soap perfume, like any other perfume, presents a variety of problems beyond that of creating an attractive fragrance. Long experience and professional skill, a thorough knowledge of perfume raw materials including the newest synthetic chemicals, good taste and understanding of the customer's actual preference are among the prerequisites for the perfumer who wants to produce a fragrance for soaps that will yield that priceless extra sales appeal.

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Heart Disease Research Symposium to Highlight U of I Lab Dedication

A symposium correlating four research areas in heart disease will be held June 16–18, 1963 at the University of Illinois in connection with dedication of The Burnsides Research Laboratory. These research areas involve: one, the clinical aspects of heart disease; two, the pathology and the composition of lipids in the aorta and the chemistry of the low density lipoproteins; three, the metabolism of lipids which may play a possible role in the development of atherosclerosis and four, factors or specific enzyme systems involved in lipid oxidation or metabolism. Complete accommodations for conference participants will be available in University dormitories. The Burnsides Research Laboratory, a new stone and glass structure consisting of two main stories, a basement and a penthouse with a total area of 21,232 square feet is occupied by the Food Chemistry Division of the Department of Food Technology. For further housing information and a copy of the program, write to: F. A. Kummerow, The Burnsides Research Laboratory, University of Illinois, Urbana, Ill.

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Spectroscopy Report . . .

(Continued from page 14)

Tentative Methods "Isolated *trans* isomers—infrared spectrophotometric method" CD 7-58, have been available from the Society through the Chairman of the Spectroscopy Committee (1 a, b, c). Approximately 50 sets of these standards have been furnished to various laboratories using this procedure. The provision in the revised procedure as published in the AOCS book of methods that the isolated *trans* content of long-chain fatty acids containing more than 15% *trans* isomers may be analyzed directly, required secondary standards for determination of *trans* isomer contents of fatty acids. These secondary standards were prepared by collaborative studies in eleven laboratories. Collaborative data from these studies is shown in Table II. Two standards for the determination of *trans* isomers in long-chain fatty acids were established, one with a high *trans* content (59.93%), and the second with a relatively lower *trans* acid content (29.99%). These values were obtained by averaging results reported by all collaborators excluding collaborator No. 9 (Table II). Results from collaborator No. 9 were excluded as their measurements were made by direct comparison with a primary oleic acid standard, rather than by the base-line technique of the official procedure. These secondary standards along with the standards for the determination of *trans* isomers and methyl esters and triglycerides will continue to be available through the Society. Questions regarding them should be addressed to the Chairman of the Instrumental Techniques Committee, American Oil Chemists' Society, P. O. Box 19687, New Orleans 19, La.

Collaborative Testing—Near Infrared Method for Hydroxyl Value

Following decisions made by the Spectroscopy Committee at the meeting in 1960 in Dallas (1c), a collaborative study was made of published methods for the determination of hydroxyl value of primary alcohols by means of near infrared spectroscopy. Results from eleven laboratories participating in this collaborative study are shown in Table III. The collaborators analyzed five Adol mixtures after calibration of their infrared spectrometers by the use of a series of highly purified primary alcohols. The results indicate reasonably satisfactory reproducibility and precision. However, in a discussion of these data at the 1962 meeting in New Orleans, considerable doubt was cast upon the value of the method because of its limited scope. From measurements in the near infrared in the overtone region at approximately 1.4 microns or from the fundamental stretching vibration at 2.8 microns analysis is limited to the determination of a primary OH group in samples containing no secondary hydroxyl groups. Considerable discussion was had at the New Orleans meeting regarding the implications of the limited scope and no specific recommendations for future activities of the Spectroscopy Committee were reached. One of the main decisions of the Spectroscopy Committee during the coming year will be to decide whether or not further collaborative studies should be made of the methods for hydroxyl value using the overtone region in the near infrared, approximately 1.4 microns, the fundamental stretching vibration region at about 2.8 microns, the bending OH vibrations near 9 microns, or to abandon use of infrared spectroscopy for hydroxyl value in favor of infrared spectrophotometric methods for other determinations which may not be so limited in scope.

Realignment of Technical Committees by the American Oil Chemists' Society's Governing Board

During the past year, a study of the organization of the American Oil Chemists' Society's various committees was initiated by the Governing Board. The study was conducted by an Organization Committee under the Chairmanship of former Society President R. W. Bates. At the 53rd Annual Meeting this Committee reported to the Governing Board; and as a result of their recommendations, the Governing Board has established a new Committee to be designated as the Instrumental Techniques Committee. This Committee will be comprised of three Subcommittees: a) Spectroscopy, b) Gas-liquid chromatography, and c) Color. Establishment of a new Instrumental Techniques Committee has now been completed as follows:

Instrumental Techniques Committee, R. T. O'Connor, Chairman.

