



## Meetings

### AOCS National Meetings

Apr. 28–May 1, 1974–Mexico City, Mexico, Maria Isabel Sheraton and Camino Real Hotels.

Sept. 29–Oct. 2, 1974–Philadelphia, Pa., Sheraton Hotel.

Apr. 27–30, 1975–Dallas, Tex., Statler Hilton.

Sept. 28–Oct. 1, 1975–Cincinnati, Ohio, Netherland Hilton.

### Other Organizations

Nov. 5–7, 1973–Course on Noise Control in Manufacturing Plants, Miami. Contact: Gloria Cianci, Bolt Beranek and Newman Inc., 50 Moulton, Cambridge, Mass. 02138.

Nov. 6–8, 1973–Three Day Course on Groundwater Pollution, Commodore Hotel, New York, N.Y. Contact: Continuing Education in Engineering, University Extension, University of California, 2223 Fulton St., Berkeley, Calif. 94720.

Nov. 11–15, 1973–American Institute of Chemical Engineers 66th Annual Meeting, Bellevue Stratford Hotel, Philadelphia, Pa. Contact: F.J. Van Antwerpen, executive secretary, AIChE, 345 East 47th St., New York.

Nov. 12–14, 1973–World Soy Protein Conference, Munich, Germany. Contact: Jim Yancey, American Soybean Association, P.O. Box 158, Hudson, Iowa 50643.

Nov. 14–16, 1973–Joint Eastern Analytical Symposium and National Meeting of Society for Applied Spectroscopy, Statler-Hilton Hotel, New York. Contact: Michael W. Miller, NL Industries, P.O. Box 420, Hightstown, N.J. 08520.

Nov. 14–17, 1973–51st Annual Meeting and 38th Paint Industries' Show, Conrad Hilton Hotel, Chicago, Ill. Contact: Dennis A. Bergren, De Soto, Inc., 1700 South Mt. Prospect Rd., Des Plaines, Ill. 60018.

Nov. 19–21, 1973–Symposium on Water Quality Parameters—Selection, Measurement and Monitoring, Analytical Chemistry Div., Chemical Institute of Canada, Burlington, Ont., Can. Contact: S. Barabas, Analytical Methods Research Div., Canada Centre for Inland Waters, Burlington, Ont., Can.

Dec. 10–12, 1973–Second Joint Conference on Sensing of Environmental Pollutants, Sheraton-Park Hotel, Washington, D.C. Contact: Philip N. Meade, Instrument Society of America, 400 Stanwix St., Pittsburgh, Pa. 15222.

Jan. 7–11, 1974–Direct Energy Conversion Short Course, College of Engineering Sciences, Arizona State University, Tempe, Ariz. Contact: Dr. Charles E. Backus, College of Engineering Sciences, Arizona State University, Tempe, Ariz. 85281.

Jan. 7–17, 1974–Ice Cream Short Course, The Pennsylvania State University, University Park, Pa. Contact: Director of Short Courses, 306 Agricultural Administration Building, The Pennsylvania State University, University Park, Pa. 16802.

Jan. 9–10, 1974–Course on Fundamental and Applied Aspects of Paint Removal for Refinishing, Sinclair Auditorium, Lehigh University, Bethlehem, Pa. Contact: Dr. Gary Poehlein, Associate Professor, Department of Chemical Engineering, Lehigh University, Bethlehem, Pa. 18015.

Jan. 15–19, 1974–47th Annual Industry Convention. The Soap and Detergent Association, Boca Raton Hotel and Club, Boca Raton, Fla. Contact: A.H. Howland, chairman, Convention Committee, The Soap and Detergent Association, 475 Park Ave. S., New York, New York 10016.

Jan. 16–18, 1974–29th Instrumentation Symposium for the Process Industries, J. Earl Rudder Conference Center, Texas A&M University, College Station, Texas. Contact: Dr. R.G. Anthony, Symposium Director, Chemical Engineering Department, Texas A&M University, College Station, Texas 77843.

Feb. 9–10, 1974–29th Annual Convention of the Oil Technologists' Assn. of India and Symposium on Problems and Prospects in Oils and Fats, Surface Coatings and Bleaching Earths and Active Carbons, Regional Research Lab., Hyderabad, India. Contact: Dr. A.J. Pantulu, Convener, OTAI Symposium, Regional Research Lab., Hyderabad-500009, India.

Mar. 1–2, 1974–22nd Annual Food Technology Conference of the St. Louis and Kansas City, Mo., sections of the Institute of Food Technologists, University of Missouri, Columbia.

Mar. 4–8, 1974–25th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy, Cleveland Convention Center, Cleveland, Ohio. Contact: Richard S. Danchik, program chairman, 1974 Pittsburgh Conference, Alcoa Labs., Alcoa Center, Pa. 15069.

Apr. 1–5, 1974–International Conference on Animal Feeds of Tropical and Subtropical Origin, London. Contact: Public Relations C, Tropical Products Institute, 56–62 Gray's Inn Rd., London WC1X 8LU, U.K.

April 23–26, 1974–26th Annual Technical Exhibition of the Oil and Colour Chemists' Assn., Empire Hall, Olympia, London, U.K. Contact: Oil and Colour Chemists' Assn, Priory House, 967 Harrow Rd., Wembley, Middlesex, U.K. HA0 2SF.

May 1–2, 1974–International Symposium on Flammability and Fire Retardants, Parkway Hotel, Cornwall, Ont., Can. Contact: Vijay Mohan Bhatnagar, editor, *Advances in Fire Retardants*, 209 Dover Rd., Cornwall, Ont., Can.

May 5–8, 1974–Sixth Annual Offshore Conference, Astrohall, Houston, Tex. Contact: Stan R. Houston, 6200 N. Central Expressway, Dallas, Tex. 75206.

May 12–16, 1974–34th Annual Meeting of the Institute of Food Technologists, New Orleans La. Contact: Edward H. Hoffman, Institute of Food Technologists, Suite 2120, 221 N. La Salle St., Chicago, Ill. 60601.

May 29–31, 1974–Canadian Sulphur Symposium, University of Calgary, Calgary, Alberta, Can. Contact: Dr. T.W. Swaddle, Dept. of Chemistry, University of Calgary, Calgary, Alberta, T2N 1N4.

(Continued on page 498A)

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Monique Quick, Ladies' Entertainment and Hospitality Chairwoman, center, welcomed wives of participants to the Ladies' Hospitality Room. Activities included a private slide lecture at the Art Institute, followed by a viewing of the Winslow Homer exhibit.



General Chairman Wally Quick greeted members and guests during the plenary session Monday morning.

## The welcome

Illinois' lieutenant governor among those who bid greetings to participants in the AOCS Fall Meeting



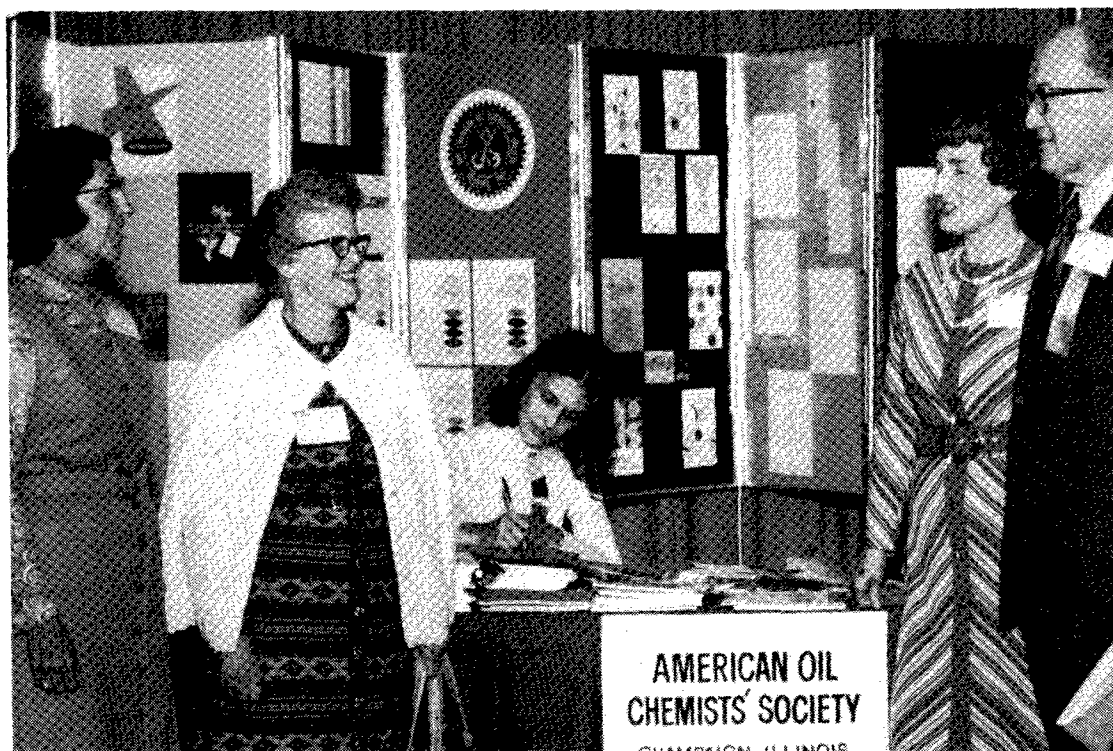
AOCS President F.A. Norris gives the call to order to begin the plenary session at the 47th Annual Fall Meeting held Sept. 16-19 at the Pick-Congress Hotel in Chicago.



Illinois Lt. Gov. Neil Hartigan welcomed plenary session participants to Chicago.



AOCs Fall Meeting participants registered during the four days of the event.



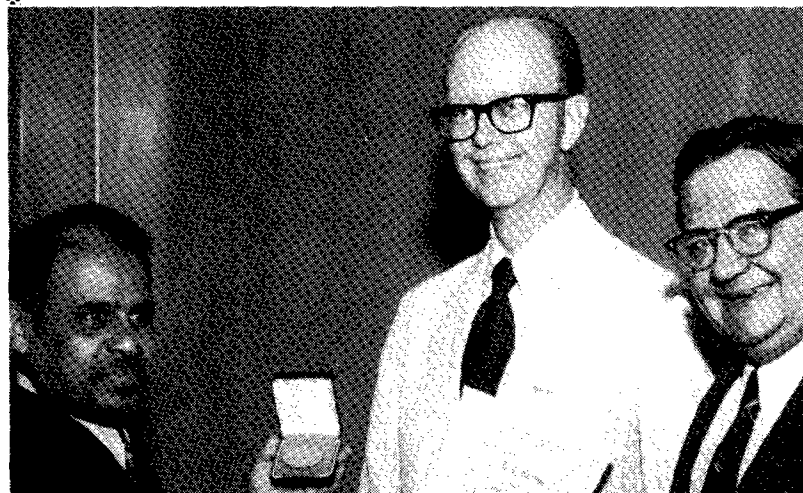
AOCs Staff Member Pam Wilson (center, seated) manned the membership booth during the Meeting. From left are Mrs. R.T. O'Connor, Mrs. R.T. Holman and Mr. and Mrs. F.A. Norris.



R.T. Holman, chairman, Honored Student Award Committee, presented certificates to the three recipients during the plenary session in Chicago. From left are: Angela K. Young, graduate student and 1974 Ph.D. candidate at the University of Guelph; Michael J. Blumenthal, Rutgers University graduate student; and Richard A. Whorton, graduate student and Ph.D. candidate at Vanderbilt University.

## The awards

AOCS continues its program of gifts and grants to deserving members of the fats and oils industry

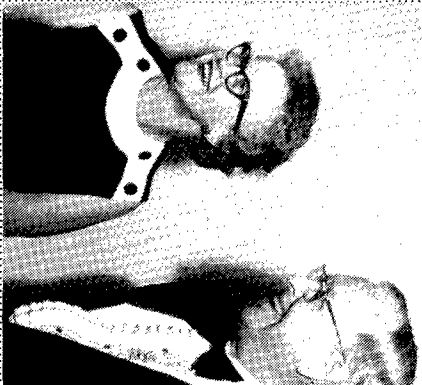


Bond Award Chairman R.G. Krishnamurthy, left, presented Bond Awards to K.D. Carlson and B.B. Perkins during the plenary session at the AOCS 47th Annual Fall Meeting in Chicago.



Arthur Rose, right, presented the 1973 Award in Lipid Chemistry to Frank D. Gunstone for "eminent contributions in the field of original research in lipid chemistry." Gunstone, professor in the Department of Chemistry, University of St. Andrews, St. Andrews, Scotland, has made vital contributions to the knowledge of the chemistry of naturally occurring and synthetic oxygenated fatty acids. The award is sponsored by Applied Science Laboratories and consists of a plaque and a \$2500 honorarium.

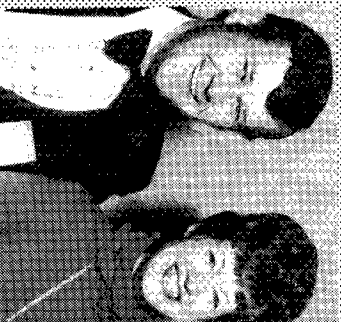
**The banquet**  
 AOCS couples enjoy  
 excellent entertainment  
 provided by  
 the Keeler Trio,  
 Franz Benteler and  
 the Arbors



Mr. and Mrs. R.T. Hoffman



Mr. and Mrs. J.C. Endres



Mr. and Mrs. James Lyon



Mr. and Mrs. F.A. Norris



Mr. and Mrs. William E. Link



Mr. and Mrs. Louis Goodman



Mr. and Mrs. Kenneth Hildebrand



Mr. and Mrs. Arthur Rose



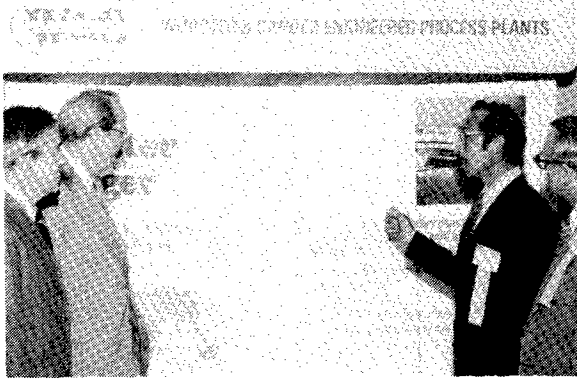
Mr. and Mrs. Wally Quirk



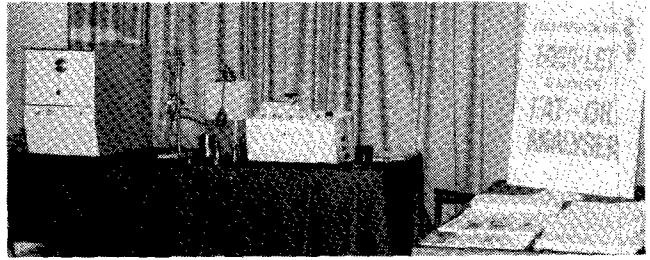
Mr. and Mrs. Bart Greenwell



Mr. and Mrs. F.D. Gunstone

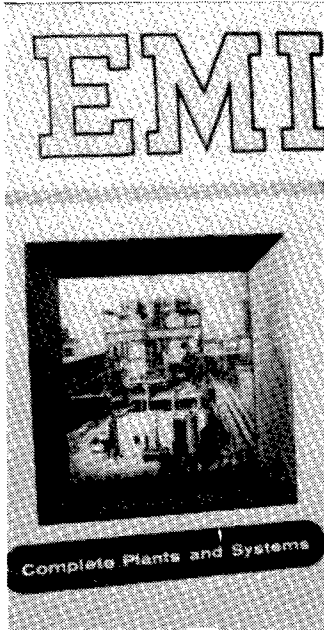


Wurster & Sanger

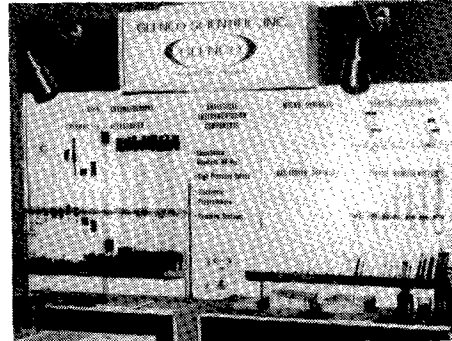


Foss America, Inc.