(a) Spectroscopy Subcommittee, R. R. Allen, Chairman.

(b) Gas Chromatography Subcommittee, E, M. Sallee, Chairman.

TABLE II

Establishment of Secondary Standards for Determination of Isolated *Trans* Isomers by Infrared Absorption

Collaborator No.	Date and instrument	Secondary Standards Fatty acids High *trans* (#5) a	 % *Trans*	 Deviation from mean	 Fatty acids Low *trans* (#6) a	 % *Trans*	 Deviation from mean
1	1st day	0.276	61.2	0.35	0.141	31.3	0.65
	2nd day	0.281	62.2	1.35	0.139	30.8	0.15
	PE-21						
2	1st day	0.298	64.7	4.85	0.147	31.9	1.25
	2nd day	0.303	65.8	5.45	0.150	32.6	1.95
	IR-4						
3	1st day	0.246	56.3	2.55	0.128	29.7	0.95
	2nd day	0.250	57.3	3.55	0.128	29.8	0.85
	PE-221						
8	1st day	0.266	59.5	3.35	0.134	28.4	2.25
	2nd day	0.274	58.2	2.15	0.135	28.7	1.95
	IR-5A						
4	1st day	0.278	62.0	1.65	0.120	29.8	1.85
	2nd day	0.272	61.3	1.45	0.126	28.8	1.85
	PE-221						
5	1st day	0.284	60.2	1.15	0.142	29.6	1.05
	2nd day	0.284	59.5	0.85	0.146	30.6	0.05
	PE-21						
6	1st day	0.269	61.0	0.65	0.130	30.6	0.05
	2nd day	0.264	60.0	0.35	0.140	31.7	1.05
	PE-21						
7	1st day	0.298	59.8	0.55	0.156	31.6	0.95
	2nd day	0.302	60.4	0.05	0.156	31.2	0.55
	IR-4						
8	1st day	0.262	60.0	0.85	0.130	29.5	1.15
	2nd day	0.283	60.1	0.35	0.136	29.4	1.25
	PE-21						
*9	1st day	0.316	64.7	4.85	0.182	37.2	6.55
	2nd day	0.324	65.1	4.75	0.192	38.5	7.85
	PE-21						
10	1st day	0.296	58.6	1.75	0.142	28.1	2.55
	2nd day	0.296	58.2	2.15	0.146	28.8	1.85
11	1st day	......	57.8	2.55	......	29.4	1.25
	2nd day	......	56.9	3.45	......	28.0	2.65
Average			60.85			30.65	
Average not including #9			(59.93)			(29.99)	
Confidence interval:							
95%			±1.7			±0.8	
99%			±2.4			±1.2	

Agreement to be Expected When Comparing Estimates Made on the Same Material

	Ratio (larger/smaller) within which the estimates should fall the stated proportion of times			
Estimate	.90	.95	.99	.999
1. Two det'n made at same time in same lab	1.038	1.047	1.067	1.099
2. Two det'n made at different times in same lab	1.046	1.057	1.082	1.121
3. Two averages made at different times in same lab (each avg from 2 det'n made at same time in same lab)	1.037	1.046	1.066	1.097
4. Two averages made at different times in same lab (each avg from 2 det'n made at different times)	1.032	1.040	1.057	1.084
5. Two averages made at different times in same lab (each avg from 4 det'n, 2 det'n at each of 2 times)	1.026	1.032	1.047	1.068
6. Two det'n made in different labs	1.119	1.143	1.218	1.330
7. Two averages made in different labs (each avg from 2 det'n made at same time)	1.116	1.144	1.211	1.319
8. Two averages made in different labs (each avg from 2 det'n made at different times)	1.114	1.142	1.208	1.315
9. Two averages made in different labs (each avg from 4 det'n, 2 det'n at each of 2 times)	1.112	1.140	1.205	1.309

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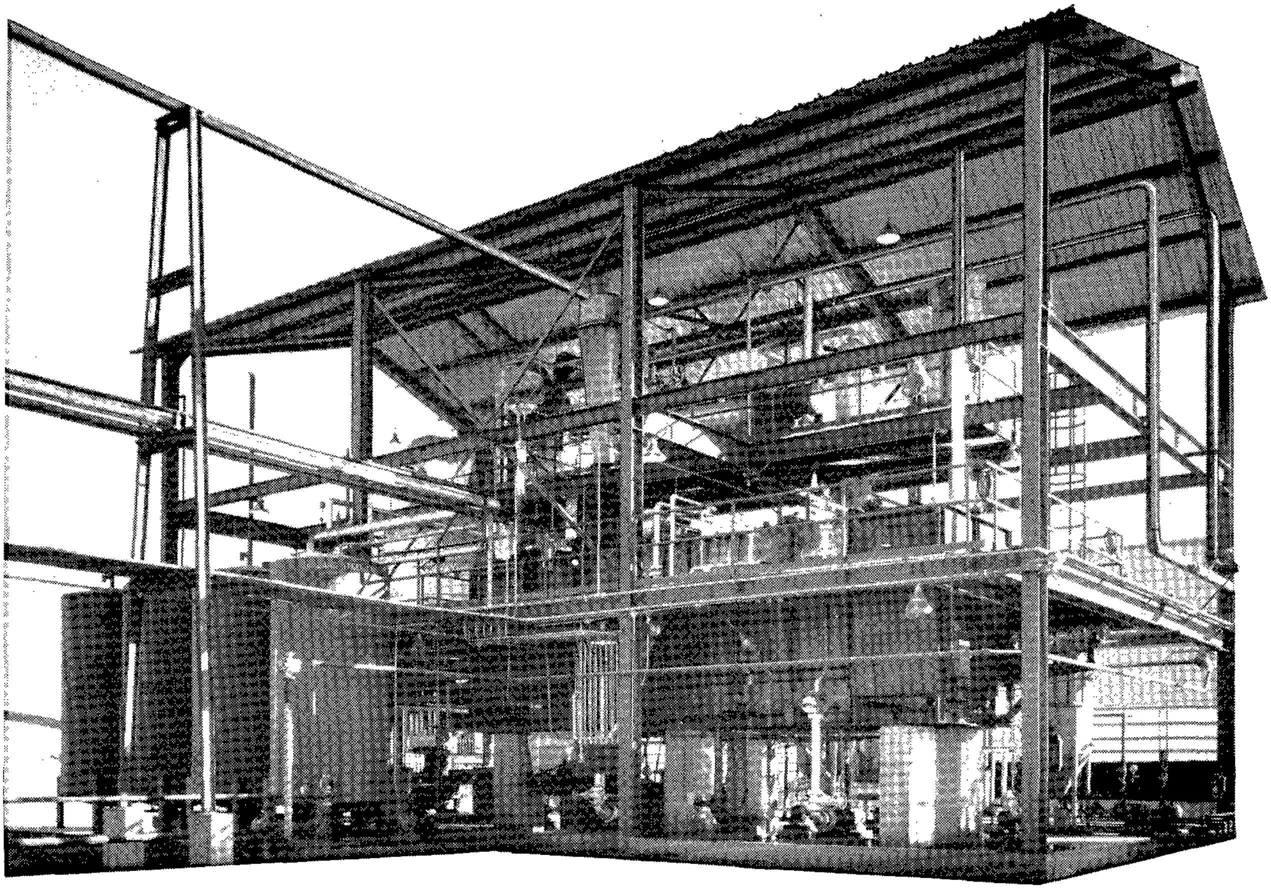