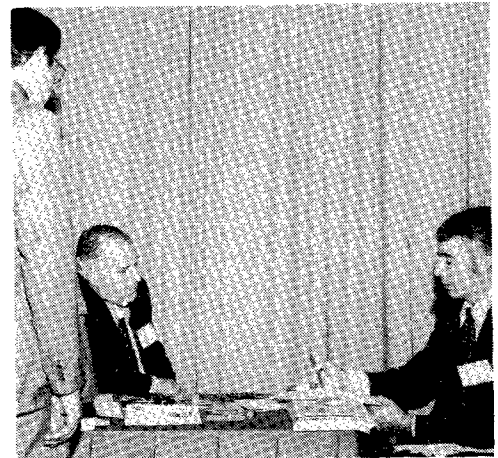
## The exhibitors 30 booths add dimension to Fall Meeting



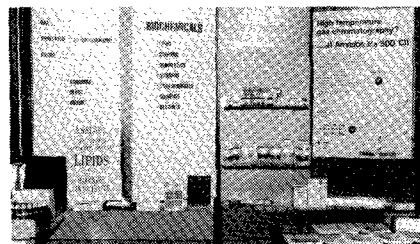
EMI



Glenco Scientific, Inc.



APV Co., Inc.



Analabs



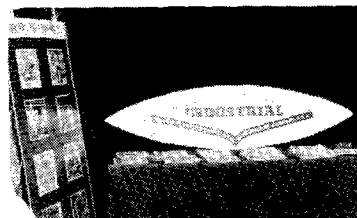
Hoffmann-La Roche, Inc.



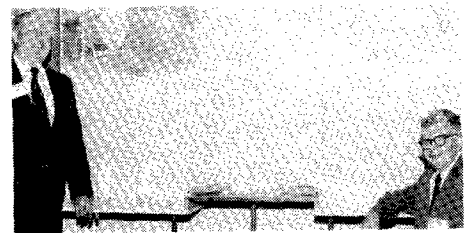
De Laval Separator



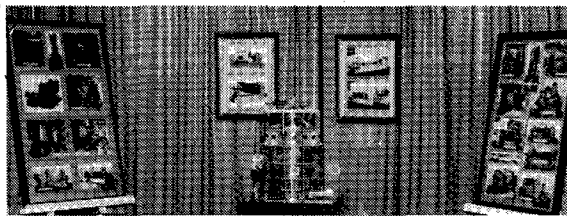
Artisan Industries, Inc.



Industrial Filter & Pump Mfg. Co.



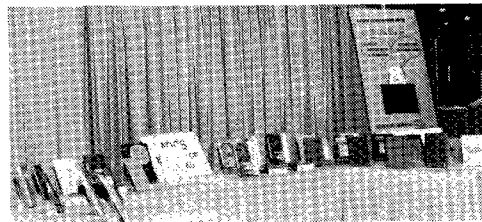
NAFCO Filtration Co.



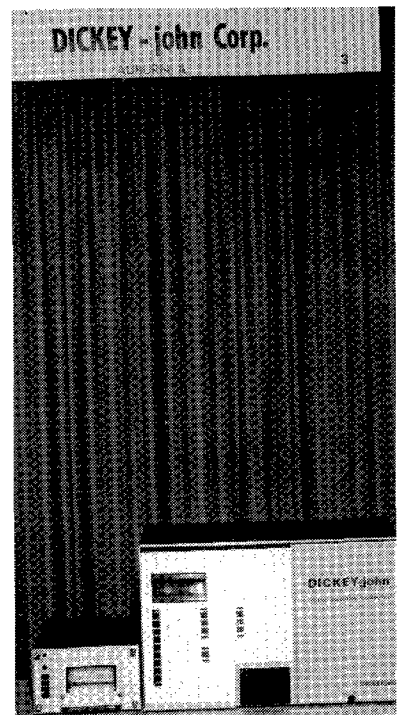
French Oil Mill Machy. Co.



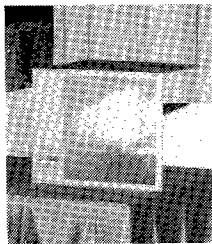
Lakeland Engineering



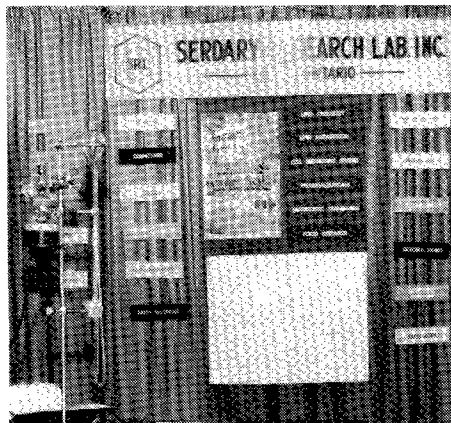
AOCSS Books Exhibit



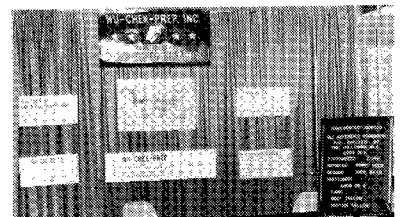
DICKEY-john Corp.



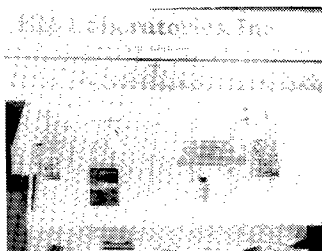
Ferrell-Ross, Inc.



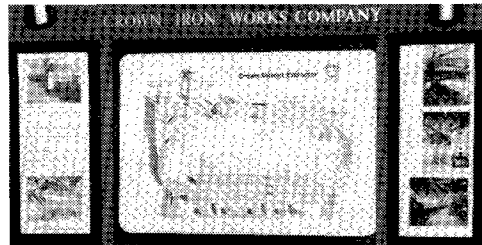
Serdary Research Lab, Inc.



Nu-Chek-Prep, Inc.



EM Laboratories



Crown Iron Works Co.



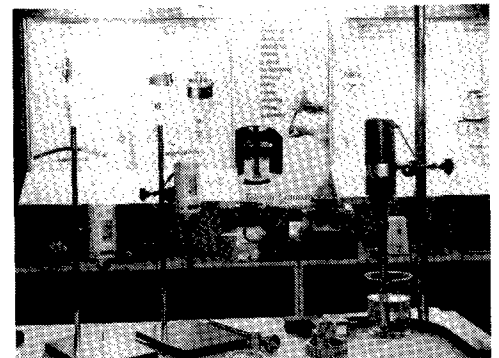
Per Corp.



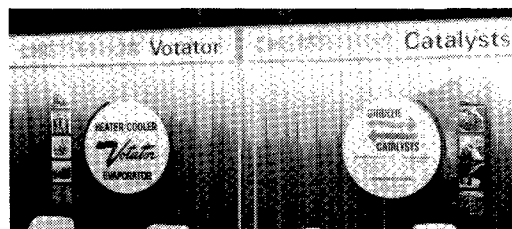
L.A. Salomon & Bro.



Dravo

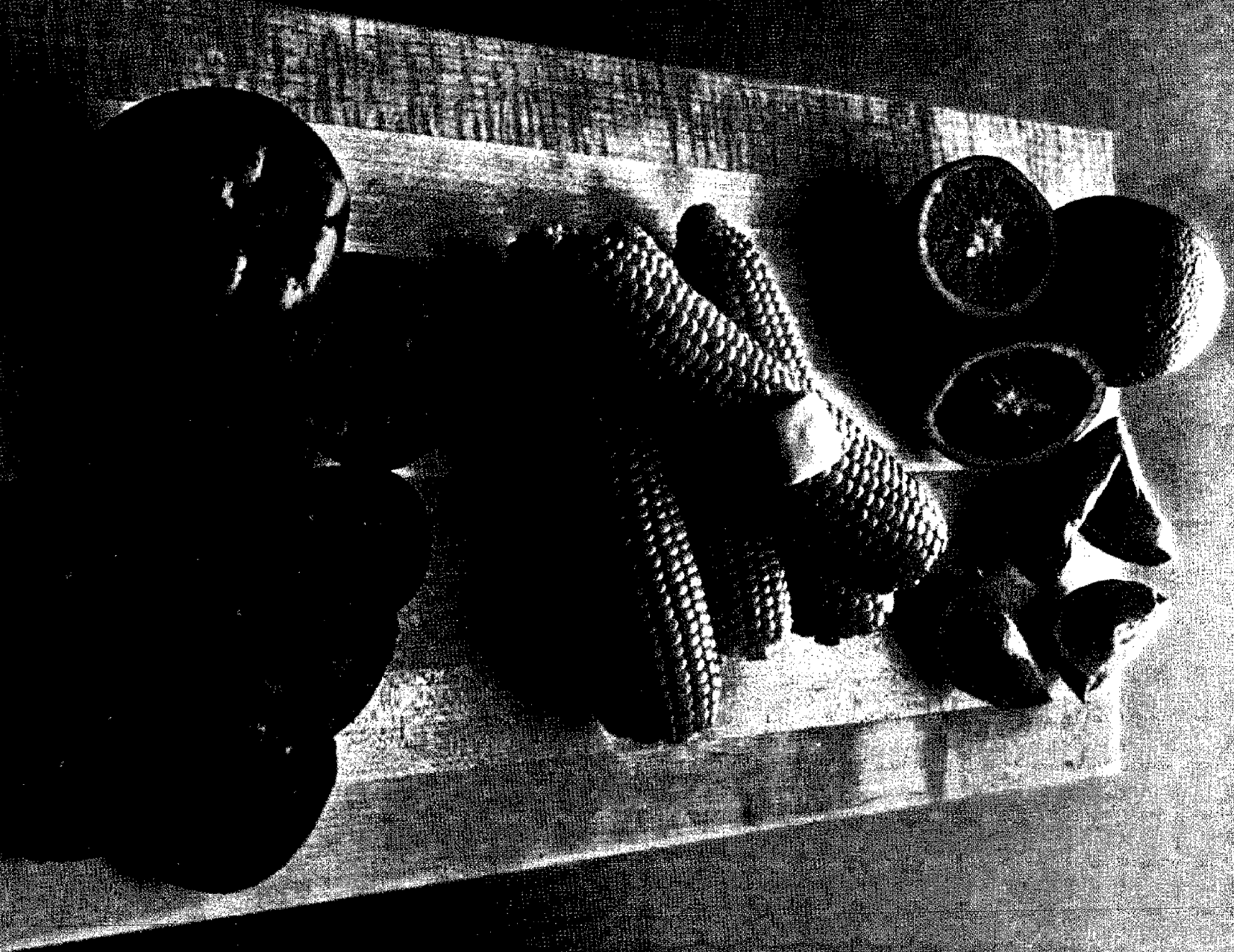


Tekmar Co.



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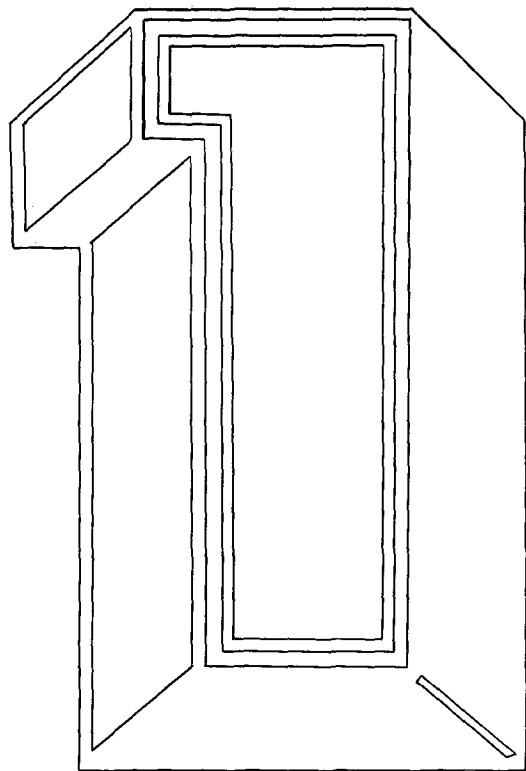
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# President's Club and Honor Roll

The members listed here have qualified for either the AOCs President's Club or President's Honor Roll. All current members who successfully recruit at least one new member qualify for Club membership. Successful recruitment of at least three new members is the qualification for the more prestigious Honor Roll. All Club and Honor Roll members will receive further recognition and the opportunity to participate in other special programs and activities. Special forms for use in recruiting new members are available from AOCs headquarters.

## Call for Nominations 1974 Honored Student Awards

Nominations are solicited and will be accepted until December 1, 1973, for the 1974 AOCs Honored Student Awards. Graduate students at any North American institution of higher learning, in any area of science dealing with fats and lipids, who are doing research toward an advanced degree and who are interested in the areas of science and technology fostered by this Society, are eligible. The student must be a registered graduate student at the time of application. To receive the award he must remain a registered graduate student, and must not have received his degree or begun career employment, prior to the AOCs meeting he is to attend. Selection of awardees is on the basis of educational qualifications and performance.

The awards provide funds equal to travel costs plus \$75.00 to permit attendance at a national meeting of the AOCs. In 1974 these meetings will be held April 28-May 1 in Mexico City and September 29-October 2 in Philadelphia. Students will be awarded travel to the nearer meeting to allow as many awards as possible from the available funds.

Nomination forms may be obtained from AOCs headquarters (508 S. Sixth, Champaign, Ill. 61820) or from the chairman of the Honored Student Award Committee. Completed nominations should be returned before December 1, 1973, to: Ralph T. Holman, HSA Committee Chairman, The Hormel Institute, University of Minnesota, Austin, Minn. 55912. ■

### Twelve

Edward G. Perkins

### Eight

Joseph G. Endres

### Six

Robert T. O'Connor

### Five

M.K. Govind Rao

### Four

Kazuo Fukuzumi  
Robert C. Hastert  
Edward M. Lloyd  
Samuel E. Pack  
Joseph Pominski  
Emmett D. Russey  
Joseph Lee Williams

### Three

Gregory E. Anekwe  
Stephen S. Chang  
Martin Freeman  
Earl G. Hammond  
Francis G. Schmid  
Brian L. Walker

### Two

Robert R. Allen  
Clyde H. Amundson  
John D. Brandner  
Lois S. Crauer  
Paul D. Cretien  
David Firestone  
Wilfred J. Frech  
H.P. Gormley  
Charles W. Hoerr  
Robert Johnson  
Karl W. Klein  
Fred A. Kummerow  
Arieh Letan  
Andrew M. Lubienski  
Gerhard Maerker  
L. Dave McClung  
John E. Middendorf  
Toshio Nakagawa  
Robert L. Ory  
Herbert L. Rice  
Lars H. Wiedermann  
Lester M. Wright

### One

Abdel-Hamid Y. Abdel-Rahman

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Peter A. Cammelot  
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Juan Chavez-Pereza  
William T. Coleman  
John G. Coniglio  
Sam D. Cunningham  
Stephen M. Davis  
John A. Decker  
Jacqueline Dupont  
Harold P. Dupuy  
Herbert J. Dutton  
Robert L. Edwards  
Tom Edwards  
Richard R. Egan  
Manuchehr Eijadi  
Reuben O. Feuge  
Homer Gardner, Jr.  
Paul Gibson  
Lawrence Gildenberg  
Cecilia Gilmore  
Charles W. Glankler  
Louis H. Going  
Dale P.J. Goldsmith  
Louis P. Goodman  
Dennis T. Gordon  
Richard L. Gregory  
Edward R. Hahn  
Ralph A. Hagberg  
Tetsutaro Hashimoto  
Albert S. Henick  
Samuel F. Herb  
Richard E. Helland  
John D. Hetchler  
Robert J. Hlavacek  
Carroll L. Hoffpauir  
Richard T. Holzman  
Joseph R. Houle  
Robert L. Husch  
Raymond H. Jones  
George M. Kreutzer  
V. Krishnamoorthi  
Paul A. Krumm  
James Laubscher  
Doris A. Lillard  
Warner M. Linfield  
William Link  
Francis E. Luddy  
Matthew Lynch  
Ahmed F. Mabrouk  
Laurence G. Mackin  
Richard Eugene Madden  
William V. Magee  
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Tommy R. Massey  
Ted P. Matson  
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Nicholas Pelick

Arthur F. Pelster  
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 Richard Purdy  
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 Robert A. Reiners  
 Raymond Reiser  
 Albert M. Reynaud  
 Louis L. Richardson  
 James A. Robertson  
 Ronald Robinson  
 George H.G. Rowland  
 Joseph Sampugna  
 Birger H. Sandal  
 Werner H. Schmidt  
 Arthur A. Schmitz  
 Irving R. Schmolka  
 Suresh M. Shah  
 B.S.K. Shastri  
 E. Griffin Shay  
 Frank G. Shea  
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 Samuel C. Smith  
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 Robert M. Starr  
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 Victor V. Studer  
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 William R. Wiggins  
 Earl R. Williamson  
 Randall Wood  
 William C. Wood  
 Clyde Thomas Young  
 Calvin T. Zehnder  
 Richard J. Zielinski

The individuals listed below have applied for membership in AOCS between September 8 and October 15, 1973.

Raymond George Gallant, sr. res. chem., Central Soya, 1825 N. Laramie Ave., Chicago, Ill. 60639.

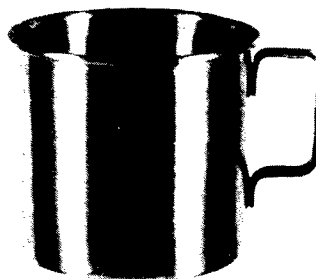
James Philip Hussey, chem., Woodson-Tenent Lab., 345 Adams Ave., Memphis, Tenn. 38101.

Juan Blay Ilagan, proj. eng.-eng. dev., SCM-Glidden Durkee, 16651 Sprague Rd., Strongsville, Ohio 44136.

Vasudeva Narayanan Paniker, asst. prof. of med. and chief of hem., Vanderbilt University, Research Lab., 21st and Garland, Nashville, Tenn. 37232.

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**THE LIQUID CHROMATOGRAPHY PEOPLE**

# Some Physical, Chemical, and Biological Properties of Natural and Synthetic Unsaturated C<sub>18</sub> Acids<sup>1</sup>

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## ABSTRACT

Series of octadecenoic (*cis* and *trans*), octadecynoic, octadecadienoic (*cis,cis* and *trans,trans*), and octadecadiynoic acids have been synthesized and employed in a comparative study of some physical (melting point, Raman spectra, NMR spectra, Ag<sup>+</sup> thin layer chromatography, and gas liquid chromatography) and biological properties. The results show that the position of unsaturation has a considerable influence on the physical and biological properties of isomeric unsaturated acids. Several chemical reactions of linoleic acid and of the related oxygenated acids ricinoleic or vernolic (12,13-epoxyoleic) furnish cyclopropane compounds and 1,4- (or 1,5-) epoxides. The formation of these is rationalized in terms of neighboring group participation. The oxymercuration-demercuration reaction is the basis of a new method of examining acids with  $\Delta 3$ ,  $\Delta 4$ , or  $\Delta 5$  unsaturation.

## INTRODUCTION

Although the study of natural products has always been a prominent part of organic chemistry, fatty acids have not been highly considered in comparison with the more favored carbohydrates, isoprenoids, and alkaloids, to mention only a few. Historically, organic chemists preferred those compounds which could be readily isolated in a pure state, and this generally implied that they were crystalline solids or volatile liquids. If they were colored, that was an added advantage. The fatty acids (and, to a lesser extent, the amino acids) did not meet these criteria; and, in addition, most fatty compounds were considered to be mixtures of a small range of acids of simple structure.

This is no longer true. New chromatographic and spectroscopic procedures make these mixtures easy to handle and study. Over 300 natural fatty acids are now

known, and biochemists continually ask questions that present interesting challenges to the organic chemists working in this field. It is 30 years ago this autumn since I became a research student in Liverpool, working with T.P. Hilditch; fatty acid chemistry is a very different activity from what it was at that time.

Nevertheless, and despite the many meetings organized by the AOCS and all that has been written in the 50 volumes of the Society's journal, there is still a lot that we do not know about the chemistry of fatty acids. There has been a concentration of study on a few acids, such as oleic acid and linoleic acid, and it has been generally assumed that what was true of oleic acid held for other monoenoic acids and that what was true of linoleic acid held for other polyenoic acids. This may be so when we consider the gross chemical behavior of these compounds. It is less likely to hold for those physical and biological properties which are more specifically dependent on the shape of the whole molecule and on the conformation it assumes at the moment of investigation.

During the past decade we have been engaged in studies designed to learn more about fatty acid chemistry. Some of my students have prepared series of closely related acids and have then made—sometimes in collaboration with other research groups—a comparative study of their properties. Other students have examined some old and some new reactions of the more common acids (such as oleic and linoleic acid) and of acids with an additional oxygenated function (such as ricinoleic and vernolic acid). In these studies the underlying theme of neighboring group participation has become apparent.

In this lecture I hope to review some of our recent investigations. Inevitably our work has impinged on the studies of other research groups; and I will refer to some of

<sup>1</sup>F.D. Gunstone received the 1973 Award in Lipid Chemistry at the AOCS 47th Annual Fall Meeting, Chicago, September 1973. This Award Address was presented at the Plenary Session.

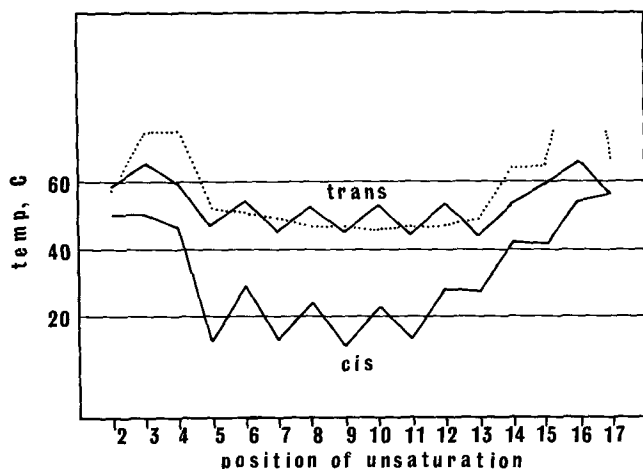


FIG. 1. Melting points of octadecenoic (*cis* and *trans*) (—) and octadecynoic acids (· · · ·).

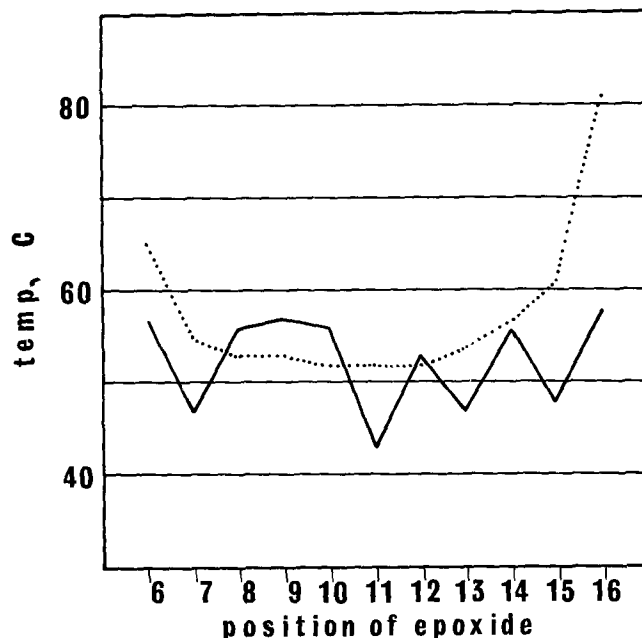


FIG. 2. Melting points of *cis* (—) and *trans* (· · · ·) epoxyoctadecanoic acids. Each isomer is indicated by a single number, e.g. 2,3-epoxyoctadecanoic acid by 2, etc.

these, but it will not be possible to do justice to these related studies. Therefore, I ask forgiveness for this egotistical outlook.

## SYNTHETIC STUDIES

### Synthesis

In the course of our work we have prepared three groups of unsaturated  $C_{18}$  acids (1-4): (a) All the *cis* and *trans* octadecenoic acids and all the octadecynoic acids (47 isomers); (b) all the 9,12-diunsaturated  $C_{18}$  acids (9 isomers); (c) a series of  $C_{18}$  acids (the *cis cis* and *trans trans* dienes and the diynes) with unsaturation at 5,12; 6,12; 7,12; 8,12; 9,12; 10,12; 6,11; 6,10; 6,9; and 6,8 (30 isomers). The dienes contain the structural unit  $-\text{CH}=\text{CH}(\text{CH}_2)_n\text{CH}=\text{CH}-$  where  $n$  ranges from 0 to 5.

In addition, we have converted most of the alkenoic and alkadienoic acids to the corresponding epoxides (5) and to the cyclopropane derivatives (6-8).

These syntheses have been achieved mainly by taking advantage of the special properties of acetylene: its ability to undergo alkylation and stereospecific semihydrogenation to *cis* or *trans* alkenes. We have also prepared some of these acids by manipulation of more-or-less readily available natural products. For example, we used methyl vernolate (*cis*-12,13-epoxyoleate) to produce the 9*c*12*t*, 9*a*12*t*, and 9*a*12*c* diunsaturated  $C_{18}$  acids by suitable combination of three reactions: (a) the stereospecific conversion of *cis* epoxides to *threo* or *erythro* diols, (b) the conversion of an alkene to an alkyne, and (c) the conversion of *threo* and *erythro* diols to *trans* and *cis* alkenes respectively (4).

### Melting Point

The melting points of the monounsaturated  $C_{18}$  acids are summarized in Figure 1 (3). Among the alkenoic acids the *cis* isomers have the lower melting points, with oleic acid the lowest of all. Both *cis* and *trans* isomers show alternation of melting point when the double bond is in the central region, and this effect is superimposed on a tendency for the melting point to rise as the double bond approaches either end of the molecule. In contrast, the acetylenic acids do not show this alternation though it has been reported to occur among alkynoic acids with an *odd* number of carbon atoms in their molecules (9).

We have also observed some interesting results with the melting points of the *cis* and *trans* epoxystearic acids (5) (Fig. 2). Alternation is apparent among the *cis* members but not among the *trans*, so that sometimes the *cis* epoxide and

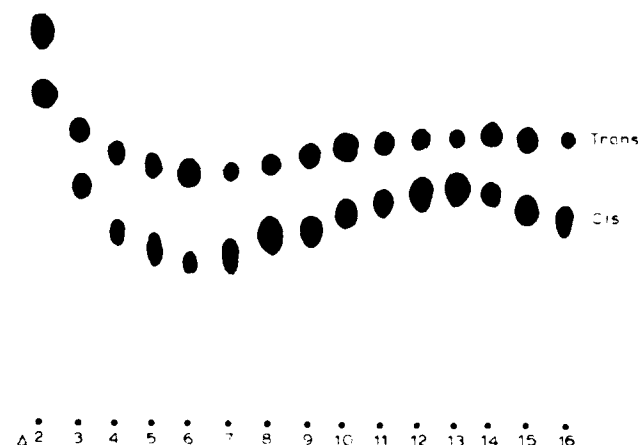


FIG. 3. Thin layer chromatogram of methyl *cis* and *trans* octadecenoates ( $\Delta 2$ - $\Delta 16$ ). With the  $\Delta 2$  esters the *cis* isomer has the higher  $R_f$  value; in all other cases the *trans* isomer has the higher  $R_f$  value. Experimental conditions: silica layers (250  $\mu$ ) containing silver nitrate (15%), activated by heating at 105 C for 75 min, developed with dibutyl ether and hexane (40:60), made visible by heating with a glassblower's torch.

sometimes its *trans* isomer has the higher melting point. The regular alternation of melting point among the *cis* epoxy acids is broken by one value which appears to be too high; but, since this refers to *cis*-9,10-epoxystearic acid and has been reported from several laboratories, there can be no doubt about the correctness of this observation.

### Spectroscopy

IR spectroscopy has been used for many years to detect *trans* unsaturation, but there is no satisfactory IR procedure for the recognition of *cis* unsaturation or acetylenic unsaturation. Using our synthetic esters, Davies and Hodge (10) showed that these compounds display C=C or C $\equiv$ C stretching bands in their Raman spectra, which can be used easily for diagnostic purposes. Using carbon tetrachloride solutions, these bands are observed at  $1656 \pm 1$  (vs) and  $1670 \pm 1$  (vs) for the *cis* and *trans* alkenoates respectively and at  $2232 \pm 1$  (s) and  $2291 \pm 2$   $\text{cm}^{-1}$  (m) for the alkynoates.

Our earlier studies of the NMR spectra of our synthetic esters on 60 and 100 MHz spectrometers (4-8,11,12) have been repeated on a 220 MHz instrument in collaboration with Frost (Unilever, Vlaardingen). Methyl oleate shows distinct signals at 9.12 ( $\text{CH}_3\text{CH}_2-$ ), 8.75 ( $\text{CH}_2$ ) $_n$ , 8.43 ( $-\text{CH}_2\text{CH}_2\text{COOCH}_3$ ), 8.01 ( $=\text{CHCH}_2-$ ), 7.79 ( $-\text{CH}_2\text{COOCH}_3$ ), and 4.72 $\tau$  ( $-\text{CH}=\text{CH}-$ ). From our studies of the various isomeric compounds, we have determined the long range deshielding effects of several groups, have shown that these are additive, and consider that it is possible to distinguish by NMR spectroscopy alone unsaturation in positions  $\omega 1$ -5 and  $\Delta 2$ -7. This means that for the methyl *cis*-octadecenoates only the  $\Delta 8$ -12 isomers are difficult to distinguish, and Frost claims that even this is possible, mainly on the basis of the shape of the  $(\text{CH}_2)_n$  signal (13). The identification of polyenoic esters is usually simpler.

### Chromatography

It is chromatography—particularly thin layer chromatography (TLC) and gas liquid chromatography (GLC)—which has most changed the behavior pattern of lipid chemists and biochemists in recent years, and we have used our series of synthetic esters to probe these techniques further. While confirming that isomeric *cis* and *trans* alkenoates can be separated from each other, we have also shown that there are minor differences, especially among the *cis*-octadecenoates, depending upon the position of the double bond (Fig. 3). This effect is also apparent in the series of isomeric *cis*-octadecenals and *cis*-octadecenyl acetates but not in the *cis*-octadecenols or *cis*-octadecenes (4,5,14-17).