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TABLE III

Determination of Hydroxyl Value of Adols by Means of Near Infrared Spectroscopy

Collaborator Number	Date and instrument	Samples									
		Adol 14		Adol 32		Adol 52		Adol 60		Adol 62	
		% OH	Deviation from mean	% OH	Deviation from mean	% OH	Deviation from mean	% OH	Deviation from mean	% OH	Deviation from mean
1	1st day 2nd day Beckman DK-2	8.34 8.28	0.14 0.20	6.51 6.64	0.24 0.11	6.93 6.84	0.05 0.14	5.65 5.56	0.03 0.12	6.39 6.39	0.04 0.04
2	1st day 2nd day Cary 14	8.50 8.80	0.02 0.32	6.75 6.75	0.00 0.00	6.99 6.95	0.01 0.03	5.68 5.63	0.00 0.05	6.48 6.31	0.05 0.12
3	1st day 2nd day Beckman DK-2	8.74 8.70	0.26 0.22	7.14 7.13	0.39 0.38	7.16 7.06	0.18 0.08	6.20 6.01	0.52 0.33	6.59 6.72	0.16 0.29
4	1st day 2nd day ....[1]	8.48 8.52	0.00 0.04	6.76 6.78	0.01 0.03	6.98 6.98	0.00 0.00	5.70 5.60	0.02 0.08	6.42 6.47	0.01 0.04
5	1st day 2nd day Cary 14	8.33 8.22	0.15 0.26	6.56 6.50	0.19 0.25	6.84 6.88	0.14 0.10	5.56 5.49	0.12 0.19	6.27 6.28	0.16 0.15
6	1st day 2nd day Beckman DK-2	8.32 8.25	0.16 0.23	6.61 6.54	0.14 0.21	6.80 6.82	0.18 0.16	5.48 5.55	0.20 0.13	6.28 6.28	0.15 0.15
7	Lab #1 Lab #2 Cary 14	8.73 8.68	0.25 0.20	6.72 6.81	0.03 0.06	7.10 7.09	0.12 0.11	5.73 5.71	0.05 0.03	6.44 6.28	0.01 0.15
8	1st day 2nd day Beckman DK-2	8.39 8.41	0.09 0.07	6.69 6.77	0.06 0.02	6.92 7.13	0.06 0.15	5.68 5.77	0.00 0.09	6.49 6.48	0.06 0.05
9	Avg of samples Avg of samples Cary 14[2]	1&2 8.46 3&4 8.47	0.02 0.01	6.83 6.74	0.08 0.01	7.06 6.95	0.08 0.03	5.68 5.62	0.00 0.06	6.44 6.40	0.01 0.03
9	Avg of samples Avg of samples Beckman DK-2[2]	1&2 8.40 3&4 8.47	0.08 0.01	6.80 6.81	0.05 0.06	6.98 6.98	0.00 0.00	5.66 5.66	0.02 0.02	6.45 6.48	0.02 0.05
10	1st day 2nd day Beckman DK-2	8.00 8.00	0.48 0.48	6.51 6.36	0.24 0.39	6.84 6.76	0.14 0.22	5.43 5.25	0.25 0.43	6.18 6.22	0.25 0.21
11	1st day 2nd day Beckman DK-2	8.86 9.12	0.38 0.64	7.14 7.07	0.39 0.32	7.27 7.31	0.29 0.33	5.92 6.12	0.24 0.44	6.68 6.79	0.25 0.36
Average.................................... Standard deviation........................		8.48	0.20 0.257	6.75	0.15 0.206	6.98	0.11 0.140	5.68	0.14 0.207	6.43	0.12 0.152

[1] Collaborator did not identify instrument used.

[2] Collaborator did not indicate whether two sets of samples were analyzed on two different days.

(c) Color Subcommittee, W. T. Coleman, Chairman.
(d) Special Task Group for the Preparation of Methyl Esters, J. R. Chipault, Chairman.

A complete roster of all the members of the Instrumental Techniques Committee and of its Subcommittees appears in the 1962 Society Directory.

Acknowledgments

For whatever success it can obtain in its collaborative testing program, the Spectroscopy Committee is aware that it is indebted to several individuals for assistance in making spectral measurements, compiling data, and offering suggestions.

The Committee acknowledges receipt of the samples of fatty acids of low *trans* content used in establishing the secondary standards and the data reported in Table II of this report, and of the highly purified primary alcohols used for the calibration of the instruments in obtaining the data reported in Table III by near infrared spectroscopy from R. O. Crisler and the Procter and Gamble Company; receipt of the sample of fatty acids of high *trans* content reported in Table II from R. R. Allen and the Anderson Clayton and Co.; and of the Adols analyzed in a collaborative study reported in Table III from W. E. Link, Archer-Daniels-Midland Company. The Committee acknowledges also the considerable assistance of Elizabeth R. McCall in compiling, recomputing, and arranging the collaborative data reported in Tables II and III, of compiling results of various polls of the members of the Committee, and in the distributing of secondary standards for the AOCS tentative methods "Isolated *Trans* Isomers-Infrared Spectrophotometric Method"; and E. F. Schultz, Jr. for the statistical analysis of the data in Table II.

REFERENCES

1. AOCS Spectroscopy Committee Report, JAOCS
(a) 1958–59, JAOCS *36,* 627–631 (1959).
(b) 1959–60, *Ibid., 38,* 180–184 (1961).
(c) 1960–61, *Ibid, 39,* 32–34 (1962).