GLC now is used routinely in lipid research, though sufficient care is not always taken in making structural

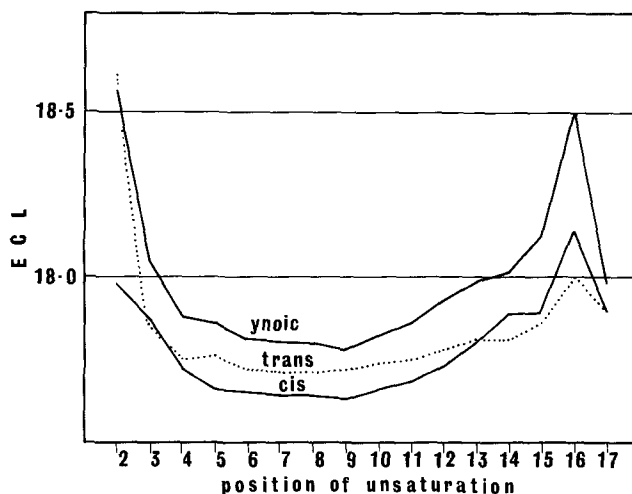


FIG. 4. Retention behavior (ECL) of isomeric methyl octadecenoates (*cis* and *trans*) and methyl octadecynoates on an APL column.

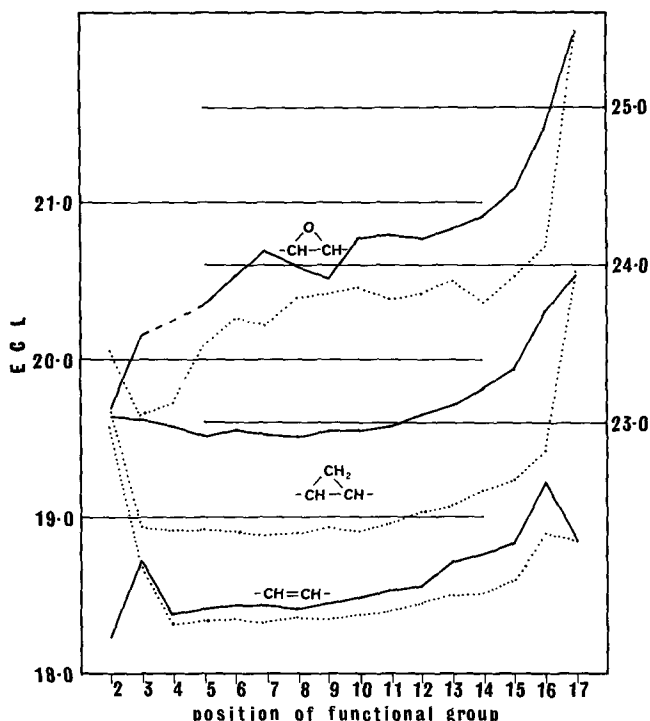


FIG. 5. Retention behavior of isomeric *cis* (—) and *trans* (· · ·) methyl octadecenoates (18.2-19.6), methyl methyleneoctadecanoates (18.9-20.6), and methyl epoxyoctadecanoates (23.0-25.5) on DEGS columns. Each isomer is indicated by a single number, e.g. methyl 2,3-epoxyoctadecanoate by 2, etc.

assignments to GLC peaks. We have investigated the GLC behavior of our synthetic esters on a variety of columns, mainly on ApL and DEGS, and some of our results are presented here (5-7,14-17). On an ApL column the *cis*-olefinic, *trans*-olefinic, and acetylenic esters behave slightly differently from each other, though for most isomers useful separations can be observed only on capillary columns. There are also small differences in retention behavior between esters differing only in the position of unsaturation. This is particularly true in the  $\Delta 2-5$  and the  $\Delta 10-17$  isomers (Fig. 4). The relative changes in retention behavior, observed first on ApL columns, are largely reproduced on more polar columns (XE60, NPGS, DEGS).

The difference between *cis* and *trans* isomers on ApL or DEGS columns is too small to be useful, and Emken (18) has described a chromatographic procedure for the determination of *cis* and *trans* alkenoates after quantitative epoxidation. This is based upon the more efficient chromatographic separation of *cis* and *trans* epoxides, at least over the range of double bond position expected in most hydrogenated fats. Our results (5,6,8) suggest that the cyclopropane derivatives would be more useful over a wider range of double bond position; but unfortunately the conversion of alkenoates to cyclopropanes is not so simple experimentally as epoxidation, nor does the reaction proceed in such good yield (Fig. 5).

We have also tried to predict retention behavior, expressed in terms of equivalent chain lengths (ECL), by the use of the concept of fractional chain length (FCL), and this has been developed by Ackman and his colleagues (19). We find that, in the important series of methylene-interrupted polyunsaturated esters, the FCL is not simply additive, and an additional correction factor has to be invoked (17) (Table I). Since this correction is fairly consistent through a range of compounds, it can be incorporated into the calculation. Though we cannot calculate ECL with a high degree of accuracy, it is possible to predict the order of elution of a series of closely related isomers, such as the 8 stereoisomers of methyl octadeca-9,12,15-trienoate. These results are most satisfactory on

TABLE I

Equivalent Chain Lengths and Fractional Chain Lengths of some Polyunsaturated  $C_{18}$  Esters on ApL Columns

Ester	ECL <sup>a</sup> (obs)	FCL <sup>b</sup>	ECL (calc) <sup>c</sup>	$\Delta(\times 100)$
18:1 (6c)	17.65	-0.35	—	—
18:1 (9c)	17.63	-0.37	—	—
18:1 (12c)	17.73	-0.27	—	—
18:1 (15c)	17.89	-0.11	—	—
18:2 (6c9c)	17.42	—	17.28	14
18:2 (9c12c)	17.50	—	17.36	14
18:3 (6c9c12c)	17.27	—	17.01	26
18:3 (9c12c15c)	17.49	—	17.25	24
18:4 (6c9c12c15c)	17.26	—	16.90	36

<sup>a</sup>ECL = Equivalent chain lengths.

<sup>b</sup>FCL = Fractional chain lengths.

<sup>c</sup>ECL (calc) for methyl linoleate is  $18.00 + (-0.37) + (-0.27) = 17.36$  and the remaining values are calculated in a similar way.

ApL columns where retention behavior is reproducible both within and between laboratories. Polar columns present added difficulties, because ECL is less reproducible on such columns.

### Biological Studies

The study of some biological properties (and of some other physical properties) of our synthetic acids has been undertaken collaboratively between ourselves and other laboratories in Europe and North America (20). I have selected for discussion here the work carried out by W.E.M. Lands and his colleagues (Michigan). This work seeks to discover the influence of fatty acid structure on the esterification reaction occurring between lysolecithins and acyl-CoA in the presence of acyltransferases derived from rat or pig liver microsomes. The enzymic acylation of 1-acylGPC and of 2-acylGPC has been examined with the four series of  $C_{18}$ -CoA esters derived from the *cis*-octadecenoic acids, the *trans*-octadecenoic acids, the octadecynoic acids, and the *cis*-methyleneoctadecanoic acids (21). Briefly, their results lead to the following conclusions: (a) The enzyme(s) responsible for acylation at the 2 position responds to an unsaturated center present at the  $\Delta 5$ ,  $\Delta 9$ , and  $\Delta 12$  positions independently of whether this is *cis*-olefinic, *trans*-olefinic, or acetylenic. (b) The enzyme(s) responsible for acylation at the 1 position responds to the conformation of the acyl group, so that there is little difference in reactivity between the *cis*-alkene and the similarly shaped *cis*-cyclopropane CoA esters. There is, however, a marked difference between the *cis*-alkenoates on the one hand and the *trans*-alkenoates and the alkynoates on the other. Lands has called attention to the alternating selectivity between the acetylenic and the *cis*-olefinic compounds, so that, for example, the 9c ester behaves like the 10t and the 10a esters, and the 10c ester behaves like the 11t and 11a esters. This is particularly true in the  $\Delta 9$  to  $\Delta 13$  region.

### CHEMICAL REACTIONS

In the reactions of a molecule with at least two functional groups, there is always the possibility that reaction at one functional group may be influenced by the second. This influence will depend upon the nature of the second functional group and on its position with respect to the first. For example, in the reactions of long chain esters containing the hydroxy alkene system  $-\text{CH}=\text{CH}(\text{CH}_2)_n\text{CH}(\text{OH})-$  we have discovered how reaction at the double bond may be influenced by the nearby hydroxyl group and how reaction at the hydroxyl group can be affected by the double bond and how, in both cases, the interaction depends markedly upon the number of methylene groups between the double bond and the hydroxyl group and sometimes on the configuration of the

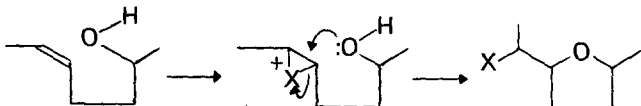


double bond. These studies have led us to new types of fatty acid derivatives and to reactions of potential value for analytical and diagnostic purposes.

We have, for example, discovered some reactions of methyl vernolate and of methyl ricinoleate which result in the formation of cyclopropane compounds and which can be explained in terms of the resonance known to occur in homoallylic carbenium ions:

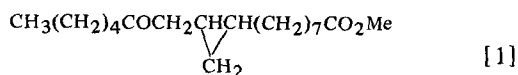


Also when a hydroxyl group becomes involved in reactions occurring at a double bond, intramolecular formation of a cyclic ether often occurs. We have observed this during epoxidation, halogenation, and oxymercuration of appropriate hydroxy alkenes:



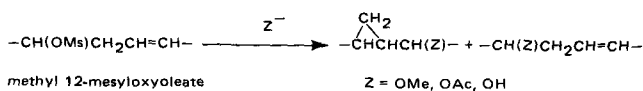
### Cyclopropane Esters from $\beta$ -Hydroxyalkenes and $\beta\gamma$ -Epoxyalkenes via a Homoallylic Rearrangement

We chanced upon this type of reaction when looking for a reagent which would convert methyl vernolate to methyl coriolate. We found that this rearrangement could be effected with lithium diethylamide ( $\text{LiNEt}_2$ ) (22) but not until we had investigated several other reagents, including boron trifluoride. As expected (23), this converted the epoxy ester mainly to oxo esters, including, surprisingly, some saturated compounds. In benzene solution these were formed in 34% yield and consisted mainly of the *cis* and *trans* isomers of the cyclopropane oxo ester [1]. We explained this observation in terms of the homoallylic



resonance which has been extensively studied among compounds of lower mol wt (24). Our results were confirmed by the independent observations of an Italian group (25).

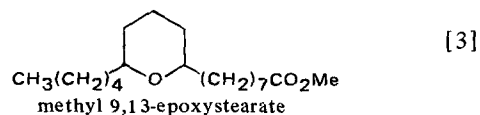
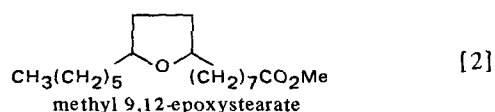
It then seemed to us that similar reactions should occur with suitable derivatives of methyl ricinoleate, and we showed that methyl 12-mesyloxyoleate can be converted in good yield (30-60%) to cyclopropane compounds so long as the reaction solution is kept alkaline (26). In acidic solution the cyclopropane compounds are unstable, and the



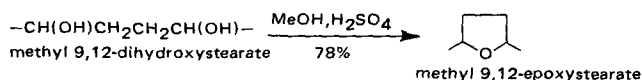
major product is a substituted octadecenoate in which the double bond is mainly *trans*. Similar results have been reported by Ucciani and Naudet (27) with methyl 12-tosyloxyelaidate. This reaction is confined to compounds which can furnish a homoallylic carbenium ion and is not observed, for example, in similar reactions of methyl 9-mesyloxyoctadec-12-enoate (a  $\gamma$ -hydroxy alkene).

### 1,4- (and 1,5-) $\text{C}_{18}$ Epoxides

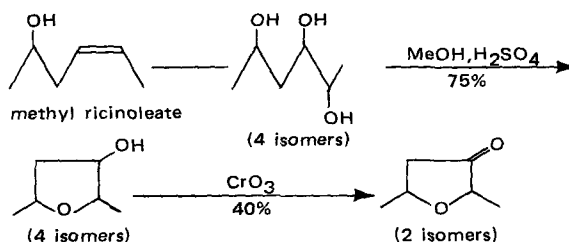
We recently have become interested in a new type of epoxy stearate ([2] and [3]), containing a five- or six-membered heterocyclic ring system, which is formed easily in several reactions of appropriate hydroxyalkenoates.



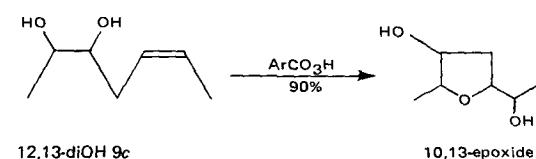
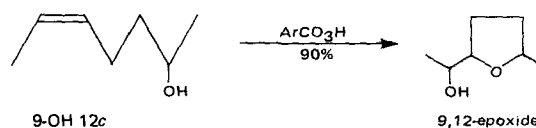
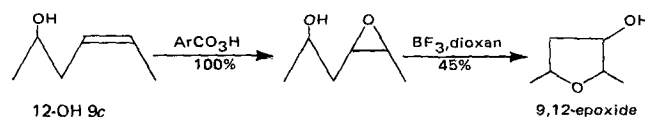
We first produced such compounds when treating methyl linoleate fairly vigorously under acidic conditions (28). The main product (48%) was a mixture of epoxy stearates (mainly 9,12 and 10,13 with a little 9,13), and we confirmed this by an independent synthesis of the 9,12-epoxide by acid-catalyzed cyclization of the already known methyl 9,12-dihydroxystearate. This reaction proceeded so



smoothly that we examined the cyclization of the methyl 9,10,12- and 9,12,13-trihydroxystearates which can be prepared from methyl ricinoleate and methyl 9-hydroxyoctadec-12-enoate respectively. Our studies led to an interesting investigation of the stereochemistry of the trihydroxy acids and of the hydroxy epoxides and oxo epoxides derived from them by cyclization and by oxidation (29). For example:



1,4-Epoxides can also be obtained from appropriate 1,2-epoxides (30). The 9,10-epoxide from the  $\beta$ -hydroxyalkene, methyl ricinoleate, for example, rearranges to a 9,12-epoxide when treated with boron trifluoride. In contrast, the  $\gamma$ -hydroxyalkene, methyl 9-hydroxyoctadec-12-enoate, did not give an isolable 12,13-epoxide but furnished the 9,12-epoxide directly. We believe, though we have not checked this, that epoxidation of methyl ricinoleate also might lead directly to the 9,12-epoxide. Methyl 12,13-dihydroxyoleate is both a  $\beta$ - and a  $\gamma$ -hydroxy alkene, but on epoxidation it reacts as the latter and gives the 10,13-epoxide directly.



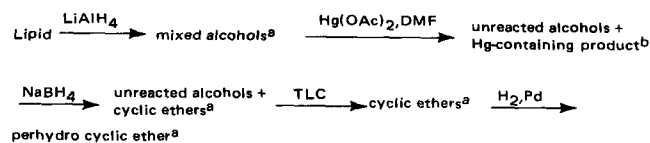
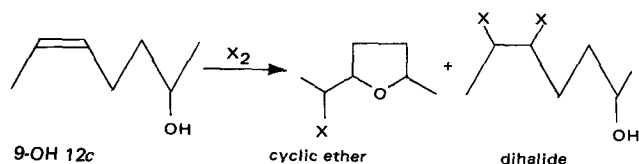
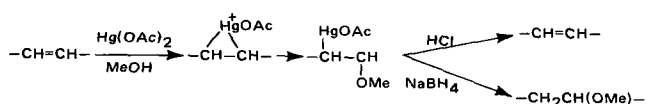


FIG. 6. Procedure for the examination of lipids containing fatty acids having unsaturation at  $\Delta 3$  (*trans*),  $\Delta 4$ , or  $\Delta 5$ . a = Product may be examined by gas liquid chromatography at these stages. b = Reaction product may be placed on a chromatographic column at this stage and eluted with ether to remove the unreacted alcohols and then with methanol containing hydrochloric acid to elute the mercury-containing product as regenerated alcohols having unsaturation at  $\Delta 3$  (*trans*),  $\Delta 4$ , or  $\Delta 5$ .

We have observed similar results during bromination and iodochlorination of hydroxyalkenoates and find the products to be a mixture of cyclic ethers and dihalides (31). Cyclic ethers are best obtained with  $\gamma$ -hydroxyalkenes and are formed in greater proportion during iodochlorination (~80%) than during bromination (~40%).



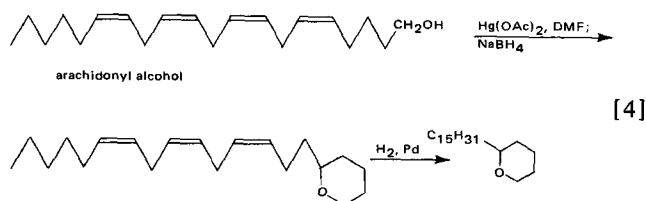
Of wider interest is the oxymercuration reaction which has been known for some time but which we now put to new use. In simple form this reaction occurs thus:



Methyl oleate, for example, by oxymercuration and demercuration, gives methyl 9(10)-methoxystearate in high yield (98%) and ethoxystearates (ethanol, 95%), acetoxystearates (acetic acid, 82%), and hydroxystearates (water, 77%) when methanol is replaced by the appropriate nucleophilic solvent (32).

The reaction proceeds on an intramolecular basis whenever the hydroxyl group and the double bond are disposed appropriately (33).  $\beta$ -Hydroxyalkenes form cyclic ethers only when the double bond has the *trans* configuration.  $\gamma$ -Hydroxyalkenes in either *cis* or *trans* configuration readily form tetrahydrofurans and  $\delta$ -hydroxyalkenes (*cis* or *trans*) form tetrahydropyrans in virtually quantitative yield. These reactions can be carried out in dimethylformamide solution but even when conducted in methanol intramolecular cyclization predominates, and no methoxy ethers are observed. Hydroxyalkenes are not common among the fatty acids, but there exist some important acids with unsaturation at  $\Delta 3t$  or  $\Delta 4$  or  $\Delta 5$  which can be reduced to alkenols and then converted easily to cyclic ethers. This observation can be employed as a means of identification or analysis.

Methyl arachidonate, for example, can be converted to the tetrahydropyran [4], and other acids with  $\Delta 5$  unsaturation will behave in a similar way. Acids with  $\Delta 4$  unsaturation (common among the  $C_{22}$ -polyenoic acids) produce the corresponding tetrahydrofurans.



[4]

The cyclic ethers are much less polar than the alcohols and are separated readily by TLC from those alcohols which do not form cyclic ethers. It is even possible to separate the alkyl and alkenyl tetrahydrofurans from the alkyl and alkenyl tetrahydropyrans and thereafter to separate each ether class into components of differing unsaturation by  $\text{Ag}^+$  TLC. Unreacted alcohols can be separated from the acetoxymethyl compounds after mercuriation and before demercuration; and  $\Delta 3$ ,  $\Delta 4$ , and  $\Delta 5$  alkenols can be regenerated from the latter by reaction with hydrochloric acid.

We have proposed a procedure for examining lipids containing acids with unsaturation at  $\Delta 3t$ ,  $\Delta 4$ , or  $\Delta 5$  which can be employed for analytical or isolation purposes and have applied this to tall oil, rat liver lipids, and to fish oils both before and after partial hydrogenation (34) (Fig. 6). By this procedure, for example, we showed the alcohols from pilchard oil to give cyclic ethers (19%) of which the major components were those arising from the  $\Delta 5$  alcohol 20:5 $\omega$ 3 (12.7%) and the  $\Delta 4$  alcohol 22:6 $\omega$ 3 (4.1%).

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(Continued on page 500A)



# GIT\* with it!

## SPECIAL FARES TO AOCS SPRING MEETING IN MEXICO CITY

GIT\* fares will apply to those registrants in a group of 15 or more from a given city (TCI will coordinate) who are staying a minimum of five nights in Mexico (not necessarily Mexico City). The fares and special hotel packages AOCS and TCI have designed, which include other special features, are described in a special brochure available from the AOCS Executive Director, 508 South Sixth Street, Campaign, Illinois 61820. The brochure describes several planned activities; however, if a registrant wants to vary his or her plan, TCI can tailor the trip to the registrant.



Earlier announcements in *JAACS* have outlined a few of the unique features of the 1974 Spring Meeting, April 27-May 2, 1974, in Mexico City. Many departures from tradition are planned for the Meeting, which itself is unique, because it is the first AOCS national meeting in a non-English speaking country.

Another unique feature afforded by the Spring Meeting is the opportunity for the Society to take advantage of the services of Travel Consultants, Incorporated, one of the world's largest and finest convention travel agents.

TCI will be able to route registrants at the lowest possible travel rates without any inconvenience to the individual involved. TCI can provide this service by organizing and grouping individuals traveling to Mexico City along the same airline routes. For example, 30-50 AOCS registrants may be traveling to Mexico City via Chicago. By arranging travel plans for all these people, TCI can form Chicago to Mexico City groups at a substantial savings to the registrant.

It's important to note that these groups will have to fly together *only one way*; a registrant can return at his or her convenience. In addition, it is not necessary for a registrant to have a group from his or her own city. A registrant may wish to be included with a group somewhere along the way; that portion of travel with the AOCS group will be at the reduced rate. A registrant also can make his or her own air reservations, simply give TCI the air schedule, and they can issue the tickets.

There is a wide variety of group travel plans available to the traveler. The chart below shows comparable fares (as of April 1, 1973) and the possible savings with the GIT\* group program. GIT\* was picked for illustrative purposes because many of the registrants will, no doubt, want to take advantage of the Mexico City meeting to extend their stay for vacation and sightseeing.

### SAMPLE ROUND TRIP AIR FARES TO MEXICO CITY INCLUDING TAX

(Effective April 1, 1973)

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Boston	399.00	315.00	218.00	97.00
Chicago	315.00	245.00	167.00	78.00
Cleveland	351.00	273.00	196.00	77.00
Dallas	203.00	155.00	110.00	45.00
Detroit	339.00	261.00	196.00	65.00
Indianapolis	311.00	243.00	167.00	76.00
Los Angeles	293.00	221.00	156.00	65.00
Miami	245.00	195.00	139.00	56.00
New York City	385.00	295.00	218.00	77.00
St. Louis	289.00	221.00	151.00	70.00
San Francisco	349.00	261.00	177.00	84.00

\*GIT refers to Group Inclusive Tour. Registrants in a group of 15 or more leaving from a given city (TCI will coordinate) who are staying a minimum of five nights in Mexico will be able to take advantage of GIT. There is an airline cancellation penalty of 25 percent of the air fare for those persons cancelling less than 21 days prior to departure. Prices quoted above are sample round trip air fares.

## • Fats and Oils

EFFECTS OF SEVERAL FOLIAR FUNGICIDES ON THE FATTY ACID COMPOSITION AND STABILITY OF PEANUT OIL. R.E. Worthington and D.H. Smith (Dept. of Food Sci. and Plant Pathol., Univ. of Georgia College of Agr. Exp. Sta., Experiment, Ga. 30212). *J. Agr. Food Chem.* 21, 619-21 (1973). Foliar fungicides applied to peanuts for the control of *Cercospora* leafspot during one and/or two growing seasons caused small but statistically significant ( $p = 0.01$ ) changes in fatty acid composition of peanut oil. Levels of linoleic acid were higher (ca 1 to 2%) in oil obtained from Argentine and Florunner varieties and lower (ca 0.5 to 1%) in Florigiant variety plots treated with fungicide compared with control samples. Treatment effects on oil stability (autoxidation induction period) were small but occasionally statistically significant. Treatment effects on oil stability and fatty acid composition were in all cases, however, no larger than normal year to year fluctuations.

CHARACTERIZATION OF A BLOOD GROUP B GLYCOLIPID, ACCUMULATING IN THE PANCREAS OF A PATIENT WITH FABRY'S DISEASE. J.R. Wherrett (Dept. of Med. (Neurology), Univ. of Toronto, Toronto 5, Canada) and Sen-Itiroh Hakomori. *J. Biol. Chem.* 248, 3046-51 (1973). An accumulation of a ceramide hexasaccharide was found in the pancreas of a patient with Fabry's disease, in addition to the accumulation of ceramide trihexoside and ceramide digalactoside. The ceramide hexasaccharide was isolated and identified as a blood group B-active glycolipid with the following structure: Gal $\alpha$ 1  $\rightarrow$  3Gal $\beta$ 1  $\rightarrow$  3 (and 1  $\rightarrow$  4)GlcNAc $\beta$ 1  $\rightarrow$  3Gal $\beta$ 1  $\rightarrow$  4Glc  $\rightarrow$  Cer  
 $\uparrow$   
 2  
 $\uparrow$   
 Fuc

The proportion of two structures, one containing Gal1  $\rightarrow$  3GlcNAc and one containing Gal1  $\rightarrow$  4GlcNAc, is 4:1.

MEASUREMENT AND SPECTRAL CHARACTERISTICS OF FLUORESCENT PIGMENTS IN TISSUES OF RATS AS A FUNCTION OF DIETARY POLYUNSATURATED FATS AND VITAMIN E. K. Reddy, B. Fletcher, Ardelle Tappel and A. Tappel (Dept. of Food Sci. and Technol., Univ. of California, Davis, Cal. 95616). *J. Nutr.* 103, 908-15 (1973). The effect of dietary polyunsaturated fats and vitamin E on accumulation in rats of fluorescent ceroid and lipofuscin-like pigments was studied by fluorescence measurements of tissue lipid extracts. Rats fed 10% lard and 1% cod-liver oil diets without added vitamin E for 4 months accumulated twice the fluorescent pigments as those fed the same diet but supplemented with 45 mg vitamin E per kilogram diet. Accumulation of pigments in adipose of rats fed 15.7% corn oil or cod-liver oil diets was inversely proportional to the dietary vitamin E concentration; the adipose of animals fed cod-liver oil had approximately three times the fluorescence of adipose from animals fed corn oil. Bone marrow, heart and spleen of rats fed vitamin E-deficient cod-liver oil diets accumulated more fluorescent pigment than vitamin E-supplemented rats. The fluorescent pigments had excitation maxima at 360 to 390 nm and fluorescence maxima at 430 to 470 nm. Weight gain was proportional to dietary vitamin E, especially that of rats fed cod-liver oil diets. The data suggest that tissue extraction and fluorescence quantitation can be used successfully as an index of fluorescent ceroid and lipofuscin-like pigment accumulation.

VIRUS-INDUCED CHOLESTEROL CRYSTALS. C.G. Fabricant, L. Krook and J.H. Gillespie (New York State Vet. College, Cornell Univ., Ithaca, N.Y.). *Science* 181, 566-7 (1973). One of the crystal types induced in cell cultures by a new feline herpes-virus was identified as cholesterol by crystal structure, polarized light microscopy and mass spectroscopy.

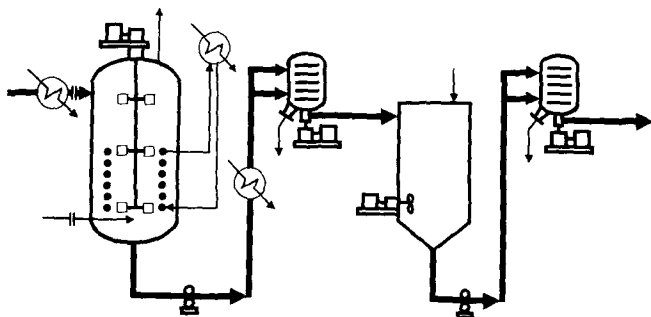
APPLICATION OF LASER SELF-BEAT SPECTROSCOPIC TECHNIQUE TO THE STUDY OF SOLUTIONS OF HUMAN PLASMA LOW-DENSITY LIPOPROTEINS. R.W. DeBlois, E.E. Uzgiris, S.K. Devi and A.M. Gotto, Jr. (General Electric Res. and Dev. Center, Schenectady, N.Y. 12301). *Biochemistry* 12, 2645-9 (1973). The technique of self-beat or homodyne spectroscopy has been applied to the determination of the translational diffusion constant of human plasma low-density lipoproteins (LDL). Both power spectrum and autocorrelation methods give equivalent results, but with conventional equipment the power spectrum measurement takes on the order of hours while autocorrelation is accomplished in minutes. The effects of concentration of LDL, scattering angle of the light, pH of the solution and buffer concentration were investigated. The diffusion constant obtained,  $D_{s,w} = 2.14 \pm 0.09 \times 10^{-2} \text{ cm}^2/\text{sec}$ , was in good general accord with conventional measures. The equivalent spherical diameter, obtained from the Stokes-Einstein relationship,  $229 \pm 10 \text{ \AA}$  was within a broad band of values given by other techniques.

USE OF CHLOROMETHYLSILYL ETHER DERIVATIVES FOR THE DETERMINATION OF HYDROXYLATED STEROIDS BY GAS CHROMATOGRAPHY-MASS SPECTROMETRY. J.R. Chapman and E. Bailey (AEI Scientific Apparatus Ltd., Barton Dock Rd., Urmston, Manchester, England). *Anal. Chem.* 45, 1636-41 (1973). Halomethyldimethylsilyl ethers, particularly chloromethyldimethylsilyl (CDMS) ethers, are valuable new derivatives for the determination of hydroxylated steroids by combined gas chromatography-mass spectrometry (GC-MS) techniques. The mass spectra of the CDMS ethers are particularly useful in distinguishing steroids that differ in the stereochemistry of the 3-hydroxyl group or of the A/B ring junction. Further distinction of compounds, such as the di-CDMS ethers of pregnanediols, that differ only in the stereochemistry of the 20-hydroxyl group is possible on the basis of retention data. The mass spectra of most CDMS ethers also show relatively intense peaks at high mass, particularly the  $M^+ - \text{CH}_2\text{Cl}$  peak. Because of this, these derivatives are ideally suited to the quantitative determination of hydroxylated steroids at low levels by single and multiple peak monitoring techniques. Examples of the determination of dehydroepiandrosterone and testosterone in plasma extracts by single peak monitoring are presented.

AN ISOLATION AND CLEANUP PROCEDURE FOR LOW LEVELS OF ORGANOCHLORINE PESTICIDE RESIDUES IN FATS AND OILS. M.L.



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Porter and J.A. Burke (Div. of Chem. & Physics, FDA, Washington, D.C. 20204). *J. Assn. Off. Anal. Chem.* 56, 733-38 (1973). An isolation and cleanup procedure is described for levels of about 0.005 parts per million organochlorine residues in fats and oils, prior to analysis by electron capture gas-liquid chromatography. The fat or oil is distributed on a column of unactivated Florisil and the residues are partitioned into an eluant of 10% water in acetonitrile. Florisil column chromatography with an elution solvent system comprised of mixtures of methylene chloride, acetonitrile and hexane is used for the final cleanup. Extracts prepared in this manner are sufficiently free of fatty extractives to permit injection of the equivalent of 50-60 milligrams of fat sample for GLC. The procedure should be especially useful in determination of low levels of organochlorine pesticide residues in the fat of certain dietary composites.

EFFECT OF POSTMORTEM AGING ON CHICKEN MUSCLE LIPIDS. J.D. Hay, R.W. Currie and F.H. Wolfe (Dept. of Food Sci., Univ. of Alberta, Edmonton, Canada). *J. Food Sci.* 38, 696-9 (1973). Neutral lipids of fresh chicken breast muscles are shown to be triglycerides, sterols and sterol esters with only traces of mono- and diglycerides and free fatty acids. Phospholipids include measurable quantities of phosphatidyl choline, phosphatidyl ethanolamine, phosphatidyl inositol, phosphatidyl serine, sphingomyelin, diphosphatidyl glycerol, lysophosphatidyl choline and lysophosphatidyl ethanolamine. Fatty acid analyses of several of the lipid fractions are also included. Decreases in phosphatidyl choline and phosphatidyl ethanolamine coupled with increases in lysophosphatidyl choline, lysophosphatidyl ethanolamine and free fatty acids after 48 hours postmortem in the cold indicate phospholipase A activity concurrent with other postmortem changes. The significance of the results is discussed.