R. T. O'Connor, Chairman
J. R. Chipault, Subcommittee Chairman

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• *New Literature*

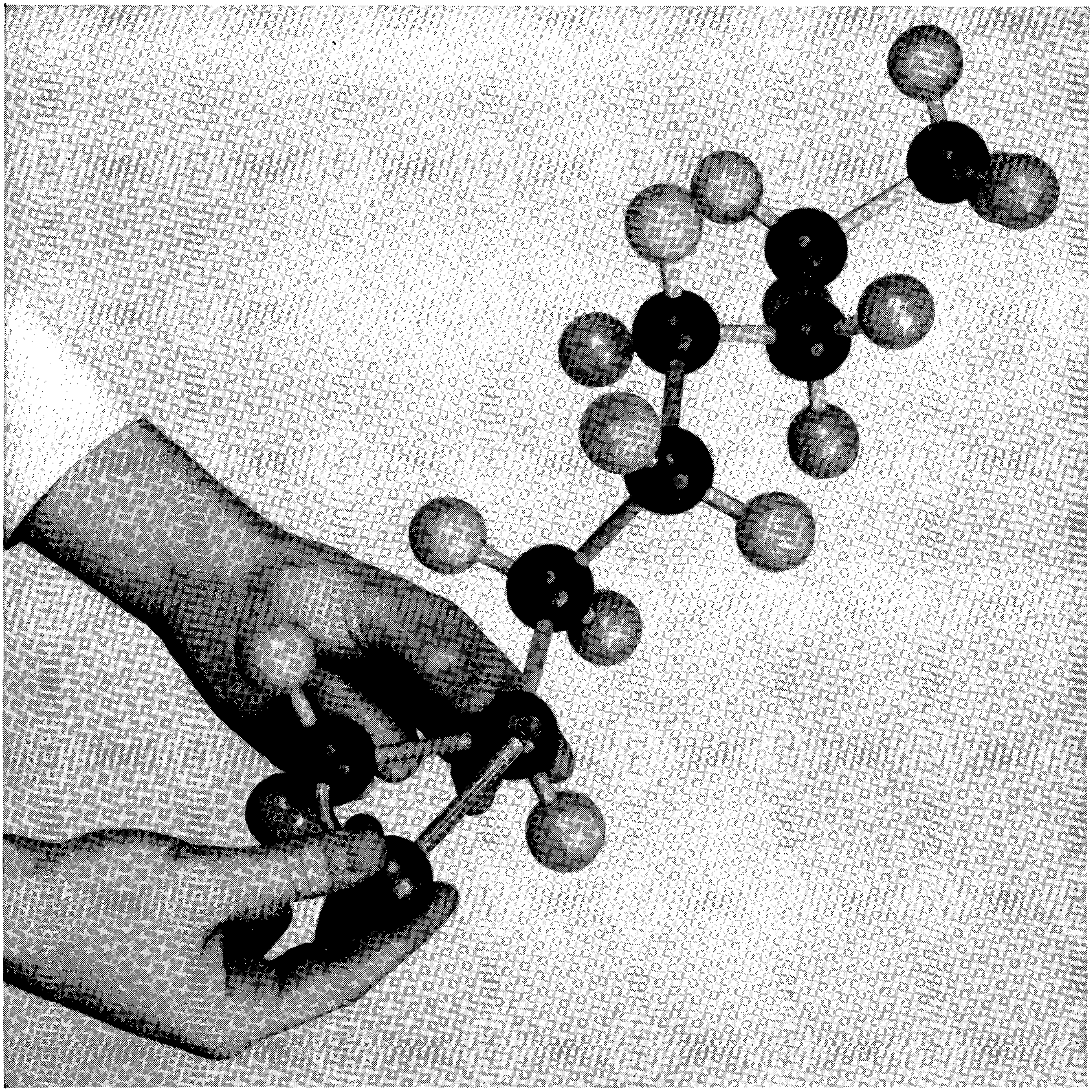
Schaar Scientific Company, recently released a 16-page brochure featuring several new product lines of special interest to laboratory personnel. (7300 W. Montrose Ave., Chicago, Illinois.)

U.S. Department of Commerce, National Bureau of Standards has announced the availability of new publication entitled "Controlled Temperature Oil Baths for Saturated Standard Cells," by Patrick H. Lowrie, Jr. (Superintendent of Documents, Technical Note 141, U.S. Government Printing Office, Washington, D. C.)

Worthington Corporation, now offers a new, 20-page bulletin describing Back Pull-Out (BPO) centrifugal pumps designed for the chemical process industries. The illustrated bulletin gives detailed information on the design, construction and application. (Advertising and Sales Promotion Dept., Harrison, N. J.)

E. H. Sargent and Company recently released its new bulletin ST-1 describing Spectrophotometric and Potentiometric Automatic Titrators. (4647 W. Foster Ave., Chicago, Illinois)

(Continued on page 39)



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(Continued from page 10)

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1954–55	10.3 Found	11.0 Lost	13.3 Lost	15.3 Found	1.4 Found
1955–56	.7 Found	12.2 Lost	.4 Lost	9.5 Found	2.5 Lost
1956–57	7.7 Lost	10.5 Lost	8.8 Lost	12.1 Found	14.4 Lost
1957–58	13.4 Found	22.4 Lost	10.4 Lost	14.5 Found	5.0 Lost
1958–59	10.0 Found	17.4 Lost	8.1 Lost	15.7 Found	.1 Lost
1959–60	13.6 Found	20.1 Lost	10.2 Lost	8.9 Found	7.9 Lost
1960–61	13.6 Found	26.2 Lost	10.6 Lost	15.8 Found	7.4 Lost
1961–62 original	.8 Found	16.4 Lost	26.9 Lost	28.1 Found	16.0 Lost
1961–62 revised	9.5 Found	13.2 Lost	26.9 Lost	28.1 Found	2.6 Lost
1962–63	11.1 Lost				

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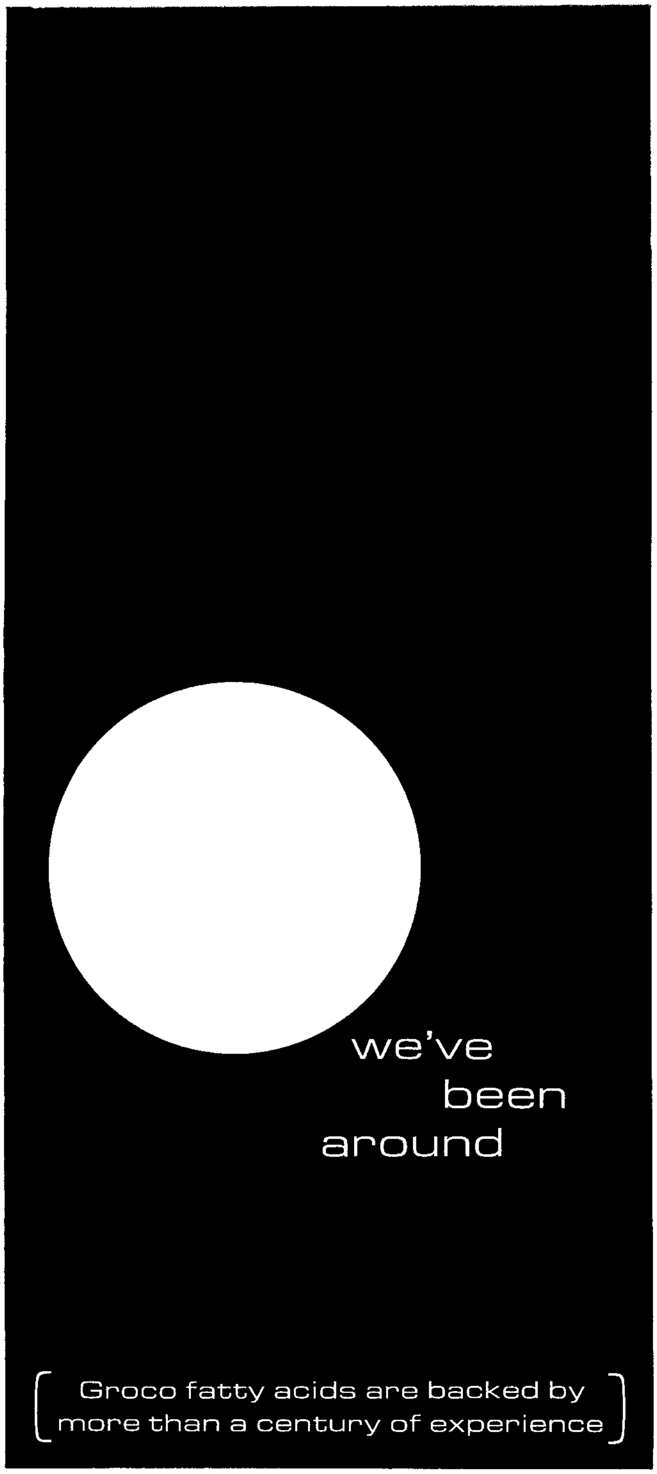

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