FACTORS RELATING TO COMPLETENESS OF SOLVENT EXTRACTION OF DIELDRIN FROM MILK. C.Y.W. Ang and L.R. Dugan Jr. (Dept. of Food Sci. and Human Nutr., Michigan State Univ., East Lansing, Mich. 48823). *J. Assn. Off. Anal. Chem.* 56, 718-20 (1973). Milk containing added dieldrin was separated into 2 lipid fractions by differential solvent extraction. Fraction 1 was extracted with nonpolar hydrocarbon solvents and Fraction 2 with the same solvent system after mixing with sodium oxalate and ethanol. Three solvent systems were compared. Dieldrin concentrations in Fraction 1 of all 3 solvent systems were much higher than in Fraction 2 on a fat basis. Less than 50% of the dieldrin was recovered in Fraction 1, probably because of low recovery of hydrocarbon solvents from fluid milk or factors that depressed the separation of solvents from milk. Factors possibly involved were the adsorption of solvents and pesticide to the hydrophobic groups of membrane proteins and/or serum proteins or the penetration of solvents and pesticide into the fat globules of milk.

CHEMICAL COMPOSITION OF ALLEPPY CARDAMOM OIL BY GAS CHROMATOGRAPHY. A.K.S. Baruah, S.D. Bhagat and B.K. Saikia (Regional Res. Lab., Jorhat, Assam, India). *Analyst* 98, 168-71 (1973). An isothermal gas-chromatographic technique used to investigate the chemical composition of cardamom oil is described. A total of twenty-one components were detected by this method. Most of the peaks, representing 98.1% of the oil, have been identified.

WHICH LIPID SYSTEMS FOR ASEPTICALLY PROCESSED FOODS? A.V. Petricea (Aseptically Processed Foods, Glidden-Durkee Div., SCM Corp., Joyce Res. Ctr., Strongsville, Ohio). *Food Eng.* 45(5), 96-9 (1973). A review article written to help food processors adapt lipid systems in existing or totally new products to the requirements of aseptic processing.


GLYCOLIPIDS: KEY TO PROTEIN-ENRICHED BREAD. Y. Pomeranz (National Barley and Malt Lab., ARS, USDA, Madison, Wis.) and K.F. Finney. *Food Eng.* 45(5), 134-42 (1973). The protein level in bread may be increased 70% without impairing consumer acceptance by the addition of glycolipids. The use of these materials entails practically no changes in dough formulation, production schedules or equipment.

GAS CHROMATOGRAPHIC DETERMINATION OF BROMINATED SESAME OIL IN ORANGE DRINKS: COLLABORATIVE STUDY. H.B.S. Conacher (Res. Labs., Health Protection Branch, Ottawa, Ontario, Canada K1A 0L2). *J. Assn. Off. Anal. Chem.* 56, 602-6 (1973). A gas-liquid chromatographic method for the determination of brominated vegetable oils in soft drinks was collaboratively studied, using a commercial orange drink spiked with known amounts of brominated sesame oil. Initial results based on calculations involving total peak area measure-

ments were considered erroneously high, since drink components co-eluted with the C<sub>16</sub> and C<sub>18</sub> methyl esters, especially at low levels of brominated oil. A modified calculation, based only on brominated ester content, showed recovery values (6 collaborators) of 94.6, 95.3, and 96.0% for drinks containing 3.10, 5.22, and 10.66 milligrams per 10 fluid ounces, respectively, with corresponding standard deviation values of 0.40, 0.46, and 0.64. The method with the modified calculation incorporated has been adopted as official first action.

COLLABORATIVE STUDY OF THE FOSS MILKO-TESTER METHOD FOR MEASURING FAT IN MILK. W.F. Shipe and G.F. Senyk (Dept. of Food Sci., Cornell Univ., Ithaca, N.Y. 14850). *J. Assn. Off. Anal. Chem.* 56, 538-40 (1973). In a third collaborative study, the Milko-tester was compared with the Babcock method in 9 laboratories. In addition, 3 laboratories analyzed the samples by an ether extraction method. The standard deviations for these methods were +0.018, +0.032, and +0.044%, respectively. These deviations are based on triplicate determinations on 5 samples by each of the laboratories. The average values for all samples were 3.99, 3.96 and 3.88% for the Milko-tester, Babcock, and ether extraction methods, respectively. The standard deviation of differences between the Milko-tester and Babcock for all 9 laboratories was +0.077%.

COMPARISON OF TWO EXTRACTION PROCEDURES FOR THE DETERMINATION OF CHOLESTEROL IN DAIRY PRODUCTS. D.E. LaCroix, R.M. Feeley, N.P. Wong and J.A. Alford (Dairy Prod. Lab., Eastern Mkt. Nutr. Res. Div., ARS, USDA, Beltsville, Md. 20705). *J. Assn. Off. Anal. Chem.* 55, 972-4 (1972). Both the Roesse-Gottlieb (R-G) and silicic acid methods have been used for the extraction of lipids from dairy products. However, the silicic acid method is widely used for the extraction of fatty acids for determination by GLC. Since it would be advantageous to use this extract for cholesterol determinations, a comparison of the extraction procedures for dairy products was carried out to determine if they gave equivalent values. Cholesterol values were determined by GLC of both extracts. Twenty samples of 6 dairy foods having milk-fat contents



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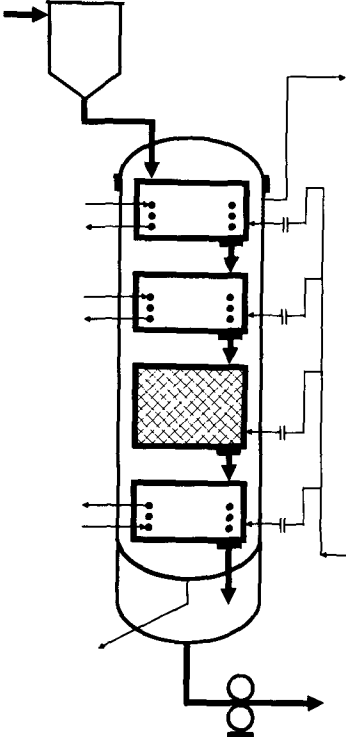
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ranging from 3 to 38% were compared. In every instance the R-G extracts had higher values for cholesterol. The R-G or similar multiple solvent extract methods should be used for all dairy products and any other food product in which part of the cholesterol may be bound to a lipoprotein or otherwise not free in the fat.

EXTRACTION OF LIPID-PROTEIN COMPLEXES FROM EGG YOLK. J.G. Navarro, F. Borie and J. Saavedera (Dept. Quimico, Facultad de Quimica y Farmacia, Universidad de Chile, Casilla 233-Santiago, Chile). *J. Assn. Off. Anal. Chem.* 55, 975-8 (1972). A residual lipid which is not removed by solvent extraction was detected and determined after enzymatic hydrolysis of defatted egg yolk protein. Free fatty acids were found to be the type of lipid bonded to the egg yolk protein. The mode of attachment of these fatty acids to the egg yolk protein is suggested and the composition of the fatty acids is reported.

GAS CHROMATOGRAPHIC DETERMINATION OF PROPIONIC, SORBIC AND BENZOIC ACID IN RYE BREAD AND MARGARINE. A. Graveland (Inst. Cereals, Flour and Bread TNO, Wageningen, The Netherlands). *J. Assn. Off. Anal. Chem.* 55, 1024-6 (1972). A rapid and accurate method is presented for the GLC determination of propionic and sorbic acids in bakery products and benzoic acid in margarine. The acids are extracted with ether containing orthophosphoric acid, to which valeric acid is added as internal standard. The extract is injected directly into the gas chromatograph. Glass columns (2 meters  $\times$  2.0 millimeters inside diameter) containing 5% Carbowax 20M-terephthalic acid coated on Chromosorb W(AW-DMCS) are used for analysis. The simplicity and convenience of the method make it suitable for routine determinations.

FISH PRESERVATION. I. STUDIES ON CHANGES DURING FROZEN STORAGE OF OIL SARDINES. L.N. Srikar and G.G. Hiremath (College of Fisheries, Univ. of Agr. Sciences, Mangalore, India). *J. Food Sci. Technol. (India)* 9, 191-3 (1972). The effect of various glazes i.e. (1) butylated hydroxy anisole, butylated hydroxy toluene mixture; (2) mono-sodium glutamate; and (3) ascorbate and citrate mixture in suppressing rancidity during frozen storage of oil-sardine was studied. No significant difference in the effect of antioxidants was found. However the storage life was extended if the fish was frozen in blocks after filling the air space with glazing solution. There was an increase in peroxide value (PV) and free fatty acids (FFA) during storage and the rate of increase of PV was more than that of FFA. An inverse relation existed between PV and salt soluble nitrogen and FFA and salt soluble nitrogen.

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INFLUENCE OF MIXING REGIME OF OIL AND WATER ON THE DEGREE OF ELIMINATION OF PHOSPHATIDES. S.N. Volotovskaja et al. (VNIIZ). *Maslozir. Prom.* 1973(1), 16-18. Degumming of sunflowerseed oil using intensive mixing with water has been examined. The quantity of water is 1% on the oil, temperature 45-50C, and contact time 20 minutes. Separation is made after 2 hours by decantation at 45C. The experiments were done in Visnevskij apparatus equipped with a special agitator (2,800 rpm). The same oil was also degummed by the conventional technique. Residual phosphorus in the oil degummed with intensive mixing was equivalent to 0.011-0.015%  $P_2O_5$ ; in oil degummed by conventional method it was 0.020-0.030%. (Rev. Franc. Corps Gras)

COTTONSEED PHOSPHOLIPIDS: IV—ISOLATION AND CHARACTERIZATION OF COTTONSEED LECITHIN. A.S. El-Nockrashy and Y. El-Shattery (Lab. for Fats and Oils, National Res. Center, Dokki, Le Caire). *Rev. Franc. Corps Gras* 20, 217-20 (1973). Lecithin (LE) has been isolated from mixed cottonseed phospholipids by column chromatography (yields 33.9 and 35.8% respectively). GLC analysis of the fatty acid methyl esters revealed saturated-unsaturated fatty acid ratios of 1:2.01 and 1:1.86 respectively. Palmitic acid constitutes 90% of the total saturated fatty acids, and linoleic acid makes up more than 80% of the total unsaturated. The nitrogen-phosphorus ratios are 1/1 and 1/0.9 for the two species, respectively. Gossypol present in the isolated LE accounts for less than 10% of the gossypol present in the mixed phospholipids. IR spectrum of isolated LE gives bands reported for LE.

SOYBEAN CULTURE IN FRANCE: PERSPECTIVES. E. Chone (Development Service of CETIOM, Paris). *Rev. Franc. Corps Gras* 20, 211-5 (1973). A study which has been done in this field by CETIOM in 1966 is reviewed. The results of this study induced the French administration to introduce soya in the EEC's regulation 136. This study comprises a varietal experimentation and an extensive research on three cultural techniques: inoculation, irrigation and weed control. Large scale culture is described in Sud-West of France. Perspectives of development of soybean in France show that probably 130,000 hectares would be cultivated and about 280,000 tons of soybean produced.

STUDY OF OXIDIZED ACIDS OCCURRING IN FATS AND OILS. IV.—EXAMPLES OF APPLICATION OF THE THIN-LAYER CHROMATOGRAPHY METHOD FOR OXIDIZED ESTERS. J. Graille and M. Naudet (National Lab. of Oils and Fats—ITERG, Univ. of Provence, Marseille, France). *Rev. Franc. Corps Gras* 20, 203-9 (1973). Oxidized acids are present in practically all crude or refined oils; their determination is very important. Oxidized esters isolated from peanut, rapeseed, soybean, sunflower, corn and palm oils and tallow were studied. Chromatoplates of these oxidized esters show that the  $R_f$  of the different components is constant, independent of the kind of fat, its origin and its state of refining. The comparison of chromatoplates for oxidized esters isolated from the fatty acids obtained by saponification, chemical hydrolysis or enzymatic hydrolysis, shows that the separation of the oxidized esters into their constituents depends on the method of obtaining the fatty acids. Hydrolysis by non-specific lipase did not alter the natural repartition; saponification and chemical hydrolysis did.

CONCENTRATION OF THE UNSAPONIFIABLE MATTER IN OILS. H. Kallel and C. Paquot (CNRS-2, 94320 Thiais, France). *Rev. Franc. Corps Gras* 20, 147-51 (1973). The unsaponifiable matter of some oils contains important components and many methods for the isolation have been applied. In this paper, the concentration of unsaponifiable matter by solvent extraction from Tunisian virgin olive oil is described. The treated oil contains 0.75% unsaponifiable matter, determined by extraction with hexane. The concentration of unsaponifiable matter was done using the benzene/methanol system (27/75-v/v). The parameters for this technique are discussed. By decreasing the temperature to -30C, it is possible to obtain an extract 9 times richer in unsaponifiable matter than treated oil.

OXIDIZED FATTY ACID FORMATION DURING STORAGE. M. Naudet and S. Biasini (National Lab. of Oils and Fats—ITERG, Univ. of Provence, Marseille, France). *Rev. Franc. Corps Gras* 20, 143-5 (1973). For many reasons, oxidized fatty acids are almost inevitably present in crude vegetable oils. Data about the content of oxidized fatty acids in some crude vegetable oils are given in the paper; the content ranges from 0.20 to 2.75g on 100g of total fatty acids. They are largely formed during the storage of the seeds (peanut, rapeseed, soybean or sunflower) by the action of their own lipoxygenases.

The elimination of oxidized fatty acids during the refining processes is only partial and most are found in refined product. If edible oils with only a small amount of oxidized acids are wanted, the presence of these acids in crude oil must be avoided. For that, it is absolutely necessary to know when they are formed and the causes of their formations.

**GLYCERIDIC STRUCTURE OF CANBRA OIL.** J.P. Sergiel (Station for Res. of Food Qual., INRA, Dijon, France). *Rev. Franc. Corps Gras* 20, 137-41 (1973). Structure of Canbra oil triglycerides has been studied by fractionation on silica gel/Ag NO<sub>2</sub> TLC and hydrolysis by pancreatic lipase. The oleic acid content is 61% and its distribution in the different positions of triglycerides varies according to the degree of unsaturation of the triglycerides. Linoleic and linolenic acids are preferentially esterified in the internal position, whereas erucic and gadoleic acids are esterified in external positions. Canbra oil contains principally triunsaturated triglycerides (38.4%) with triolein predominating (28%) and the tetraunsaturated dioleolinoleic triglyceride, dioleolinolinin (21%).

**DETERMINATION OF TRACE QUANTITIES OF METALS IN OILS BY ATOMIC ABSORPTION (GRAPHITE FURNACE).** A. Prevot and M. Gente (Inst. for Fats and Oils, Paris, France). *Rev. Franc. Corps Gras* 20, 95-98 (1973). Determination of small quantities of metals in oils is very important; especially important are iron and copper, which initiate oxidation, nickel that originates from hydrogenation, calcium and magnesium derived from phospholipids and sodium remaining after neutralization. For metal determination, atomic absorption is recommended, especially when atomization is done in a graphite furnace. In this way, sensitivity is increased compared with classical atomic absorption.

**DETERMINATION OF THE WAX CONTENT IN SUNFLOWER SEEDS, HULLS AND OIL. DEVELOPMENT OF A MODIFIED EXTRACTION METHOD.** Biserka Ostric-Matijasevic and J. Turkulov (Faculty of Technol., Univ. of Novi Sad, Novi Sad, Yugoslavia). *Rev. Franc. Corps Gras* 20, 5-10 (1973). This method is a modification of Zolocewskij and Sterlin's method which consists of selective extraction by means of different solvents. The modified method is more rapid and accurate. A new extractor is used and described. Extraction is done at low temperature and the waxes are isolated without traces of other components (triglycerides, phosphatides, FFA, etc.). The wax content in sunflowerseeds is 0.09-0.11%; in hulls 0.33-0.43%; in crude oil 0.04-0.08% and in the solid phase after winterization 3.3-3.7%.

**LIPYOXYGENASE ACTION DURING BREAD MAKING. OXIDATION OF ESSENTIAL FREE FATTY ACIDS, CAROTENOIDS AND TOCOPHEROLS; HEXANAL FORMATION.** R. Drapron (Biochem. and Physicochemistry Station for Cereals of INRA-CERDIA, 91305 Massy, France). *Rev. Franc. Corps Gras* 20, 83-87 (1973). In breadmaking, during the high speed mixing of wheat flour containing soy bean flour, an almost complete disappearance of free linolenic acid is observed, whereas the content of saturated fatty acids and oleic acid remains constant. Complete destruction of carotenoids and of the major part of tocopherols is also observed, while the content of vitamins B<sub>1</sub> and B<sub>2</sub> doesn't change. The disappearance of essential fatty acids and vitamins is explained by their oxidation by lipoxygenase. The linoleic acid hydroperoxide formed is destroyed to yield hexanal which is responsible, in part, for the bread flavor alteration.

**ANALYTICAL PROBLEMS IN THE FIELD OF FATS AND OILS.** J.P. Wolff (Superior School for Fats and Oils Application, Wolff Labs., Paris, France). *Rev. Franc. Corps Gras* 20, 79-82 (1973). The applications of modern analytical methods for the fats industry are reviewed as well as the positive and the negative aspects of some of them. In some cases the accuracy of the analysis makes control problems by detecting "pollution" technically quite normal when an oilmill produces different simultaneously oils. The following example is shown: it is easy to determine 0.2% of erucic acid in the mixture of fatty acids; if this is found in peanut oil, we cannot consider this oil pure. But if peanut and rapeseed oils were treated in the same factory this "pollution" nearly is unavoidable.

**STEROL DISTRIBUTION DURING DEODORIZATION IN VEGETABLE OILS.** M. Naudet, M. Rakotovao and G. Cecchi (National Lab. for Fats and Oils—ITERG, Univ. of Provence, Marseille, France). *Rev. Franc. Corps Gras* 20, 27-31 (1973). Sterols obtained in distillates during deodorization are free sterols but they represent only a small fraction of the total sterols of the

treated oil. The quantitative composition of the sterol fraction of this distillate is significantly different from those of total sterols. The differences observed can not be explained by molecular weight differences alone, sterol-triglycerides molecular association must be taken in consideration.

**ULTRAVIOLET SPECTROPHOTOMETRY, A METHOD FOR MEASURING THE STATE OF OXIDATION OF UNSATURATED LIPIDS.** N. Yanishlieva and A. Popov (Inst. Organic Chem., Acad. of Science of Bulgaria, Sofia, Bulgaria). *Rev. Franc. Corps Gras* 20, 11-26 (1973). The content of the conjugated diene is used as a criterion for measuring the degree of oxidation of unsaturated lipids. The experiments have been done with oleate and linoleate as model substances and sunflower seed and olive oil as natural substances. The diene content is estimated by the UV absorption at 232 nm ( $E_{0.2\%}^{232}$ ). The different factors

which influence this absorption were studied: degree of unsaturation, oxidation degree (hydroperoxides, parasitic absorption, etc.). The authors have prepared a table of  $E_{0.2\%}^{232}$  values for evaluating the degree of oxidation, taking into account both the unsaturation (expressed in % of linoleate) and the peroxide value.

**DIETARY POLYUNSATURATES AND SERUM ALPHA-TOCOPHEROL IN ADULTS.** M.M. Christiansen and E.B. Wilcox (Dept. Nutrition and Food Sciences, Utah State Univ.). *J. Am. Dietetic Assoc.* 63, 138-42 (1973). The effects of diets with and without an increased amount of polyunsaturated fatty acids on serum alpha-tocopherol concentration and on susceptibility of erythrocytes to peroxide hemolysis was measured in 20 normal subjects. For 24 weeks, half of the subjects ate a self-selected modified diet which had a polyunsaturated:saturated fatty acid ratio of 1.0 or greater, compared with 0.45 for the controls. Dietary tocopherol:fatty acid ratios (E:PUFA) were calculated. Tocopherol levels in sera from the same individual showed wide variation from week to week. However, mean tocopherol values were within normal limits and were approximately the same for men and women. Serum tocopherol levels did not appear to be directly influenced by the E:PUFA ratio, but were related to dietary tocopherol. The high polyunsaturated fat diet did not cause a decrease in serum tocopherol levels under the conditions of the study, rather a slight increase. Other measures of vitamin E status, i.e., creatine:creatinine ratio and erythrocyte hemolysis, emphasized that no deficiency of tocopherol was evident in the tissues. An increased susceptibility of red blood cells to hemolysis by hydrogen peroxide was not found in the experimental group. The use of increased amounts of highly unsaturated fats as readily available vegetable oils contributed substantial amounts of tocopherol to the diets.

**PREPARATION OF A LACTIC SPREAD.** C.J. Cox and J.J. Hepburn (Lever Bros.). *U.S. 3,749,583*. The process uses the steps of incubating under microaerophilic conditions with thermobacteria an aqueous dispersion containing 0.5-25% protein, 15-70% fat, and a dry matter content of 20-75%, to obtain a pH of 4.8-5.4. The incubated mixture is pasteurized, cooled, and worked to form a plastic mass.

**CATALYST FOR SELECTIVE HYDROGENATION OF POLYUNSATURATED VEGETABLE OILS.** S. Koritala (U.S. Sec'y of Agriculture). *U.S. 3,749,681*. There is described a catalyst which will effectively reduce the linolenic acid moiety of polyunsaturated vegetable oils. A procedure for the preparation of the highly active catalyst by chemisorption of copper ammonium complex on silica gel is described. The catalyst has excellent reuse properties.

**OXIDATION PROCESS FOR THE PRODUCTION OF FATTY ACIDS.** K.P. Kammann, Jr. (Emery Industries, Inc.). *U.S. 3,749,745*. Primary and secondary long chain n-alkylbenzenes are oxidized

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in the presence of a catalyst at atmospheric pressure to produce fatty acids. The process gives an improved distribution of desirable high molecular weight fatty acids. When primary *n*-alkylbenzenes are used, the process is highly selective to acids of specific chain lengths to the near exclusion of low molecular weight, less desirable, fatty acids.

**SHORTENING FOR HIGHLY AERATED CREAMY FROSTINGS.** P. Seiden (Procter & Gamble). *U.S. 3,751,265*. The shortening comprises: (a) a liquid glyceride such as partially hydrogenated soybean oil, (b) propylene glycol monostearate, (c) partial esters of polyglycerol and (d) polyoxyethylene sorbitan tristearate.

**EXTRACTION OF UNSAPONIFIABLE FRACTIONS FROM NATURAL FATS.** A. Rancurel (Laboratoires Pharmascience). *U.S. 3,751,442*.

**CLEAR COOKING AND SALAD OILS HAVING HYPOCHOLESTEROLEMIC PROPERTIES.** B.A. Erickson (Procter & Gamble). *U.S. 3,751,569*. The oil is prepared by adding 0.5–10% (free sterol equivalent) of plant sterol monocarboxylic acid ester to clear, liquid base oil.

**PROCESS FOR ANTIOXIDATION AGAINST LIPID.** S. Maruyama and T. Wakayama (Kongo Yakuhin Kabushiki Kaisha). *U.S. 3,752,832*. Tocopherol and unsaponifiable matter from rice oil are combined to produce an antioxidant effect in lipids.

**CO-OXIDATION PROCESS FOR THE PRODUCTION OF SYNTHETIC FATTY ACIDS.** E.P. Kammann, Jr. (Emery Industries, Inc.). *U.S. 3,754,010*. Aliphatic monocarboxylic acids are obtained by the autoxidation of paraffin/olefin mixtures in the presence of a catalyst at atmospheric pressure. The process provided efficient oxidation of both the paraffin and the olefin at low temperatures while maintaining acceptable reaction rates and gives an improved distribution to a narrow range of preferred synthetic fatty acids.

**PLICATIC ACID ESTERS.** J. Howard and T.D. McIntosh (Rayonier Inc., Olympic Res. Div.). *U.S. 3,754,937*. The

use of alkyl and aryl esters of plicatic as antioxidants for fats and oils is disclosed.

**PREPARATION OF FATTY ACID ESTER MIXTURES ENRICHED IN UNSATURATES.** J.P. Hutchins (Procter & Gamble). *U.S. 3,755,335*. The process comprises transesterifying natural fats and oils with a lower alcohol and selectively extracting the unsaturated fatty acid esters with a two phase solvent system comprising a hydrocarbon and gamma-butyrolactone.

**METHOD OF SEPARATING FATTY ACIDS.** T.L. Blaney (Procter & Gamble). *U.S. 3,755,389*. A process for separating saturated fatty acids (predominantly palmitic and stearic) from unsaturated fatty acids (predominantly oleic) comprises the steps of dissolving the fatty acids in methyl formate, cooling the solution to 30F, and separating the crystallized fatty acids from the solution.

**PROCESS FOR CONTINUOUSLY SEPARATING GLYCERIDES.** A. Viarengo and R. Pasculli. *U.S. 3,755,390*. The process comprises providing a very dilute mixture of glycerides and solvent, cooling and filtering it, and separating the crystallized glycerides from the solvent. An apparatus for accomplishing the separation is also described.

## • Fatty Acid Derivatives

**REACTIONS OF ISOPROPENYL STEARATE WITH DIETHYL MALONATE, ACETOACETIC ESTER AND RELATED KETO ESTERS, ENOL ESTERS.** XVII. E.S. Rothman, G.G. Moore and S.S. Hecht (Eastern Regional Res. Lab., Philadelphia, Pa. 19118). *J. Org. Chem.* 38, 2540–3 (1973). The major product from the acid-catalyzed reaction of isopropenyl stearate with diethyl malonate is identified as the  $\alpha$ -pyrone, 6-ethoxy-3-hexadecyl-4-stearoyloxy-2H-pyran-2-one. Alcoholysis of the 6-alkoxy  $\alpha$ -pyrone proceeds unusually easily without requiring catalysis. Acetoacetic ester and 3-oxoglutarate esters react analogously with isopropenyl stearate to form  $\alpha$ -pyrones.

(Continued on page 497A)

## Call for Nominations Award in Lipid Chemistry

### Sponsored by Applied Science Laboratories

In April 1964 the Governing Board of the American Oil Chemists' Society established an Award in Lipid Chemistry under the sponsorship of the Applied Science Laboratories Inc., State College, Pa. Previous awards were presented as follows: Erich Baer, August 1964; Ernest Klenk, October 1965; H.E. Carter, October 1966; Sune Bergstrom, October 1967; Daniel Swern, October 1968; H.J. Dutton, October 1969; E.P. Kennedy, September 1970; E.S. Lutton, October 1971; A.T. James, September 1972; and F.D. Gunstone, September 1973.

The award consists of \$2500 accompanied by an appropriate certificate. It is now planned that the 11th award will be presented at the AOCS Fall Meeting in Philadelphia, September 29-October 3, 1974.

### Canvassing Committee Appointees

Policies and procedures governing the selection of award winners have been set by the AOCS Governing Board. An Award Nomination Canvassing Committee has been appointed. Members are: C.D. Evans, Chairman; C.W. Williams; D.L. Berner; G. Fuller; and R.J. Buswell. The function of this committee is to solicit nominations for the 11th award. Selection of the award winner will be made by the Award Committee whose membership will remain anonymous.

### Rules

The rules prescribe that nominees shall have been responsible for the accomplishment of original research in lipid chemistry and must have presented the results thereof through publication of technical papers of high quality. Preference will be given to individuals who are actively associated with research in lipid chemistry and who have made fundamental discoveries that affect a large segment of the lipid field. For award purposes, the term "lipid chemistry" is considered to embrace all aspects of the chemistry and biochemistry of fatty acids, of naturally occurring and synthetic compounds and derivatives of fatty acids, and of compounds that are related to fatty acids metabolically, or occur naturally in close association with fatty acids or derivatives thereof. The award will be made without regard for national origin, race, color, creed or sex.

Letters of nomination together with supporting documents must be submitted in octuplicate to C.D. Evans, Northern Regional Research Center, 1815 N. University, Peoria, Ill. 61604 before the deadline of April 1, 1974. The supporting documents shall consist of professional biographical data, including a summary of the nominee's research accomplishments, a list of his publications, the degrees he holds, together with the names of the granting institutions, and the positions held during his professional career. There is no requirement that either the nominator or the nominee be a member of the American Oil Chemists' Society. In addition, letters from at least three other scientists supporting the nomination must be submitted in octuplicate.

**Remember the DEADLINE, April 1, 1974**



## • Abstracts . . .

(Continued from page 496A)

EFFECT OF OZONATION MEDIUM, AND DECOMPOSITION CONDITIONS ON THE YIELDS OF OZONOLYSIS REACTIONS. M. Naudet and A. Pelloquin (National Lab. of Oils and Fats—ITERG, Univ. of Provence, Marseille, France). *Rev. Franc. Corps Gras* 20, 89-94 (1973). With the use, during ozonation, of a polar hydrated solvent which cannot react with the substrate or the reaction products, the formation of secondary products—principally parasitic esters—during preparative ozonolysis, may be greatly reduced. The maximum amount of water which may be used is that compatible with a total solubilization of the substrate.

STABILITY OF SULFATED OILS AFTER HYDROLYSIS. J. Pore and C. Chasseboeuf (Society of Houghton Products, 92-Puteaux, France). *Rev. Franc. Corps Gras* 20, 153-7 (1973). To determine their stability, sulfated oils derived from foots, castor oil and spermaceti were hydrolyzed in acid medium at various temperatures and pH values. The combined  $\text{SO}_3$  was determined by Epton's method and the variation of this content was used as a criterion of degree of hydrolysis. It was found that this hydrolysis is very slow. The poor stability of certain oils is not due to hydrolysis but to a separation of aqueous and oily phases during storage or uses.

DEHYDROGENATION OF FATTY ACIDS TO THE CORRESPONDING  $\alpha,\beta$ -UNSATURATED DERIVATIVES. G. Cainelli, G. Cardillo and A. Umami Ronchi. *J. Chem. Soc. Chem. Comm.* 1973, No. 3, 94-5. The  $\alpha$ -anions of linear fatty acids can be dehydrogenated with 2,3-dichloro-5,6-dicyanobenzoquinone to give exclusively the (*E*)- $\alpha,\beta$ -unsatd. derivatives. (World Surface Coatings Abs. No. 372)

INCORPORATION OF NON-WHEAT FLOURS OR STARCHES INTO BAKED GOODS. C.C. Tsen and W.J. Hoover (Kansas State Univ. Research Found.). *U.S. 3,752,675*. There is disclosed a method for incorporating non-wheat grain or tuber flours or starches into wheat flour-based bread, baked, or fried goods doughs at levels which would deleteriously affect the quality of the end products. The method comprises introduction of 0.1-3% of an additive selected from the group of sodium salts of acyl lactylates of  $\text{C}_{14}$ - $\text{C}_{22}$  fatty acids, and the condensation product of 10-95 parts of ethylene oxide and 90-5 parts of a partial glycerol ester of  $\text{C}_{10}$ - $\text{C}_{24}$  fatty acid containing at least 10% monoglyceride. The supplemental flour or starch may be added at levels as high as 40% of the wheat flour by use of 0.5% of the additive. A protein source material may also be added to supplement the wheat protein in the dough so long as the additive is present.

MULTIFUNCTIONAL EMULSIFICATION AGENTS. B.D. Buddemeyer. *U.S. 3,752,770*. There are disclosed powdered, free flowing, relatively non-hygroscopic emulsification compositions for improving the physical properties and quality of food products, and especially carbohydrate containing food products, confections, and prepared mixes. The compositions contain (a) 20-80 parts of at least one of the aliphatic polyol esters of  $\text{C}_{10}$ - $\text{C}_{24}$  fatty acids, glyceryl lactopalmitate, glyceryl lactostearate, succinylated monoglycerides, and acetylated tartaric acid esters of mono- and diglycerides; (b) 80-20 parts of at least one polyoxyethylene derivative of any of the polyol esters of fatty acids and having a total of 5-100 moles of ethylene oxide per mole of polyol ester; and (c) a hydrogenated triglyceride to the extent of 20-80% of the composition.

PHOSPHATIDE SEPARATION. R. Aneja and J.S. Chadha (Lever Bros.). *U.S. 3,752,833*. *N*-acyl phosphatides, e.g., *N*-acetylcephalin, are separated from phosphatides without an acylatable amino group, such as lecithin, by lowering the pH

## Laubscher promoted to president of Woodson-Tenent

American Biomedical Corporation recently announced the promotion of James Laubscher to president of Woodson-Tenent Laboratories, Memphis, Tenn. He has been with Woodson-Tenent as vice president for two and one-half years.

Laubscher received his M.S. degree in agricultural chemistry from the University of Arizona in 1968. He has had advanced studies in pesticide residue, chemistry, and business management. ■

to less than 3.5 under aqueous conditions and then solvent fractionating with acetone or methyl acetate.

SELECTIVE REACTION OF FATTY ACIDS AND THEIR SEPARATION. B.F. Ward (Westvaco Corp.). *U.S. 3,753,968*. A process for making a dicarboxylic acid having 21 carbon atoms from fatty acids is accomplished by reacting the linoleic acid portion of a fatty acid mixture with up to 26% of fatty acids of acrylic acid and with 0.01-0.05% of other fatty acids in the presence of iodine catalyst at 200-270°C. The fatty acid-dicarboxylic acid mixture is then separated by distillation into an oleic-type fatty acid and a  $\text{C}_{21}$  dicarboxylic acid. This process is especially applicable to separating tall oil fatty acids.

## • Biochemistry and Nutrition

EFFECT OF  $\text{CO}_2$  CONCENTRATION OF PHOSPHOLIPID METABOLISM IN THE ISOLATED PERFUSED RAT LUNG. W.J. Longmore, C.M. Niethe, D.J. Sprinkle and R.I. Godinez (Dept. of Biochem., St. Louis Univ. Schl. of Med., St. Louis, Mo. 63104). *J. Lipid Res.* 14, 145-51 (1973). Studies have been carried out on the incorporation of [ $^{14}\text{C}$ ]glucose, [ $^{14}\text{C}$ ]pyruvate, [ $^{14}\text{C}$ ]acetate, and [ $^{14}\text{C}$ ]palmitate into the phospholipids of the isolated perfused rat lung in the presence of either 6 or 45 mM total  $\text{CO}_2$  concentration in the perfusion medium. Incorporation of [ $^{14}\text{C}$ ]glucose into total phospholipid and into the phosphatidylcholine fraction was increased 10-53% over the 2-hr perfusion period in lungs perfused with medium containing 45 as compared with 6 mM  $\text{CO}_2$ . The incorporation of [ $^{14}\text{C}$ ]acetate, [ $^{14}\text{C}$ ]pyruvate and [ $^{14}\text{C}$ ]palmitate was not affected by the change in medium  $\text{CO}_2$  concentration. Increased incorporation of [ $^{14}\text{C}$ ]glucose combined with a shift toward greater incorporation into the fatty acids of the phosphatidylcholine fraction produced a maximum increase of 90% in [ $^{14}\text{C}$ ]glucose incorporation into the fatty acids of phosphatidylcholine after 2 hr of perfusion in the presence of medium containing 45 mM  $\text{CO}_2$  as compared with 6 mM  $\text{CO}_2$ . The increase in medium  $\text{CO}_2$  concentration produced as much as a 150% increase in [ $^{14}\text{C}$ ]glucose incorporation into palmitate derived from the phosphatidylcholine fraction. The results provide evidence that glucose functions as an important precursor of palmitate in the phosphatidylcholine fraction of lung phospholipids and that the  $\text{CO}_2$  concentration of the perfusion medium affects the incorporation of glucose into palmitate.

USE OF THE ISOLATED PERFUSED RAT LUNG IN STUDIES ON LUNG LIPID METABOLISM. R.I. Godinez and W.J. Longmore (Dept. of Biochem., St. Louis Univ. Schl. of Med., St. Louis, Mo. 63104). *J. Lipid Res.* 14, 138-44 (1973). A procedure for the use of the isolated perfused rat lung in studies on metabolic regulation has been developed. The procedure, reasonably uncomplicated, yet physiological, maintains the lung so that edema is not observed. The phospholipid content remains normal, and incorporation of [ $^{14}\text{C}$ ]palmitate, [ $^{14}\text{C}$ ]acetate and [ $^{14}\text{C}$ ]glucose is linear with time for a minimum of 2 hr. The incorporation of [ $^{14}\text{C}$ ]palmitate and [ $^{14}\text{C}$ ]acetate into the total lung phospholipid fraction and into the phosphatidylcholine and phosphatidylethanolamine fractions has been studied. Increasing the concentration of palmitate in the medium from 0.14 to 0.51 mM increased by 60% the incorporation of [ $^{14}\text{C}$ ]palmitate into the total lung phospholipid fraction at 2 hr. When the palmitate concentration of the medium was 0.14 mM, addition of 0.11 and 0.79 mM oleate to the medium decreased [ $^{14}\text{C}$ ]palmitate incorporation into the total lung phospholipid fraction at 2 hr by 37 and 49%, respectively. The results suggest that the incorporation of exogenous fatty acids, present in the medium perfusing the lung, into lung phospholipids may depend upon the fatty acid composition of the medium. Known specific acyltransferase activities may be responsible for the ordered incorporation of available fatty acids into lung phospholipids.

*N*-HEXYL-O-GLUCOSYL SPHINGOSINE, AN INHIBITOR OF GLUCOSYL CERAMIDE  $\beta$ -GLUCOSIDASE. J.S. Erickson and N.S. Radin (Mental Health Res. Inst., Univ. of Michigan, Ann Arbor, Mich. 48104). *J. Lipid Res.* 14, 133-7 (1973). A synthetic

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analog of glucocerebroside, N-hexyl-O-glucosyl sphingosine, was found to inhibit the glucosidase in rat spleen that hydrolyzes glucocerebroside. At a concentration of 1  $\mu$ M, the analog inhibited the enzyme by 48%. The mode of action appeared to be competitive, probably aided by tight binding of the amine group to a carboxyl group near the enzyme's active site. Increasing or decreasing the chain length of the n-alkyl group attached to the nitrogen atom lead to decreased effectiveness. The inhibitory effect was maximal at pH 7.0, but it was still considerable at the enzyme's optimal pH, 5.0. It is suggested that the compound may be useful for inducing an animal model of Gaucher's disease.

**ALTERATIONS IN LIPID COMPOSITION OF PLASMA LIPOPROTEINS DURING DEPOSITION OF EGG YOLK.** D.A. Gornall and A. Kuksis (Dept. of Biochem. and Banting and Best Dept. of Med. Res., Univ. of Toronto, Toronto, Canada). *J. Lipid Res.* 14, 197-205 (1973). The profiles of total lipids and of the molecular species of individual lipid classes were compared among corresponding lipoproteins of plasma and yolk of the laying hen. A close qualitative correspondence was found in the makeup of the molecular species of glycerophospholipids and triglycerides of the very low density lipoproteins and the high density lipoproteins of plasma and yolk. There was a lower proportion of the trienoic triglycerides and of the dienoic glycerophospholipids in the egg yolk than in the plasma lipoproteins, and the greatest differences (20-30%) were noted between the high density lipoproteins. It was also observed that the plasma high density lipoproteins lost their cholesteryl esters upon entering the yolk. On the basis of these and comparable analyses of the plasma lipoproteins of the nonlaying hen, it is concluded that the laying hen synthesizes specific lipoproteins for deposition in the yolk, and these are carried in plasma and selectively transferred to the developing ovum without significant equilibration with the other plasma lipoproteins.

**EFFECT OF DIETARY FAT ON PANCREATIC LIPASE LEVELS IN THE RAT.** L.I. Gidez (Depts. of Biochem. and Med., Albert Einstein College of Med., Yeshiva Univ., Bronx, N.Y. 10461). *J. Lipid Res.* 14, 169-77 (1973). The effect of dietary fat on levels of lipase and other enzymes in rat pancreas has been studied. It was possible to raise levels of lipase in animals by supplementing their commercial chow diet with added fat or by raising the level of fat in semipurified diets from 4% to 22%. Pancreatic amylase levels decreased in rats fed the high fat diets, whereas levels of chymotrypsinogen and trypsinogen were unaffected. The type of carbohydrate in the semipurified diets made no difference. Thus, the levels of enzymes in rats fed dextrose-containing diets or cornstarch-containing diets were similar. On the basis of the present data, and results of others, it would appear that levels of pancreatic lipase are increased when the fat content of the diet is raised from about 5% to 15-22%, but that little or no additional increase in lipase levels can be attained by any further increase in the amount of dietary fat.

**CELLULAR AND ENZYMATIC CHANGES IN PORCINE ADIPOSE TISSUE DURING GROWTH.** D.B. Anderson and R.G. Kauffman (Dept. of

Meat and Animal Sci., Univ. of Wisc., Madison, Wisc. 53706). *J. Lipid Res.* 14, 160-8 (1973). Experiments were designed to define some of the cellular and metabolic changes in various areas of porcine adipose tissue during growth and to establish a relationship between these changes and the accumulation of fat in the domestic pig. 35 Male castrate pigs were killed at various ages from late fetal to 6.5 months. The following determinations were made on each animal: total carcass fat, adipose cell size and number by fixation of adipose tissue with osmium tetroxide, and the activities of acetyl CoA carboxylase, citrate cleavage enzyme, glucose-6-phosphate dehydrogenase, 6-phosphogluconate dehydrogenase and malic enzyme from perirenal adipose tissue and each of the three layers of subcutaneous backfat. Carcass adipose tissue expanded by a combination of adipocyte hyperplasia and hypertrophy up to 5 months, after which adipose expansion was accomplished by cellular hypertrophy only, with no significant increase in cell number. The activities of the selected lipogenic enzymes (expressed on an adipose cell basis) increased markedly at weaning and again during the rapid increase in percentage of body fat between 3.5 and 5 months. Enzyme activities reached a peak at 5 months, after which activities decreased to values approaching mature levels.

**KINETIC DEPENDENCE OF PHOSPHOLIPASE A<sub>2</sub> ACTIVITY ON THE DETERGENT TRITON X-100.** E.A. Dennis (Dept. of Chem., Univ. of California at San Diego, La Jolla, Cal. 92037). *J. Lipid Res.* 14, 152-9 (1973). A kinetic analysis is presented for the dependence of one form of phospholipase A<sub>2</sub> from cobra (*Naja naja*) venom on the presence of the nonionic detergent Triton X-100 for its activity towards egg phosphatidylcholine and synthetic dipalmitoyl glycerophosphorylcholine as substrates. An automatic recording pH-stat apparatus was employed in order to continuously monitor enzyme activity. The results obtained in this study are interpreted in terms of a change in the physical state of the phospholipid when Triton X-100 micelles convert phospholipid bilayers into mixed Triton X-100-phospholipid micelles; this is consistent with the requirement of this enzyme for substrates which are in micellar form rather than either monomers or bilayers. An apparent inhibition of phospholipase A<sub>2</sub> activity at high concentrations of Triton X-100 is described and discussed in terms of the micellar nature of the substrate.

**SELECTIVE MEASUREMENT OF TWO DIFFERENT TRIGLYCERIDE LIPASE ACTIVITIES IN RAT POSTHEPARIN PLASMA.** R.M. Krauss, H.G. Windmueller, R.I. Levy and D.S. Fredrickson (Molecular Disease Branch, Natl. Heart and Lung Inst., and Lab. of Nutr. and Endocrinology, Natl. Inst. of Arthritis, Metabolism and Digestive Diseases, Natl. Insts. of Health, Bethesda, Md. 20014). *J. Lipid Res.* 14, 286-95 (1973). Conclusive evidence has been obtained for the presence of both hepatic and extrahepatic triglyceride lipase activities (TGLA) in rat postheparin plasma, and an assay has been devised for their selective measurement. Heparin-released TGLA in plasma from the intact rat, like TGLA in post-heparin hepatic perfusate, was relatively resistant to inactivation by salt and protamine. Postheparin TGLA obtained from the supradaphragmatic portion of the rat, where any hepatic contribution was eliminated, was nearly completely inactivated by salt and protamine. Utilizing the different sensitivities to protamine inactivation of extrahepatic and hepatic TGLA, assay conditions were selected to achieve simultaneously the maximal reduction of extrahepatic TGLA with preservation of hepatic TGLA. This assay was validated using postheparin plasma from partially hepatectomized rats. The protamine-inactivated activity was independent of the amount of liver removed, whereas protamine-resistant activity was directly proportional to the amount of liver remaining. In the intact rat, liver appeared to be the major source of heparin-released TGLA measured at pH 8.6 with triolein substrate. It was further shown that both hepatic and extrahepatic lipases catalyzed hydrolysis of triglyceride-rich lipoproteins.

**EPINEPHRINE BINDING AND THE SELECTIVE RESTORATION OF ADENYLATE CYCLASE ACTIVITY IN FAT-FED RATS.** R.R. Gorman, H.M. Tepperman and J. Tepperman (Dept. of Pharmacol., State Univ. of N.Y., Upstate Med. Center, Syracuse, N.Y. 13210). *J. Lipid Res.* 14, 279-85 (1973). Fat feeding results in a progressive loss of epinephrine- and glucagon-stimulated adenylate cyclase activity in adipocyte plasma membrane sacs (ghosts). Basal and NaF-stimulated adenylate cyclase activities in fat-fed animals are not significantly different from those in preparations obtained from chow-fed rats. The high fat diet increases the mean adipocyte diameter rapidly, but increased cell size, at least in the case of epinephrine stimulation, is not responsible for the decreased hormone-stimulated

• Meetings. . .

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June 4-8, 1974—International Rapeseed Conference, Giesen, West Germany. Contact: Sekretariat des Internationalen Rapskongresses, D-44, Münster, Germany, Diepenbrockstrasse 32.

Jul. 22-24, 1974—Sixth International Sunflower Conference, Bucharest, Romania. Contact: Ion Trifu, Academy of Agricultural and Forestry Sciences, Blvd. Marasti 61, Bucharest 1, Romania.

Sept. 8-12, 1974—Sixth International Congress of Essential Oils, San Francisco, Calif. Contact: Sixth International Congress of Essential Oils, 60 E. 42nd., New York, N.Y. 10017.

Oct. 7-9, 1974—21st Canadian Spectroscopy Symposium, Ottawa, Can. Contact: J.L. Dalton, secretary, 21st Canadian Spectroscopy Symposium, Department of Energy, Mines and Resources, Mines Branch, 555 Booth St., Ottawa, Ont., K1A 0G1, Can. ■